



This is a report on the achievements of the Decommissioning, Contaminated Water and Treated Water Management Project (Development of Analysis and Estimation Technology for Ascertaining the Attributes of Fuel Debris) subsidized by METI Agency for Natural Resources and Energy that commenced in FY2025.

**TEPCO**

1

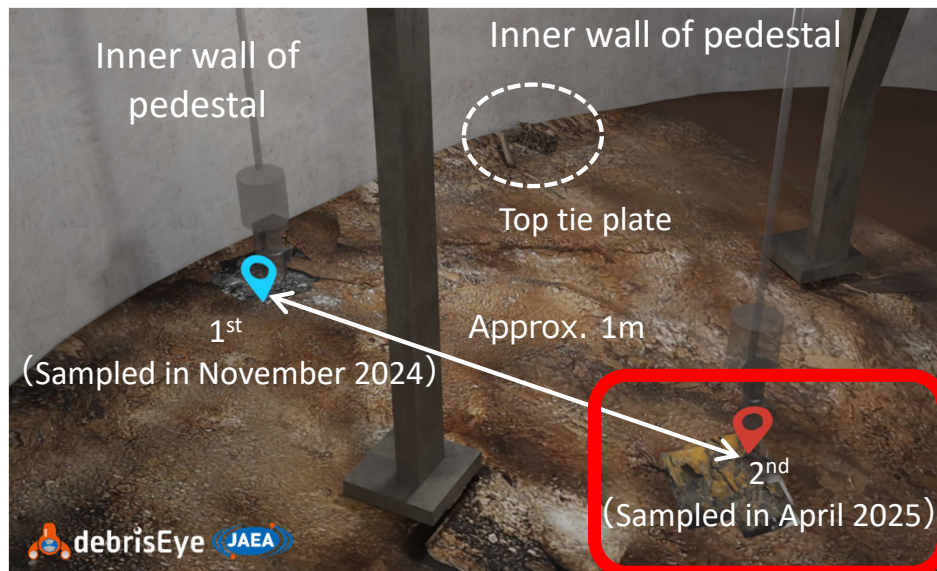
# **Analysis results for the fuel debris samples obtained during the first and second trial retrievals**

**August 27, 2026**

**Japan Atomic Energy Agency**

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

- To date, Tokyo Electric Power Company Holdings, Inc. has taken two fuel debris samples from the bottom of the Unit 2 pedestal.
- **The second fuel debris sample obtained through trial retrieval** (hereinafter referred to on occasion as, “2nd fuel debris” (the same goes for the 1<sup>st</sup> fuel debris sample)) was delivered to JAEA Oarai Nuclear Engineering Research & Irradiated Fuels Monitoring Facility (FMF) on April 25, 2025. <sup>[1]</sup> Thereafter, the sample was subject to non-destructive analysis, batching and sample transport through August 2025. The results of this process have already been reported. <sup>[2]</sup>
- **This report focuses mainly on the results of detailed analysis performed thereafter.**



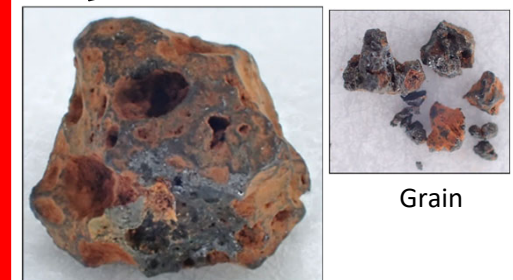
Schematic of the sampling locations of fuel debris during a Unit 2 trial retrieval (Bottom of the pedestal )

- ✓ Completion of originally planned analyses <sup>[3]</sup>
- ✓ Additional analyses implemented  
⇒ **Refer to the reference materials in this report**



(Approx. 9mm×Approx. 7mm)  
**1st Fuel debris**

- ✓ Non-destructive analysis and batching completed <sup>[2]</sup>
- ✓ Completion of originally planned analyses  
⇒ **Reported on in this report**



Clump  
(Approx. 5mm×Approx. 4mm)  
**2nd Fuel debris**

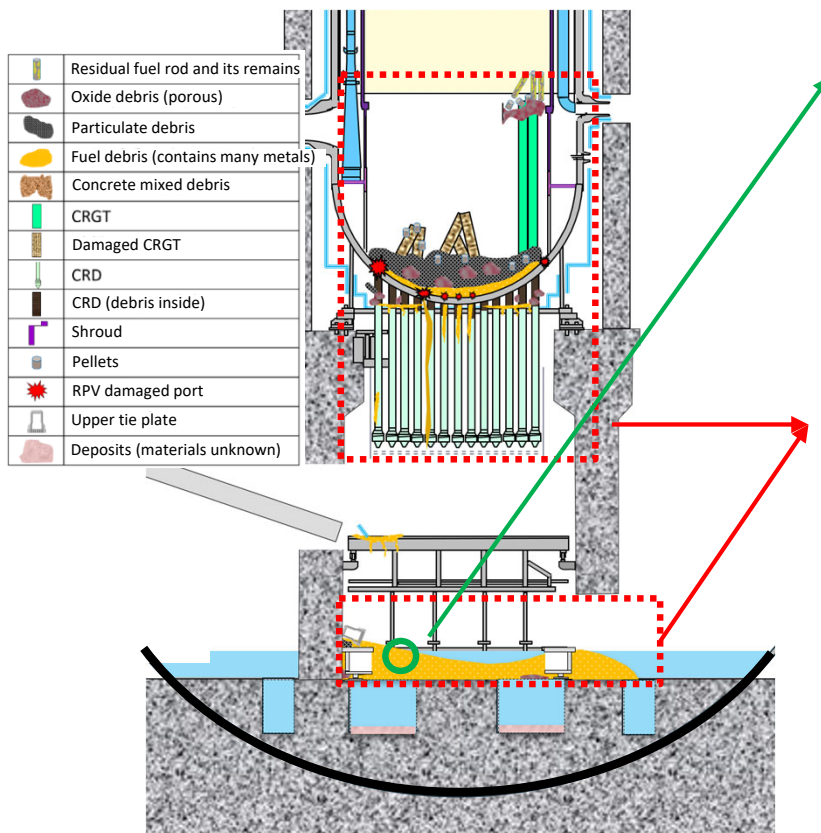
External appearance of fuel debris samples and analysis progress status (after delivery to the JAEA FMF)

[1] JAEA, TEPCO HD, Fuel debris Sample (2nd) Non-destructive analysis results (Most recent data), Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (138<sup>th</sup>), May 29, 2025, document 3-3

[2] JAEA, TEPCO HD, Fuel debris Sample (2nd) Non-destructive analysis results (Most recent data), and batching results, Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (141<sup>st</sup>), August 28, 2025, document 3-3

[3] JAEA, TEPCO HD, Fuel debris Sample (1st) analysis results (Most recent data), Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (144<sup>th</sup>), November 27, 2025, document 3-3

- 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



## 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)

Estimated drawing of the condition inside the core <sup>[1]</sup>

[1] JAEA, FY2024 Report of the FY2023 Subsidized Project of Decommissioning, Contaminated Water and Treated Water Management (Development of Analysis and Estimation Technology for Grasping Fuel Debris Properties (3. Development of Technology for Estimating RPV damage status and fuel debris migration behavior inside the PCV)).

## 1. Analysis for grasping the condition of the sampled area (Grasping the condition of fuel debris samples)

| Analysis items                                                                                                                                         | Analysis methods                                                                                             | Evaluation details                                                                                          | Examples of major applications for decommissioning                                                                                                          |
|--------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Basic information <span style="color: red;">【Already reported <sup>[1]</sup>】</span><br>• External appearance, weight, dose rate, density distribution | • Exterior, weight, dose rate measurement<br>• Imaging plate (IP)<br>• X-ray CT                              | Organization of basic information                                                                           | Basic information to review retrieval (existence and mount of pores, etc.)                                                                                  |
| Element content (1)<br>(elemental composition)                                                                                                         | • ICP-MS      • ICP-AES                                                                                      | Content of fuel components<br>Origin of major components                                                    | Basic information to review safety measures at retrieval, such as criticality evaluation, and storage methods                                               |
| Isotope ratio (2)                                                                                                                                      | • TIMS      • SIMS                                                                                           | U isotope ratio                                                                                             |                                                                                                                                                             |
| Radioactive concentration (3)                                                                                                                          | • γ-ray spectrometry<br>• α-ray spectrometry<br>• β-ray spectrometry<br>• Liquid scintillation counter, etc. | Accompanied condition of U with focal nuclides<br>Amount of radioactivity of nuclides targeted for analysis | Information to review technology development for non-destructive measurement at fuel debris retrieval<br>Data required to deliberate treatment and disposal |
| Element and compound distribution (4)                                                                                                                  | • SEM-EDX      • SEM-WDX<br>• TEM-EDX<br>• XRD                                                               | Evaluation of distribution of elements and compounds (including pores)                                      | Basic information to review retrieval methods and tools (estimation of hardness, toughness, etc.)                                                           |

## 2. Analysis for 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 (5) | • SEM-EDX      • SEM-WDX<br>• TEM-EDX      • Raman spectroscopy<br>• XRD<br>• μ-XRF/XRD/XAFS | Estimation of temperature and atmosphere when U particles, etc. are formed<br>Oxidation state of U, etc. | Precise estimated drawing of the condition inside the core to review retrieval methods and internal investigation |

- Detailed analysis of the 2<sup>nd</sup> fuel debris sample was carried out by four agencies in light of batching status and the amount required [for analysis].
- Additional analysis performed by the NDC was carried out using the 1<sup>st</sup> fuel debris sample.

【Fukushima Daiichi Nuclear Power Station Unit 2】

↓ Fuel debris transport

【JAEA Oarai Nuclear Engineering Institute】

Oarai Nuclear Engineering Institute (JAEA)  
NSI: Nuclear Science Research Institute (JAEA)  
NDC: MHI Nuclear Research & Development Co., Ltd.  
NFD: Nippon Nuclear Fuel Development Co., Ltd.

Non-destructive analysis

X-ray CT  
External appearance, weight,  
dose rate, IP  
γ-ray spectrometry  
SEM-WDX

- In order to assess the primary types and content ratios of crystal phases in the fuel debris, the NDC additionally analyzed the crystalline structure using XRD.
- A radioactivity analysis was also additionally performed by the NDC in order to add nuclide analysis targets

**Additional analysis was conducted on the first sample.**

【JAEA Harima】  
(SPring-8)

Solid analysis<sup>1</sup>  
(mechanical analysis)

μ-XAFS  
μ-XRF  
μ-XRD

Liquid analysis<sup>1</sup>  
(chemical analysis)

SEM-WDX  
TEM-EDX  
SIMS

ICP-MS  
Radioactivity  
analysis

【JAEA NSI】

ICP-AES  
TIMS  
Radioactivity  
analysis

【NDC】

**XRD**

ICP-MS  
ICP-AES  
**Radioactivity  
analysis**

【NFD】

SEM-EDX  
TEM-EDX  
Raman  
spectroscopy

Major analysis  
results

Micro crystal  
structure,  
Valence of U,  
etc.

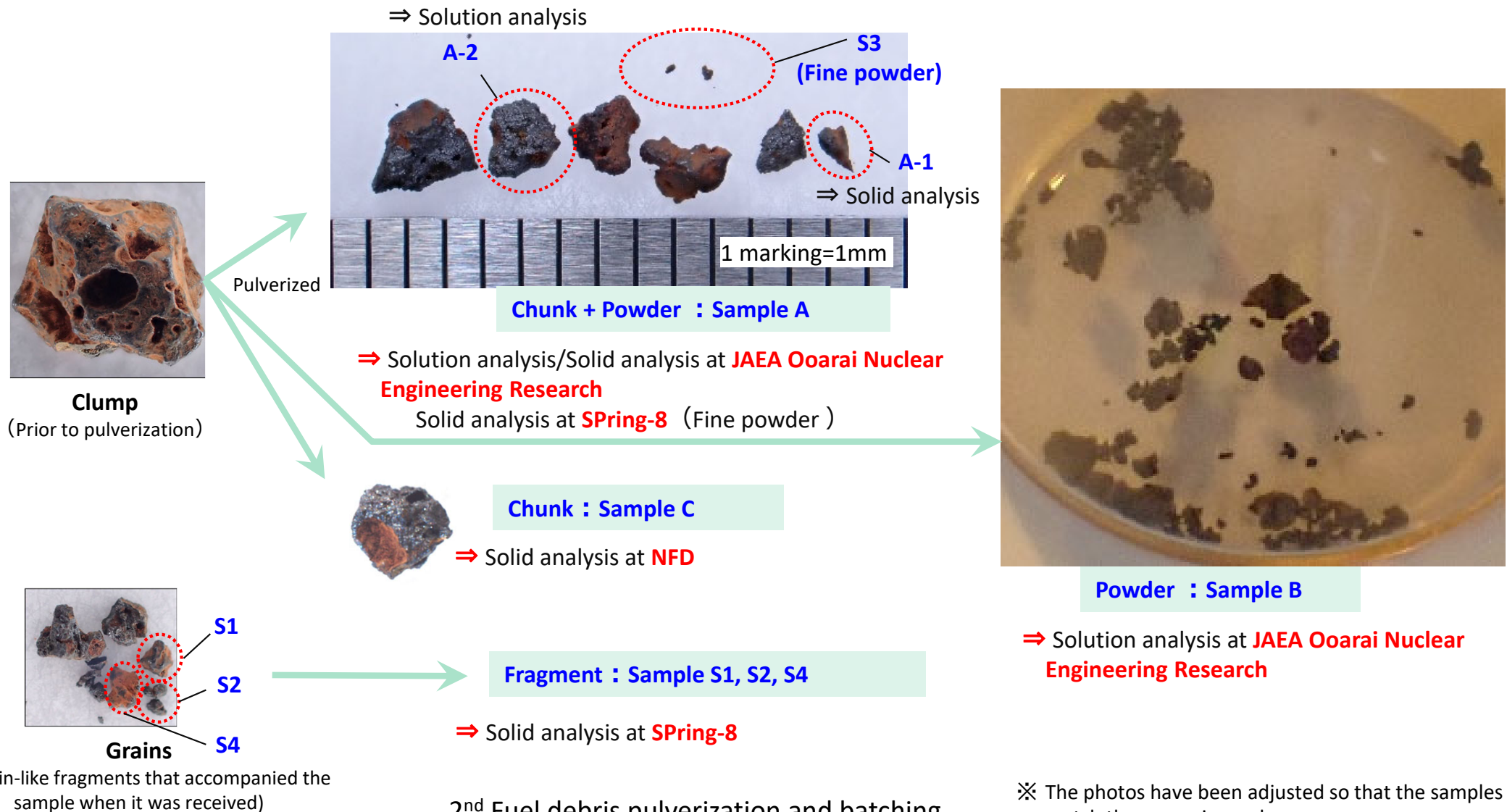
Fuel component element,  
U isotope ratio,  
Element and compound distribution,  
Radioactive concentration

Major elemental  
composition,  
U isotope ratio,  
Radioactive  
concentration

Element  
composition  
U isotope ratio  
**Radiation  
concentration  
Crystal structure**

U Crystalline  
structure,  
Composition,  
Element  
distribution

- As with the 1<sup>st</sup> sample, clump samples were pulverized (struck with a stainless steel rod weighing approximately 250 grams) to separate the clump into chunks and powder.
- Based on the size of the sample and the amount required for analysis, four sample batches were created for four agencies.



Powder B

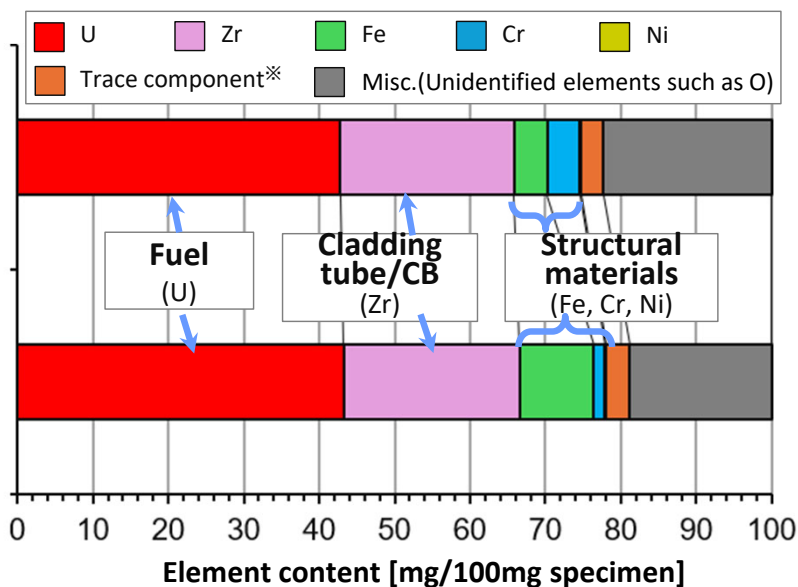


Mass: 43.5mg

ChunkA-2



Mass: 11.9mg



※ Trace components of Pu, Nd, Gd, B, Si, Ca, Mg, S, Ti, Mn, Co, Cu, Zn, Mo, Ru, Rh, Pd, Ag, Sn detected (Each was less than 1% of the sample mass)

Figure 1 Primary elements of the 2<sup>nd</sup> Fuel debris sample

Converted to molar ratios of major elements

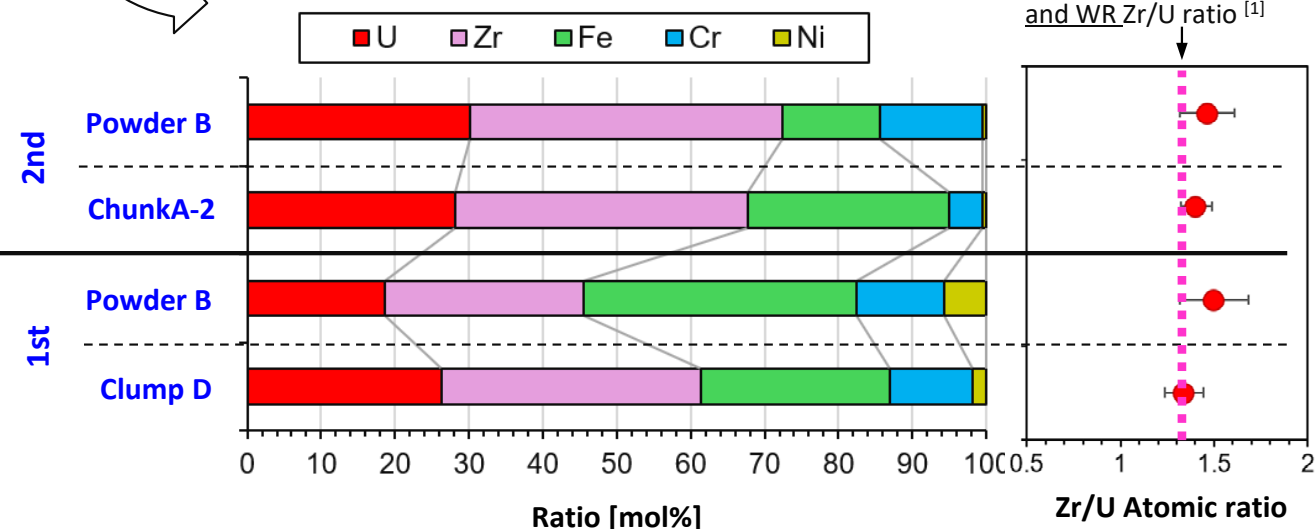


Figure 2 Comparison with 1st Fuel debris (Primary element ratio)

- As with the first sample, ratio of U to mass in the 2<sup>nd</sup> Fuel debris was the highest. (Figure 1)
- The ratios of U and Zr are higher than the 1<sup>st</sup> Fuel debris, but the ratios of structural materials, and Ni in particular, are lower. (Figure 2)
- In both the 1<sup>st</sup> and 2<sup>nd</sup> Fuel debris samples, the atomic ratio of Zr/U is close to the ratio of Zr/U that takes into consideration core cladding tubes and CB, etc. (Figure 2)

➤ It is assumed that both the 1<sup>st</sup> and 2<sup>nd</sup> Fuel debris samples comprise not just fuel, but also material from the cladding tubes and channel boxes, etc.  
⇒ Can be used as reference data for U concentration range.

[1] Sato et al., Nucl.Eng.Des., 404, 112205. Calculated based on Table1 (CB: Channel box, WR: Water load)

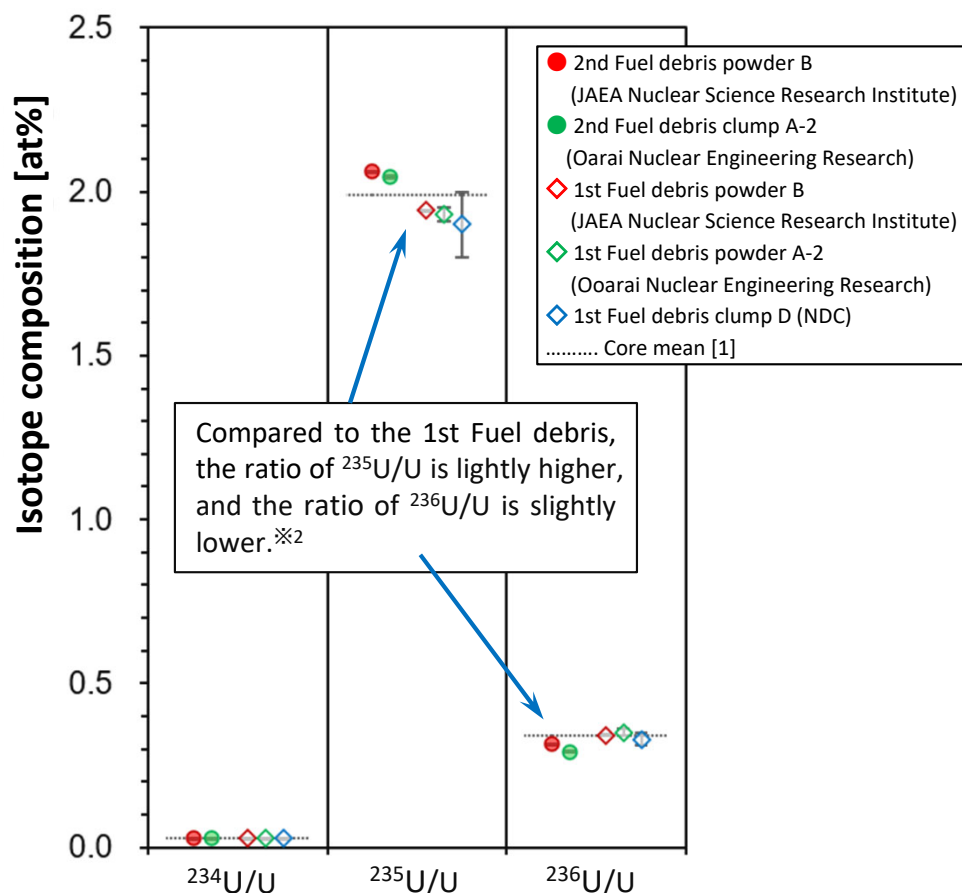


Figure 2<sup>nd</sup> Fuel debris U isotope composition and comparison with the 1<sup>st</sup> fuel debris sample

■ The  $^{235}\text{U}/\text{U}$  ratio of the 2<sup>nd</sup> Fuel debris is 2.02–2.04wt% close to the core mean [1] of Unit 2 even though there is a slight difference compared to the 1<sup>st</sup> Fuel debris sample.

✓ The  $^{235}\text{U}/\text{U}$  ratio in Unit 2 prior to the accident varies\*1, but in both the 1<sup>st</sup> and 2<sup>nd</sup> samples, [the ratio] is close to the core mean.

➤ This suggests mixture over a fairly wide area and not just in the limited area of the core.  
⇒  $^{235}\text{U}/\text{U}$  ratio trends of the fuel debris will be considered while deepening our understanding of how the accident progressed by looking at how the fuel debris was formed, and also while analyzing more of fuel debris samples taken from a wider area in the future.

\*1 The ratio of  $^{235}\text{U}/\text{U}$  (uranium concentration) in the core prior to the accident was between less than 1% to approx. 4%.

\*2 As fuel is burned,  $^{235}\text{U}$  is consumed, and  $^{236}\text{U}$  is formed as it captured neutrons, so there is no contradiction.

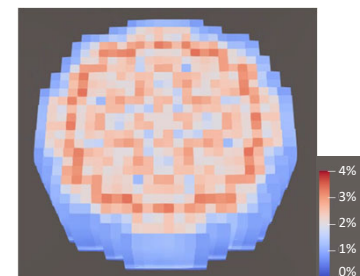
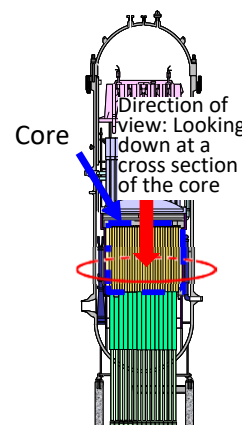


Figure Example of the uranium concentration distribution in a BWR reactor core [2]

(The redder the color the higher the concentration)

[1] Okumura et. al., Atomic Energy Society of Japan 2021 Spring Conference, 3B01.

[2] JAEA, TEPCO HD, Fuel debris Sample (1<sup>st</sup>) analysis results, Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (140<sup>th</sup>), July 31, 2025, Document 3-3

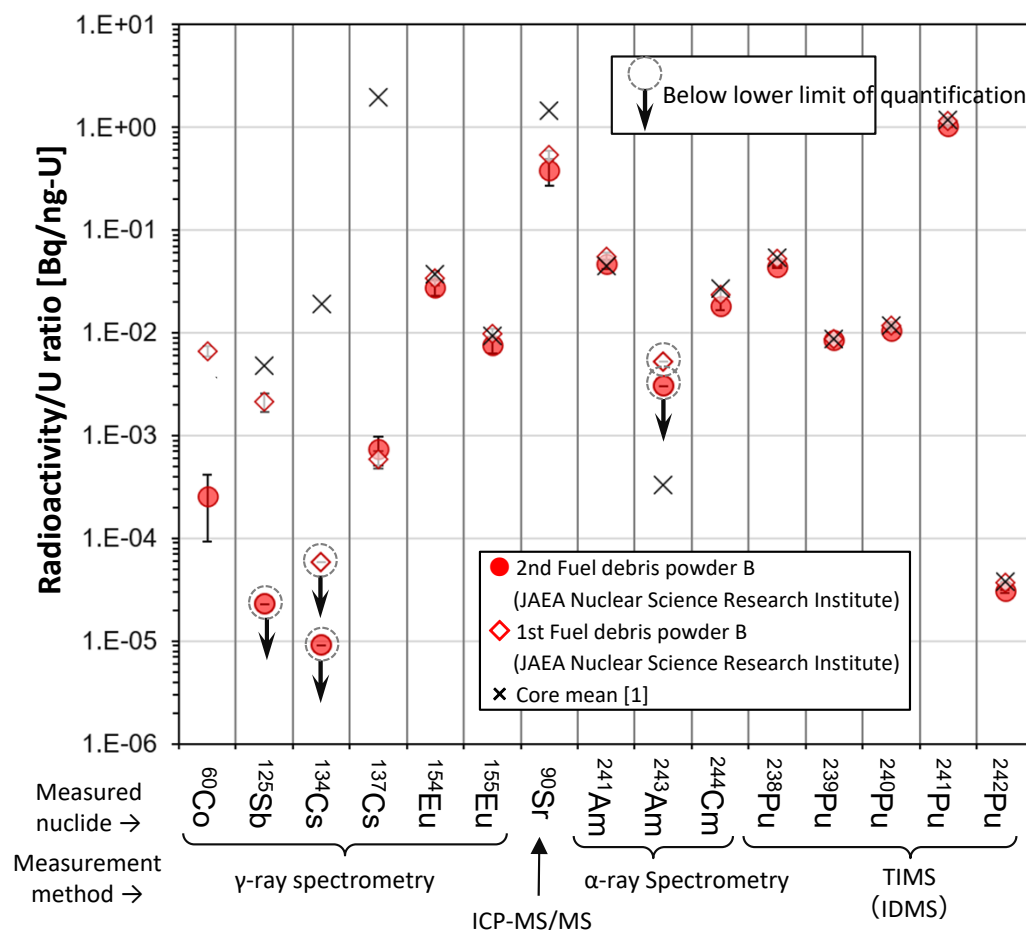


Figure Ratio of U mass to radioactivity for each nuclide in the 2<sup>nd</sup> Fuel debris sample and comparison with the 1<sup>st</sup> sample

Note: Radioactivity measurements were revised on June 30, 2026

Pu isotope radioactivity Was assessed using isotope composition obtained from TIMS (Thermal ionization mass spectrometry) and the Pu element Atomic weight measured with IDMS (isotope dilution mass spectrometry)

■ The breakdown of radioactivity in the second fuel debris sample shows the **same trends as the first sample with the exception that <sup>60</sup>Co and <sup>125</sup>Sb are scarce.** (Left figure)

- ✓ The contribution from <sup>90</sup>Sr, α nuclides (<sup>241</sup>Am, <sup>244</sup>Cm and Pu isotopes) and <sup>154</sup>Eu is large
- ✓ The contribution from <sup>134</sup>Cs and <sup>137</sup>Cs is small and small when compared to the core mean

➤ In both the 1st and 2nd samples, **it is possible that cesium volatilized during the accident due to the high temperatures thereby forming a sample with low γ dose rates**

■ The ratio between Eu, Am, Cm and Pu isotopes and U is close to the core mean in both the first and second samples.

➤ Association was confirmed between **<sup>154</sup>Eu and <sup>244</sup>Cm,※** which are focused on as indicator nuclides for detecting fuel debris, **and U between the locations where the 1<sup>st</sup> and 2<sup>nd</sup> samples were taken.**

⇒ Data on the association with U will be provided to the non destructive measurement technology development project that is underway.

## Region classification by cross-sectional observation

Chunk C

Through an optical microscope the metal phase appears white (looks bright)

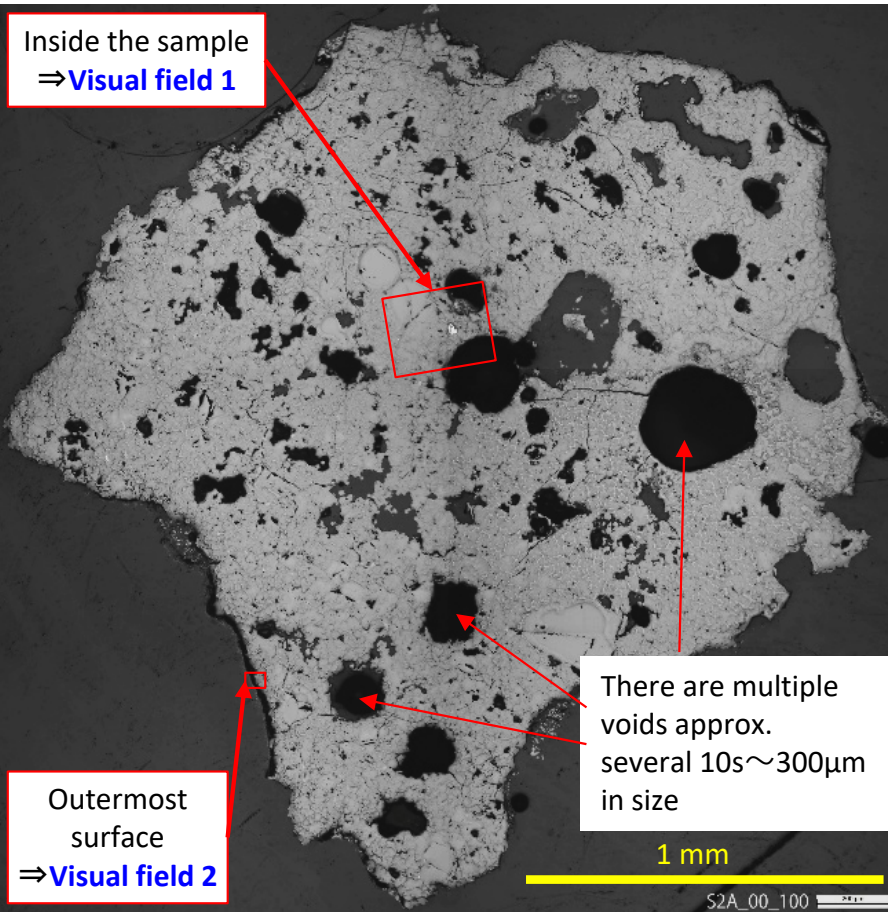


Figure 1 Optical micrograph of a cross section of the 2nd Fuel debris

Reference Area ratio of each region (Image analysis of Figure 1) Units: area %

| Fine mixed regions | Coarse U-Zr-O | Fe-Ni metal | Void |
|--------------------|---------------|-------------|------|
| 80                 | 2             | < 0.1       | 18   |

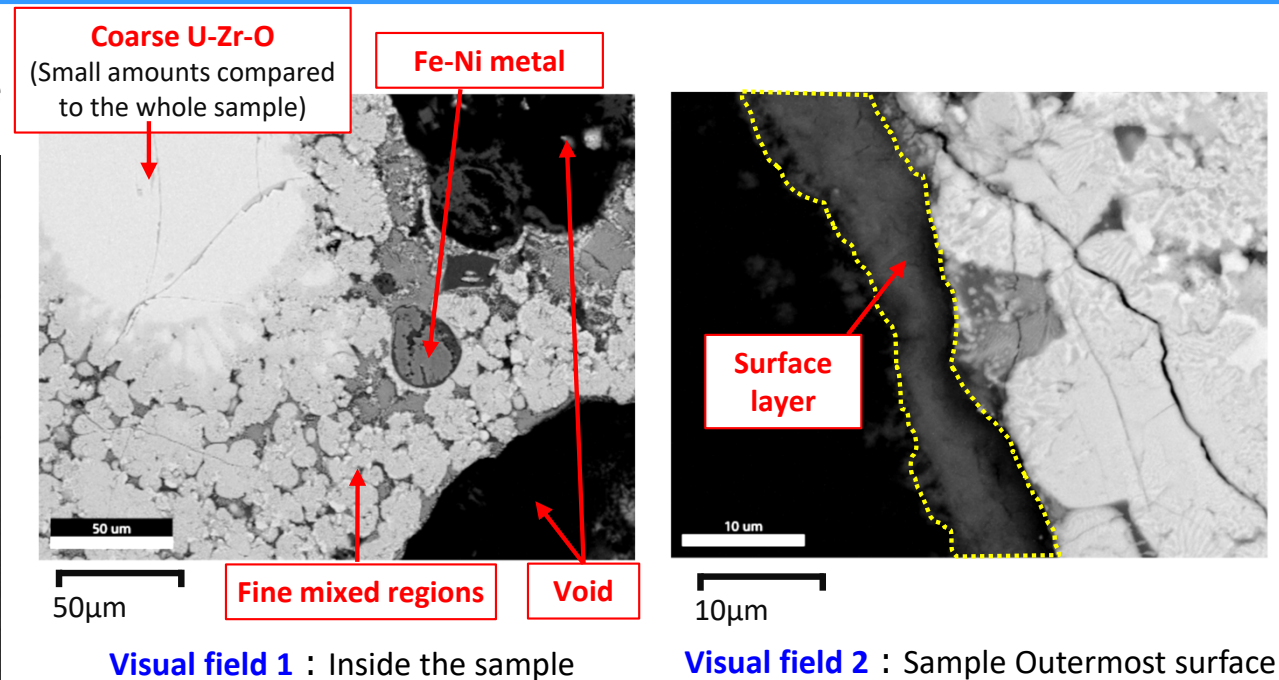
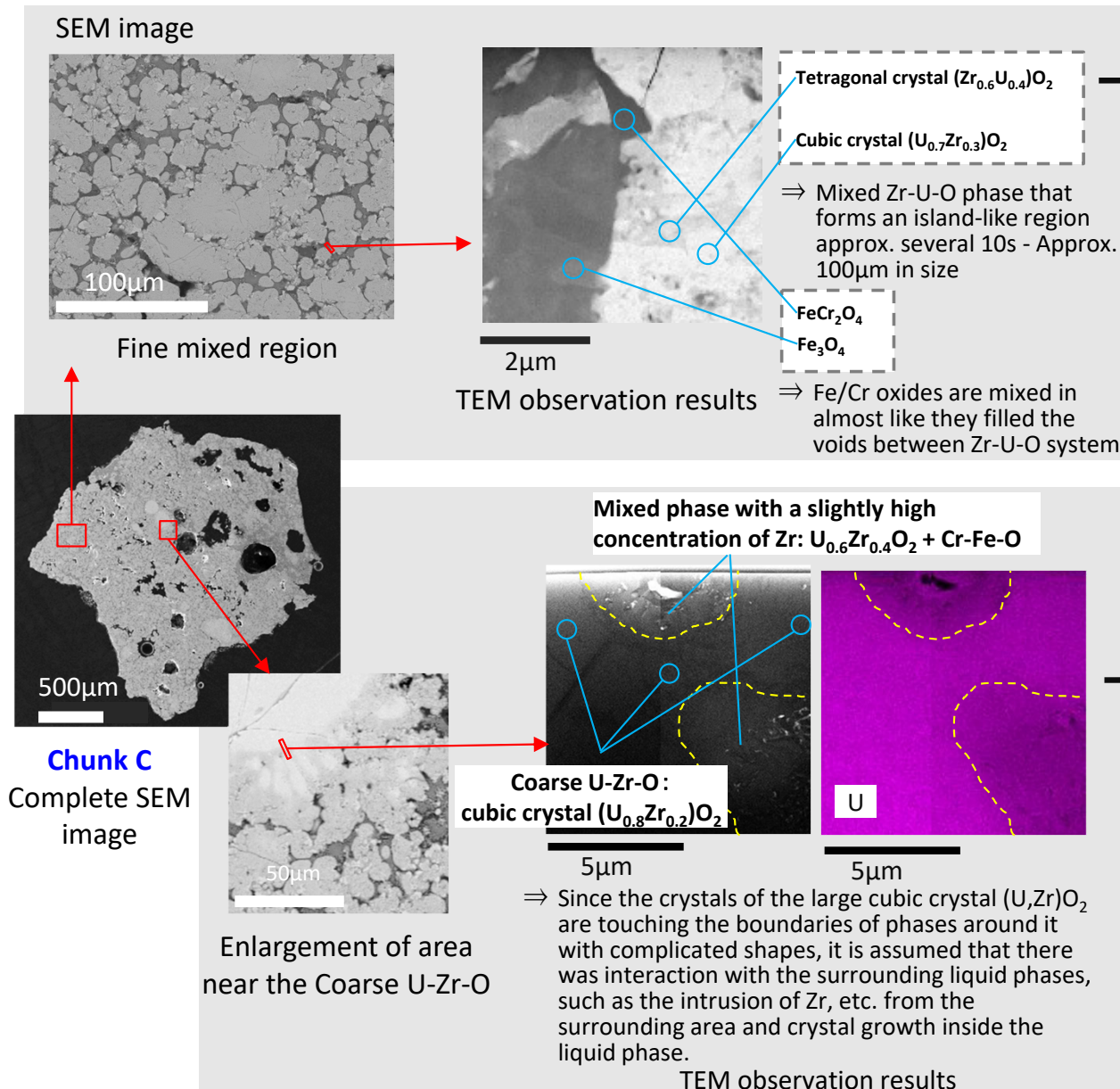


Figure 2 SEM Observation images of a cross section of the 2nd Fuel debris

- The 2<sup>nd</sup> Fuel debris sample was classified as having five major regions for the most part: a **Fine mixed region (Zr-U-Fe-Cr-O composite phase)**, a **Coarse U-Zr-O region**, a **Fe-Ni metal region**, **Voids**, and a **Surface layer (Ni-Fe-O)**. (Figures 1 and 2)
- Compared to the first sample, there are very few Fe-Ni metal phases and the fine mixed region makes up the majority of the sample.
- **There are voids distributed throughout the entire fine mixed region.**

➤ Compared to the first sample the second sample was easier to pulverize  
 ⇒ Basic data needed to deliberate retrieval methods and tools

For regions showing unique fine structures or elemental distributions, SEM-EDX and TEM-EDX analyses were used to identify oxide phases, metal phase composition, and crystalline structure, in order to form hypothesis about the process through which these regions were formed.



**Fine mixed region:** Occupies the majority of the cross section

- The fine structure observed shows characteristics consistent with the melting and solidification of **Zr-U-Fe-Cr-O systems**.
- It is hypothesized that during the accident **cladding tubes and CB, etc. and structural materials oxidized while they melted and mixed with the fuel** after which they cooled and solidified

**Coarse U-Zr-O:** Only a small amount exists in the cross section

- This is a large cubic crystal  $(\text{U,Zr})\text{O}_2$  that shows signs that it was touching the surrounding molten material
- It is hypothesized that there was **suspension consisting of the molten material and Coarse U-Zr-O**

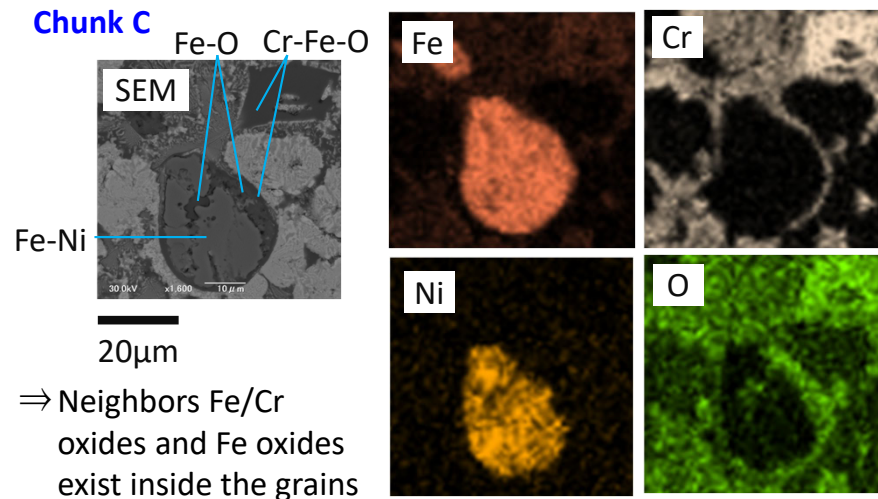


Figure 1 Fe-Ni metal and surrounding elements distribution

**Fe-Ni metal:** Only a very small percentage of the content ratio

- The Fe-Ni metal phase is in contact with  $\text{Fe}_3\text{O}_4$  and  $(\text{Fe,Cr})_3\text{O}_4$  (Figure 1).  $\text{Fe}_2\text{O}_3$  was not observed, and resembles mixtures forming through oxidation caused by steam.
- Out of all the elements coming from structural materials (Fe, Cr, Ni), Cr and Fe oxidized first due to the steam generated, leaving a metal phase with a high concentration of Ni.

## Chunk C

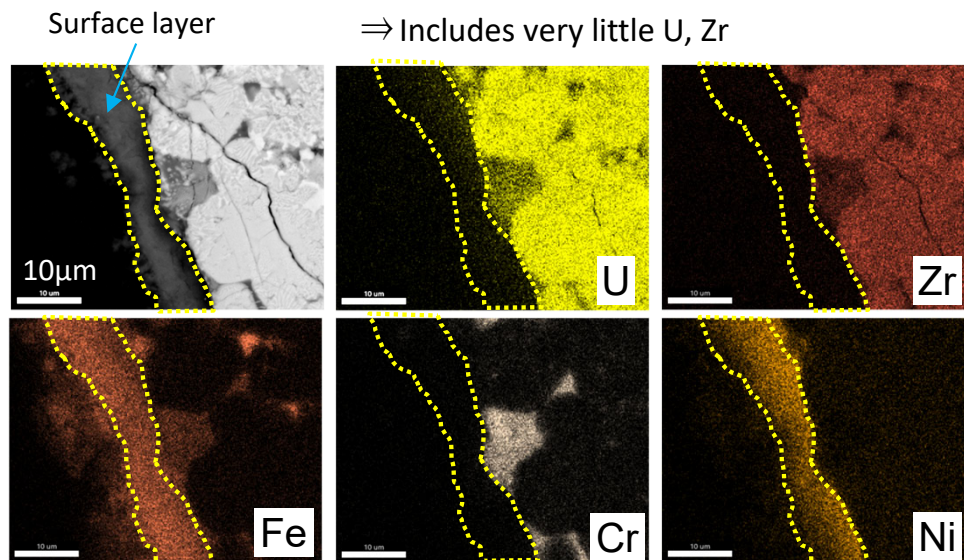


Figure 2 Primary element distribution near the outermost surface of the sample

**Surface layer:** Deposited to a thickness of approx. 10 - 50 μm

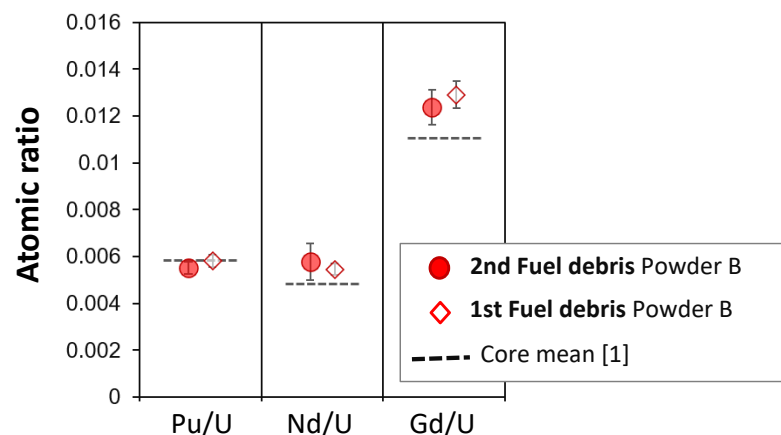
- Comprised mainly of Ni-Fe-O. Similar layers were seen in fragments accompanying the clump, thereby suggesting it contains hydroxides.
- It is assumed that after the most of the sample was formed through melting and solidification, this layer was formed as dissolved constituents in the cooling water precipitated onto the surface.

## Mixing with elements originating from the core

- If we focus on U, Pu, Nd and Gd<sup>※</sup>, even though there is only a slight difference in the isotope composition of both the first and the 2<sup>nd</sup> sample, the values obtained are close to the core mean for both. (Figure 1)

※ The isotope compositions of U/Pu (actinoid nuclide), Nd (fission product that indicates burn-up) and Gd (burnable poison) have distributions in the radial and axial directions consistent with the core prior to the accident.

➤ When the original molten material of the first and second samples was formed, it is hypothesized **materials throughout a relatively wide of the core**



Reference Ratio of U to Pu, Nd and Gd

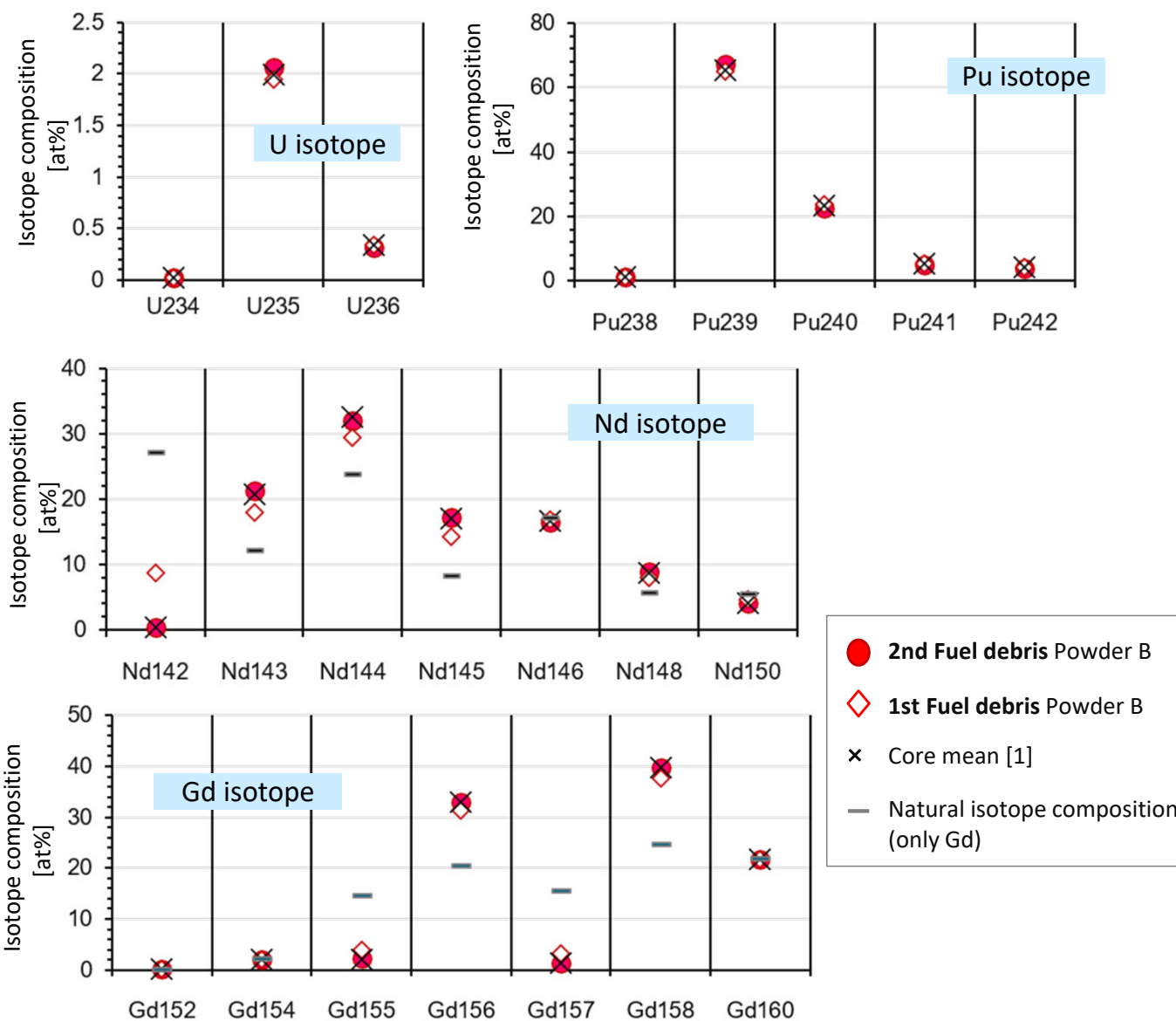


Figure 1 1<sup>st</sup>/2<sup>nd</sup> Fuel debris Pu, Nd and Gd isotope composition

(Comparison of TIMS analysis results from the JAEA Oarai Nuclear Engineering Research)

## Entrainment of structural material elements

- The percentage of structural material elements in the first and second samples is larger than the average composition of the core prior to the accident (Figure 1)
- Both samples suggest the possibility that as the molten material that formed in the core fell to the bottom of the RPV, it became entrained with structural material※<sup>1</sup> elements at the bottom of the core.
- ※<sup>1</sup> Lower tie plates, fuel support fixtures, etc.
- Compared with the first sample, the second fuel debris sample has almost no Ni and few metal phases. (Figure 2)
- It is possible that the entrained structural material elements, such as stainless steel, oxidized and separated into Fe-Cr oxides and Fe-Ni metal phases, and the second fuel debris sample comprises this oxide phase (the metal remained in another fragment)
- The second fuel debris sample has an angular shape, and most likely broke off of a larger fused solid.
- For both the first and second fuel debris samples, it is hypothesized that molten material was formed as the structural material elements at the bottom of the core became entrained after which they separated into oxide phases and metal phases thereby forming through different processes.

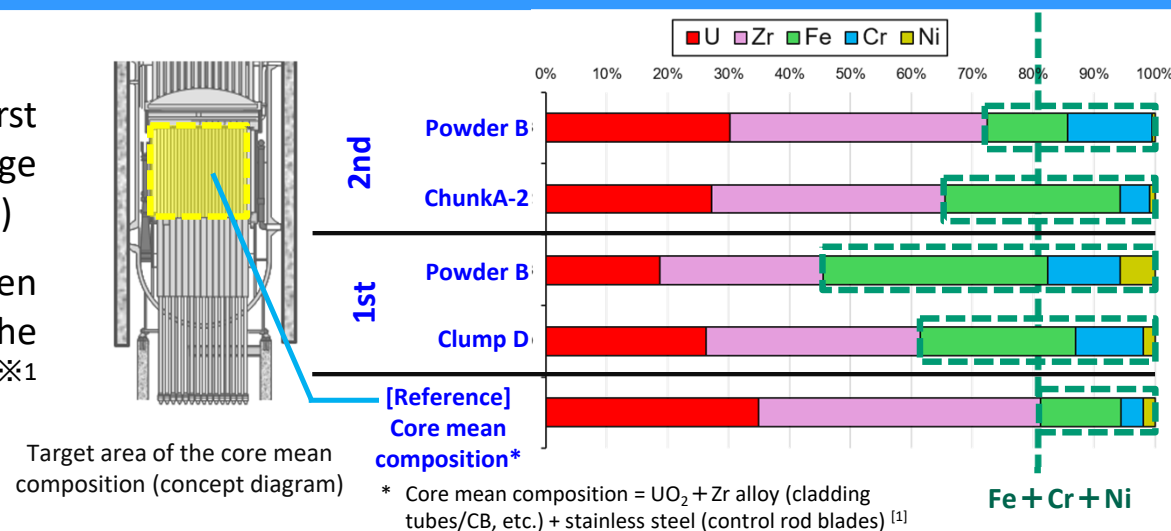


Figure 1 Solution analysis results (ratio of the primary five elements) and mean composition comparison

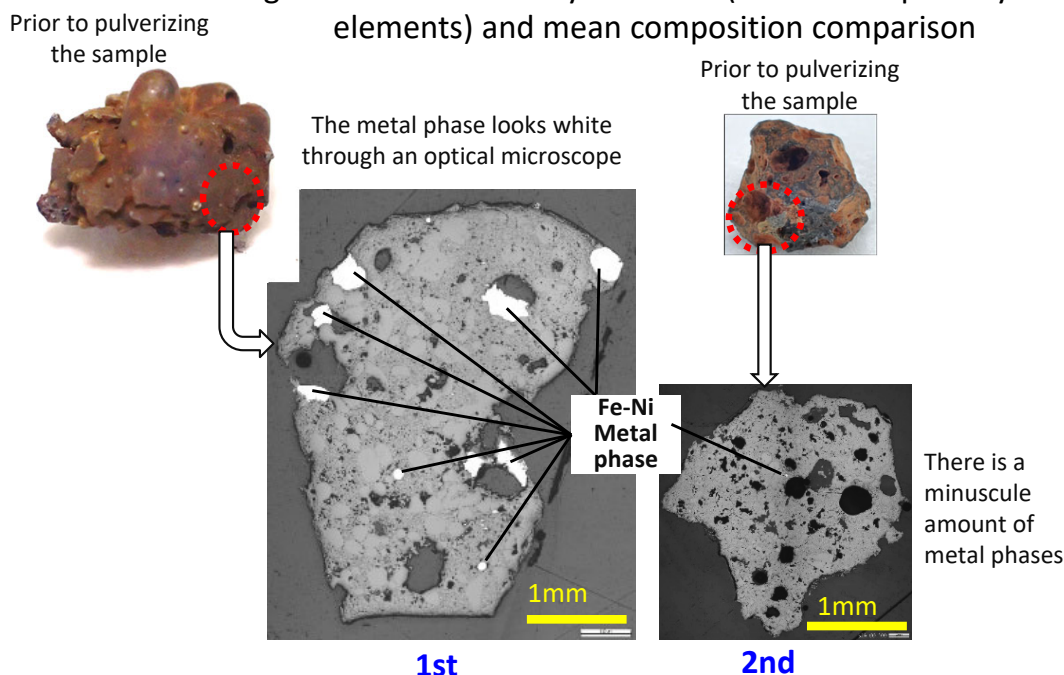
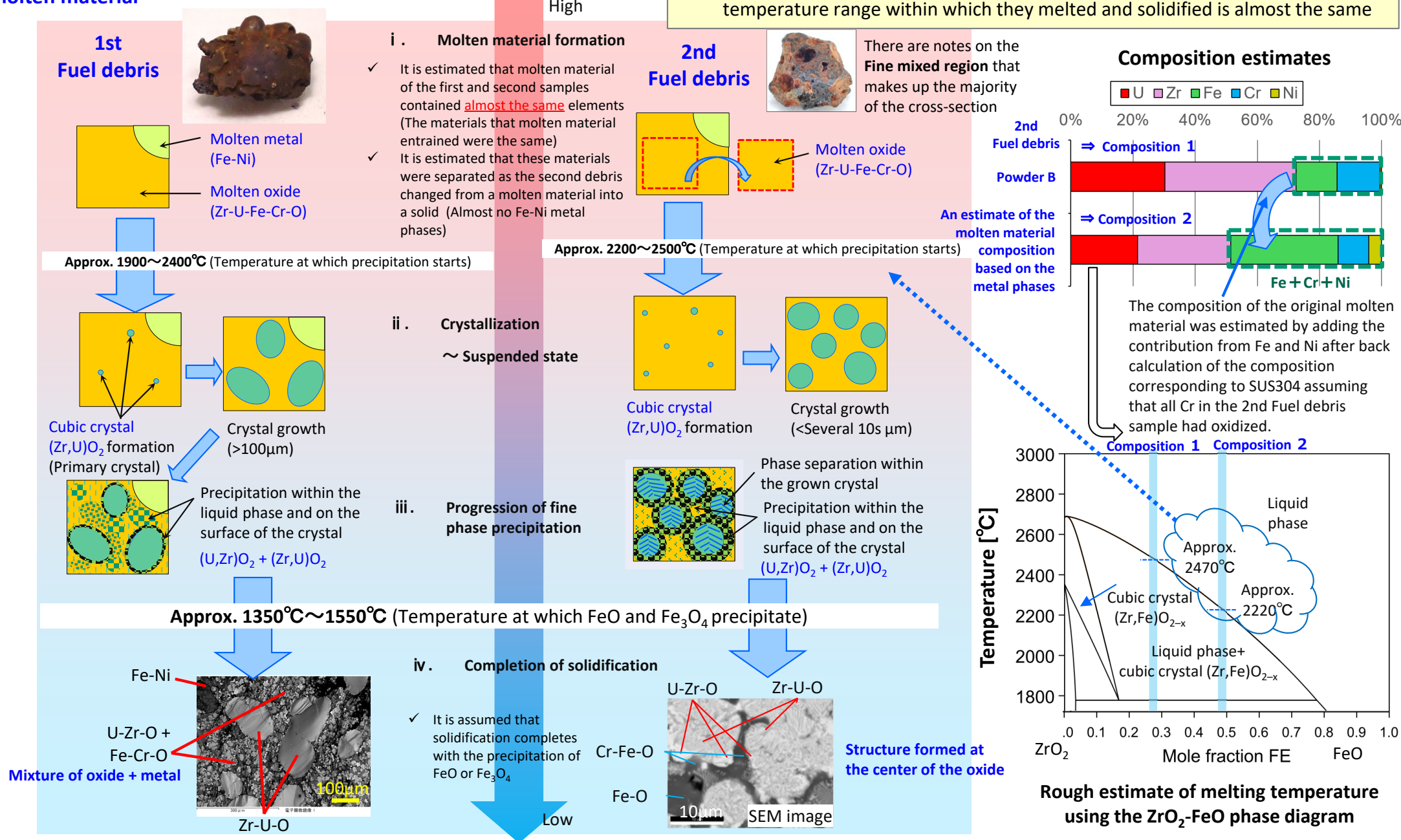
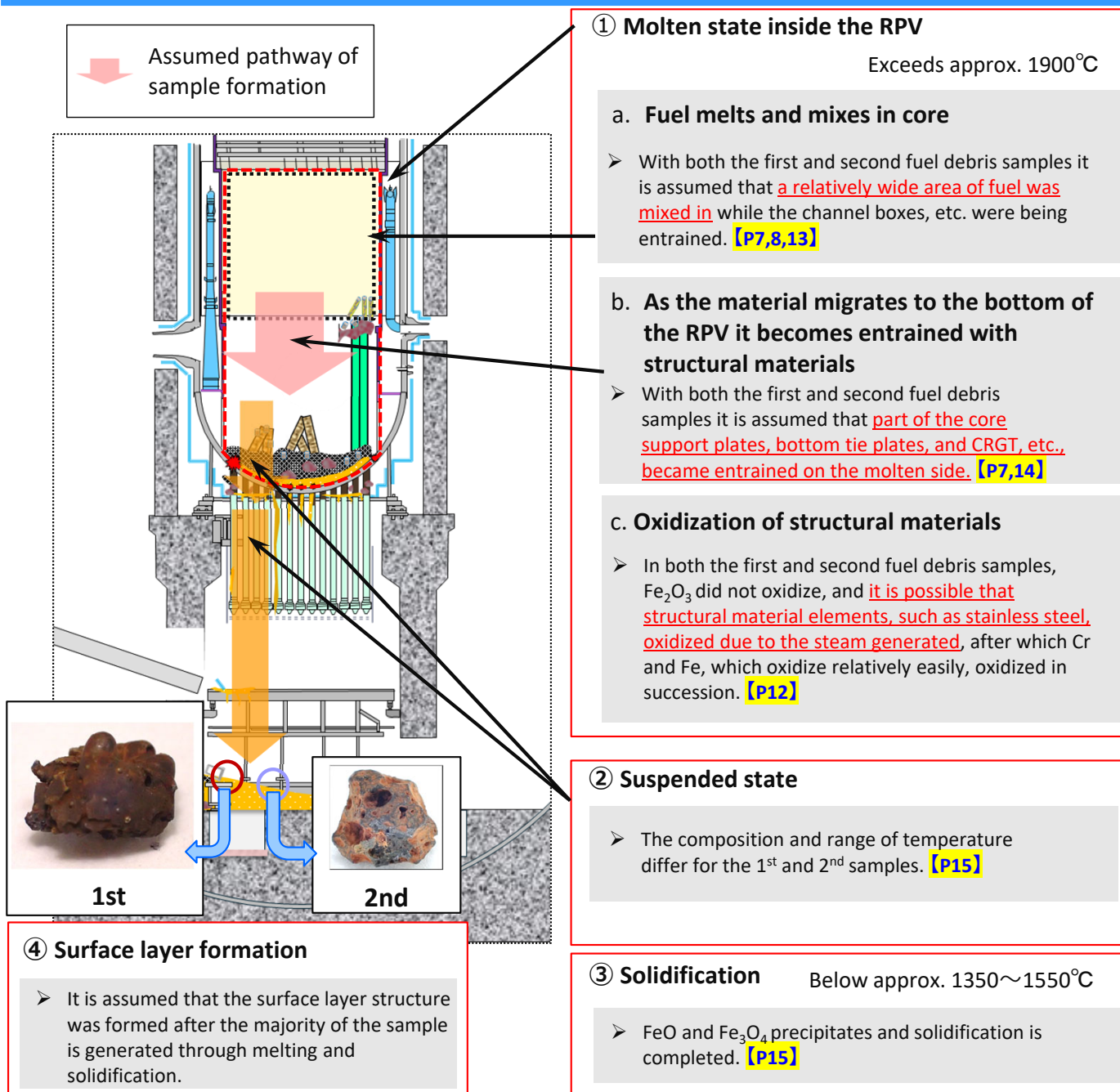


Figure 2 Observation of the cross-sections through an optical microscope

## Hypothesis about the solidification process of the molten material

➤ Whereas there are differences in crystal growth progress and phase separation process between both the first and the 2nd fuel debris samples, The temperature range within which they melted and solidified is almost the same





## Summary of hypothesis

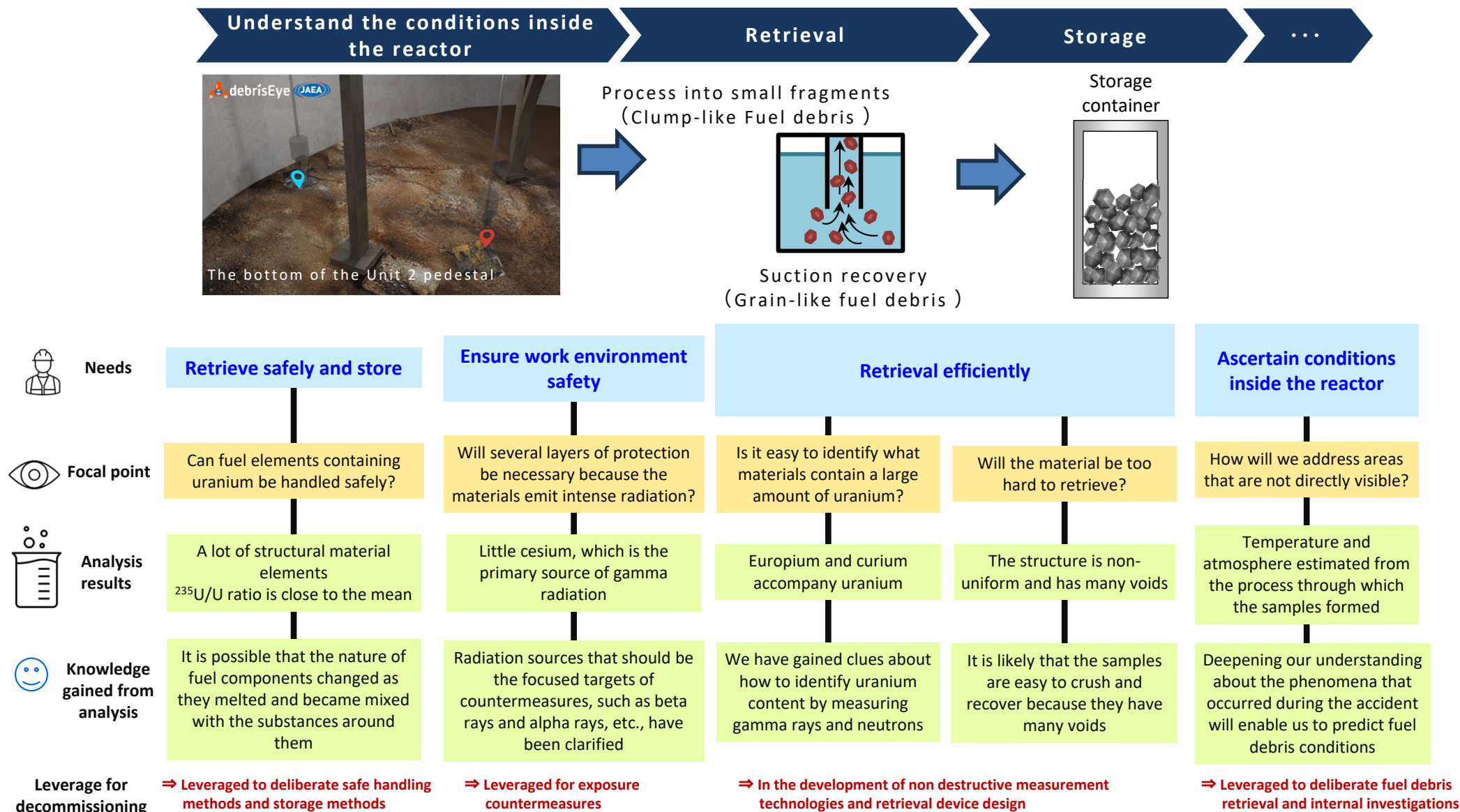
- Even though it is possible that the formation conditions, such as temperature, etc., differed slightly, **the first and second fuel debris samples formed through mostly the same pathway (Left diagram①~④)**

- By using analysis results to deepen our standing of the phenomena that actually occurred, we may be able to estimate conditions within the core that we are unable to see thereby enabling us to deliberate fuel debris retrieval methods and future internal investigations
- ⇒ Since these estimates are provisional, we will continue to engage in analysis and assessment with the intent of updating and refining them.

Figure Rough estimate of the generation process of the 1<sup>st</sup>/2<sup>nd</sup> Fuel debris samples

- The fuel debris sample obtained through the second trial retrieval displayed the following attributes:
  - ✓ As with the first sample, the U weight ratio was high. It is assumed that **Zr elements, cladding tubes and channel boxes, etc. were entrained along with structural material elements (FE, Cr, Ni).**
  - ✓ The  $^{235}\text{U}/\text{U}$  ratio of the sample was slightly different than the first sample, but close to the core mean. This suggests that fuel got mixed in not in a limited region of the core, but rather in a relatively wider region.
  - ✓ In regards to radiation concentration, although the concentration of **radioactive cesium was low**, as with the first fuel debris sample, the contributions from  $\beta$ -ray and  $\alpha$ -ray emitting nuclides, such as  $^{90}\text{Sr}$  and Pu isotopes, are large.
  - ✓ As with the first sample, there is a wide distribution of voids inside the sample, which is most likely why it was easy to pulverize.
  - ✓ Compared with the first fuel debris sample the percentage of Fe-Ni metal phases originating from structural material elements is low. During formation of the 2nd fuel debris sample the metal phase and the oxide phase separated, so it is possible that the metal phase migrated separately from the oxides. And, it is possible that the composition of the molten material prior to separation is similar to the first fuel debris sample. This does not contradict our assumptions to date, and gives us clues for estimating the behavior of fuel and structural materials during the accident and also the condition of the core.
- Even though the first and second fuel debris samples were taken from different locations, they are similar in terms of composition, structure, and the process through which they formed. No conclusions can be drawn about the conditions inside the core from these results alone, but it is possible that fuel debris that formed through a similar process exists in the vicinity of the location from which the second sample was taken.
- We will continue to analyze the sample in order to ensure safety during decommissioning and improve the accuracy of estimates about the conditions inside the reactor (refer to the next page for more information)

## – Leveraging the knowledge obtained through analysis for decommissioning –



Analysis will enable us to deliberate handling technology that matches fuel debris conditions and design equipment, and this knowledge will be leveraged for decommissioning

- When analyzing fuel debris with unknown composition and non uniform attributes, there are cases where the conventional methods for analyzing spent fuel where the composition is very heterogeneous and unique pretreatment, such as alkali fusion, etc., is performed, cannot be applied. So, even after the analysis results that have already been reported <sup>[1][2]</sup> were publicly released (hereinafter referred to as, “initial report”), we continued to advance analysis methods based on discussions with external experts.
- While using the analysis results to assess the attributes of the sample and the process by which it formed, we also compared analysis methods, matched nuclides, and inspected analysis conditions. As a result, we have found that there is room for improvement with conventional analysis methods in regards to the following three issues, and we have performed a reassessment of them.

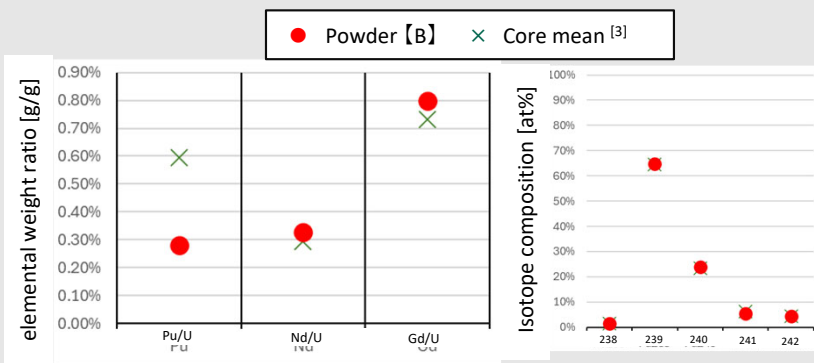
### (1) U, Pu, Nd and Gd element concentration measured with isotope dilution mass spectrometry (IDMS)

#### Focal point

Approx. 0.1g



1st Fuel debris Powder [B]



The ratio of U to Pu, Nd and Gd in the initial report <sup>[1][2]</sup>

- ✓ The ratio of Pu/U was approximately half the core mean. However, the ratios of Nd/U, Gd/U, and the Pu isotope composition, and the ratio of <sup>241</sup>Am/U<sup>※</sup> were close to the core mean, and we have examined how well this aligns with these trends while also improving the methodology.

※ At current time it is assumed that the majority of <sup>241</sup>Am was formed from the decay of <sup>241</sup>Pu

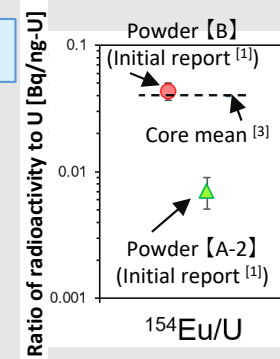
### (2) Radioactivity concentration by γ-ray/α-ray spectrometry

#### Focal point

Approx. 0.01g



1st Fuel debris Powder [A-2]



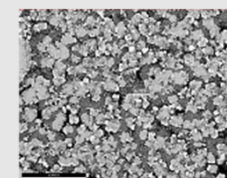
Radiation concentration comparison of the batched specimens

- <sup>154</sup>Eu/U ratio assessment example – (Initial report value <sup>[1]</sup> used)
- ✓ There was a large difference in the radiation concentration between batched specimens so the cause of this was deliberated and methodology was improved.

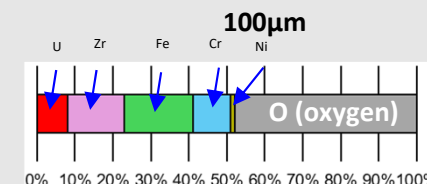
### (3) Primary element ration from SEM-EDX surface analysis

#### Focal point

Fine mixed phase



1st Fuel debris Clump [C]



SEM-EDX surface analysis semi-quantitative results for the fine mixed phase

- ✓ Even though observation results show the majority to be oxides, we examined how well this aligns with the fact that oxygen concentration values are low.

Analysis methods have been improved to address these areas for improvement

[1] JAEA, TEPCO HD, Fuel debris Sample (1st) analysis results, Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (140<sup>th</sup>), July 31, 2025, Document 3-3  
 [2] JAEA, TEPCO HD, Fuel debris Sample (1st) analysis results (update), Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (144<sup>th</sup>), November 27, 2025, Document 3-3  
 [3] Okumura et. al., Atomic Energy Society of Japan 2021 Spring Conference, 3B01.

### (1) Measuring U, Pu, Nd and Gd element concentration using isotope dilution mass spectrometry (IDMS)

#### ① Improvements to pretreatment by revising the Pu valence control process (Pu)

- ✓ The analysis flow conventionally used for spent fuel has been revised and a chemical separation/pretreatment methodology suitable for alkali fusion specimens was introduced.
- During pretreatment, the valence is adjusted in consideration of hexavalent Pu in the specimen by adjusting the added reagents, thereby achieving chemical equilibrium with the Pu in the tracer solution. ⇒ This has been reflected in the procedure

#### ② Concentration evaluation formula improvements (for U, Pu, Nd, and Gd)

- ✓ The evaluation formula for converting isotope composition to element concentration was revised.
- The density difference※ between specimens was reflected in the assessment of the tracer solution and added amount of measured specimen (fuel debris solution) used for the evaluation formula, and more strictly converted (applied) based on volume rather than mass.

※ Since highly concentrated nitric acid and a lot of alkaline flux is used during the analysis of fuel debris fuel debris, the density is higher than the tracer solution (primarily 1M nitric acid solution)

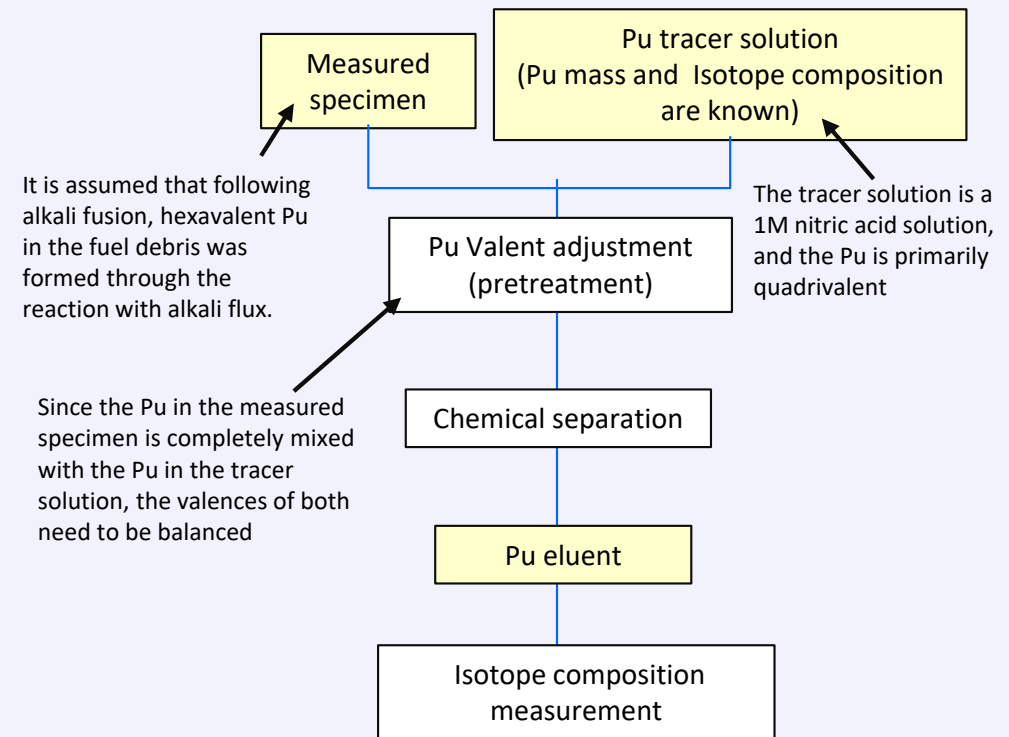


Figure Summary of Pu analysis flow using IDMS

### (2) Measuring radioactivity concentration using γ-ray/α-ray spectrometry

- ✓ Spectrum analysis methodology and analysis conditions have been re-inspected
- Efficient calibration conditions for γ-ray and α-rays when measuring fuel debris were optimized

### (3) Measuring primary element ratios from SEM-EDX surface analysis

- ✓ EDX spectrum analysis methodology and software settings were re-inspected
- Software setting conditions (stage tilt, etc.) when measuring fuel debris were optimized
- Attention was given to the accuracy of oxygen analysis results, which is inherently difficult to quantify, during assessment.

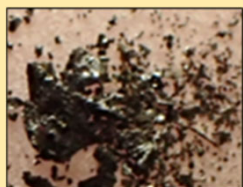
Measurements will be performed again as necessary in light of the improved analysis methodology, analysis results will be reassessed.

### Reassessed results

#### (1) Measuring U, Pu, Nd and Gd element concentration using isotope dilution mass spectrometry (IDMS)

Initial report value (Already reported<sup>[1]</sup> p.31)

|    | Analysis value [mg/100mg <sub>specimen</sub> ] |
|----|------------------------------------------------|
| U  | 29.9 ± 0.2                                     |
| Pu | 0.084 ± 0.001                                  |
| Nd | 0.098 ± 0.001                                  |
| Gd | 0.239 ± 0.002                                  |

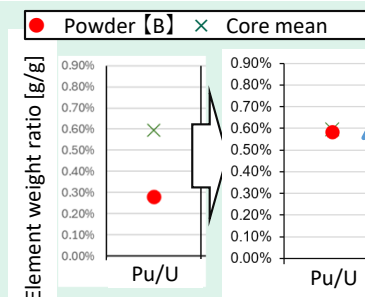


Powder [B]

After reevaluation

|    | Analysis value [mg/100mg <sub>specimen</sub> ] |
|----|------------------------------------------------|
| U  | 34.48 ± 0.64                                   |
| Pu | 0.201 ± 0.008                                  |
| Nd | 0.114 ± 0.005                                  |
| Gd | 0.294 ± 0.012                                  |

### Impact on attribute assess



The Pu/U ratio is close to the core mean, and matches other data trends for <sup>241</sup>Am, etc.

⇒ Matches the assumption that fuel was mixed in over a relatively wide area

#### (2) Measuring radioactivity concentration using γ-ray/α-ray spectrometry

Initial report value (Already reported<sup>[1]</sup> p.34)

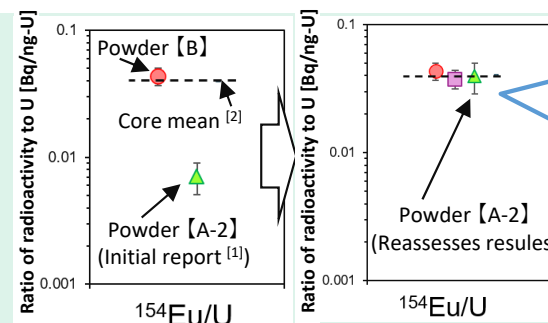
| Nuclide                              | Analysis value [MBq/g- specimen] |
|--------------------------------------|----------------------------------|
| <sup>60</sup> Co                     | 2.0 ± 0.5                        |
| <sup>125</sup> Sb                    | 2.0 ± 0.5                        |
| <sup>137</sup> Cs                    | 0.6 ± 0.2                        |
| <sup>154</sup> Eu                    | 1.8 ± 0.5                        |
| <sup>241</sup> Am                    | 2.3 ± 0.6                        |
| <sup>242</sup> Cm                    | 0.02 ± 0.02                      |
| <sup>243</sup> Cm+ <sup>244</sup> Cm | 3.5 ± 0.2                        |
| <sup>241</sup> Am+ <sup>238</sup> Pu | 11.5 ± 0.4                       |
| <sup>239</sup> Pu+ <sup>240</sup> Pu | 4.8 ± 0.3                        |



Powder [A-2]

After reevaluation

| Nuclide                              | Analysis value [MBq/g- specimen] |
|--------------------------------------|----------------------------------|
| <sup>60</sup> Co                     | 9 ± 2                            |
| <sup>125</sup> Sb                    | 6 ± 1                            |
| <sup>137</sup> Cs                    | 0.30 ± 0.09                      |
| <sup>154</sup> Eu                    | 10 ± 3                           |
| <sup>241</sup> Am                    | 13 ± 3                           |
| <sup>242</sup> Cm                    | 0.04 ± 0.03                      |
| <sup>243</sup> Cm+ <sup>244</sup> Cm | 8 ± 2                            |
| <sup>241</sup> Am+ <sup>238</sup> Pu | 25 ± 5                           |
| <sup>239</sup> Pu+ <sup>240</sup> Pu | 6 ± 1                            |



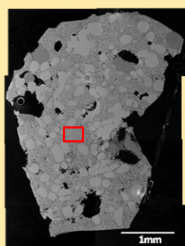
Discrepancies between batch specimens have become clear

⇒ The assessment of nuclide associations has been improved by performing an assessment in light of the non uniformity of the samples

#### (3) Measuring primary element ratios from SEM-EDX surface analysis

Initial report value (Already reported<sup>[1]</sup> p.42)

| Primary element | Ratio of primary elements [at%] |
|-----------------|---------------------------------|
| O               | 48                              |
| Cr              | 10                              |
| Fe              | 18                              |
| Ni              | 1                               |
| Zr              | 15                              |
| U               | 8                               |



Clump [C]

After reevaluation

| Primary element | Ratio of primary elements [at%] |
|-----------------|---------------------------------|
| O               | 74                              |
| Cr              | 4                               |
| Fe              | 8                               |
| Ni              | < 0.5 (very little exists)      |
| Zr              | 9                               |
| U               | 4                               |

⇒ It has become easier to determine whether the main element is a metal or an oxide, and the accuracy of the assessment has been improved through the mutual comparison of solid analysis such as TEM observation, etc.

[1] JAEA, TEPCO HD, Fuel debris Sample (1st) analysis results, Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (140<sup>th</sup>), July 31, 2025, Document 3-3  
 [2] Okumura et. al., Atomic Energy Society of Japan 2021 Spring Conference, 3B01.

- Analysis accuracy was improved through the reassessment and there are no changes to the estimates<sup>※</sup> pertaining to the fuel debris formation process, but some knowledge was reinforced.

※ Already reported assessment

- It is possible that fuel melted and mixed over a relatively wide area and structural materials from the bottom of the core were entrained
- Structural materials oxidized to an extent, but it is possible that they melted at temperatures between 1900 - 2400°C.

- Data integration and checks to confirm that differing methodology align were continually implemented in order to assess the attributes of fuel debris. During the reassessment, the knowledge obtained through this process was reflected in improvements to analysis methodology, which led to improvements in fuel debris analysis accuracy and assessment reliability. Going forward we will continue to improve the accuracy of the analysis of fuel debris that is non-uniform and has unknown composition, and revise our assessment results as necessary.

- Revisions that were made to past reports in conjunction with the reassessment are as follows:

| No. | Report                                                                                                                                                                                                        | Page | Revision details                            | Reason for the revision                                                                                                                                                                   |
|-----|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------|---------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| 1   | Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (140 <sup>th</sup> ), July 31, 2025, "Fuel debris sample (1 <sup>st</sup> ) analysis results" | p.7  | Graph revision                              | Graph revised in conjunction with their reassessment of the U analysis values from IDMS                                                                                                   |
| 2   |                                                                                                                                                                                                               | p.9  | Graph revision                              | Graph revised in conjunction with their reassessment of the U and P analysis values from IDMS                                                                                             |
| 3   |                                                                                                                                                                                                               | p.31 | Chart revision                              | Graph revised in conjunction with their reassessment of the U, Pu, Nd and Gd analysis values from IDMS                                                                                    |
| 4   |                                                                                                                                                                                                               | p.34 | Chart revision                              | Chart revised in conjunction with a reassessment of the radioactivity concentration analysis values from γ-ray spectrometry and α-ray spectrometry                                        |
| 5   |                                                                                                                                                                                                               | p.35 | Chart revision                              | Revision of the Zr/U ratio and Gd/U ratio assessment values in conjunction with the reassessment of the analysis values for U and Gd from IDMS                                            |
| 6   |                                                                                                                                                                                                               | p.42 | Chart and Graph revision                    | Chart and graph revised in conjunction with a reassessment of the SEM-EDX semi-qualitative values and U analysis values from IDMS                                                         |
| 7   | Meeting of the Secretariat of the Team for Countermeasures for                                                                                                                                                | p.19 | Graph revision and addendum added to report | Graph revised in conjunction with their reassessment of the U, Pu, Nd and Gd analysis values from IDMS, and addendum added to the report in conjunction with a revision of the Pu/U ratio |
| 8   | Decommissioning and Contaminated Water Treatment (144 <sup>th</sup> ), November 27, 2025 "Fuel debris sample (1 <sup>st</sup> ) analysis results (update)"                                                    | p.32 | Same as Chart No. 1 in this report          | Meeting materials from the 140 <sup>th</sup> meeting quoted as reference                                                                                                                  |
| 9   |                                                                                                                                                                                                               | p.34 | Same as Chart No. 2 in this report          | Meeting materials from the 140 <sup>th</sup> meeting quoted as reference                                                                                                                  |
| 10  |                                                                                                                                                                                                               | p.37 | Same as Chart No. 6 in this report          | Meeting materials from the 140 <sup>th</sup> meeting quoted as reference                                                                                                                  |

Revisions to portions of the initial report (No.1-7) noted in the reference materials

# Reference materials

### ◆ Radioactivity analysis

- In order to acquire information that will contribute to deliberations pertaining to treatment and disposal, the range of nuclides targeted for radioactivity analysis was expanded further than those analyzed in a conventional fuel debris sample.
- The nuclides targeted for analysis focused on  $\beta$ -ray emitting nuclides, for which hardly any data has been acquired from the fuel debris samples to date, while referencing the nuclides targeted for analysis in solid waste<sup>[1]</sup> (Refer to the chart on the right). In order to make effective use of the samples and in consideration of the analysis method, the first fuel debris solution (stored at the NDC) was used for the analysis.

Chart Nuclides targeted for radioactivity analysis by the NDC and measurement method

| Nuclide                                                                                                          | Measurement method                            |
|------------------------------------------------------------------------------------------------------------------|-----------------------------------------------|
| $^3\text{H}$ , $^{14}\text{C}$ , $^{63}\text{Ni}$ , $^{79}\text{Se}$                                             | Liquid scintillation counter                  |
| $^{36}\text{Cl}$ , $^{99}\text{Tc}$ , $^{90}\text{Sr}$ , $^{129}\text{I}$                                        | $\beta$ -ray spectrometer or gas-flow counter |
| $^{41}\text{Ca}$                                                                                                 | X-ray measurement                             |
| $^{93}\text{Zr}$ , $^{93}\text{Mo}$ , $^{107}\text{Pd}$                                                          | ICP-MS                                        |
| $^{126}\text{Sn}$                                                                                                | $\gamma$ -ray spectrometry (LE-Ge)            |
| $\gamma$ -ray emitting nuclide ( $^{137}\text{Cs}$ , $^{154}\text{Eu}$ , $^{241}\text{Am}$ , $^{60}\text{Co}$ )※ | $\gamma$ -ray spectrometry                    |

※ Since data for these  $\gamma$ -ray emitting nuclides was obtained from batched specimens made from the first fuel debris sample<sup>[2]</sup>, they were also subjected to additional analysis in order to deliberate differences between batched specimens

1st Fuel debris Clump [D]



External view of the first fuel debris sample received by the NDC



Solution

Stored at the NDC after obtaining element composition data<sup>[2]</sup>

Sample subjected to additional analysis

Figure Sample subjected to radioactivity analysis

### ◆ Assessment of phase conditions using SEM-EDX and XRD

- In order to further deepen our understanding of the process by which the first sample, which has a complicated shape, was formed, the phase status of locations with unique external appearances were assessed.
- Protrusions in the sample seen prior to pulverization (see the figure on the right) were chosen and the fine structure and crystalline structure/composition were compared to other regions in order to examine the differences in formation process between regions.

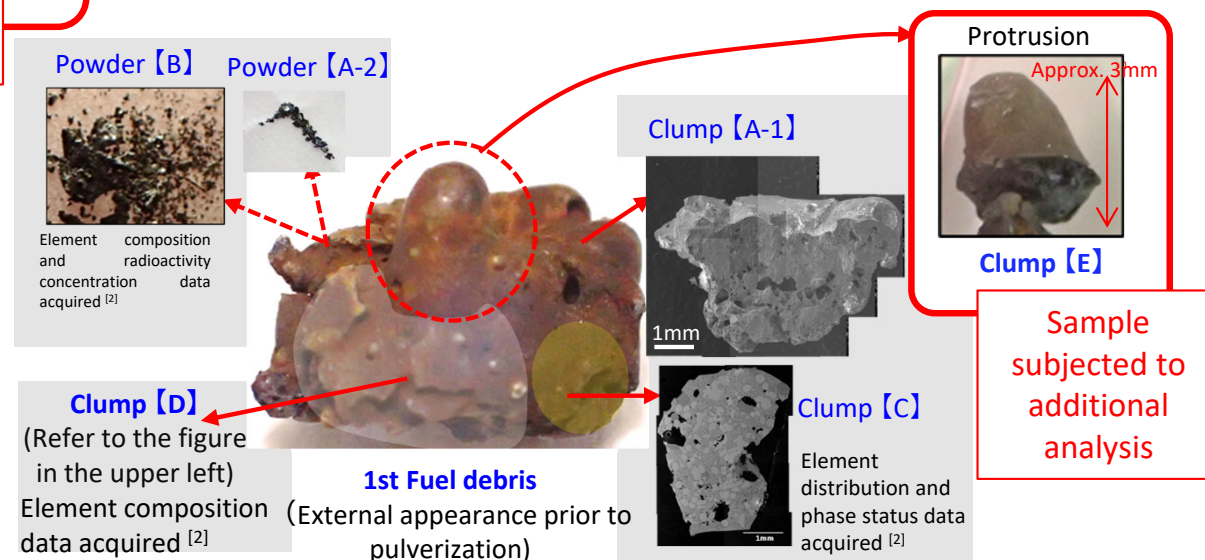
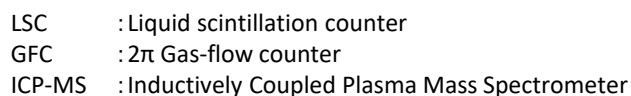


Figure 1st Fuel debris sample analysis status and additional analysis sample targets

[1] TEPCO HD, Selecting target nuclides for analysis during the analysis of solid waste from 1F, 23<sup>rd</sup> Technical Review Meeting on the Specified Nuclear Power Facility Implementation Plan, December 5, 2024, Document 2-1

[2] JAEA, TEPCO HD, Fuel debris Sample (1st) analysis results, Meeting of the Secretariat of the Team for Countermasures for Decommissioning and Contaminated Water Treatment (140th), July 31, 2025, Document 3-3

- A suitable amount of each nuclide was batched to make analysis specimens, and then subjected to pretreatment and analysis



### ◆ Radioactivity concentration

- In addition to the  $\gamma$ -ray emitting nuclide targeted for analysis to date,  $^{63}\text{Ni}$  and  $^{99}\text{Tc}$  were also detected. (Chart 1)
- In regards to the concentration of  $\gamma$ -ray emitting nuclides, for which data has already been obtained from other batch specimens of the first fuel debris sample,  $^{154}\text{Eu}$ , which is thought to be easily associated with U, was set as a benchmark nuclide for comparison. (Figure 1)
- The difference in concentrations of  $^{241}\text{Am}$  between samples is small, and it is assumed that it was easy for the nuclide to associate with U during formation of the sample. Whereas the differences in the concentration of  $^{90}\text{Sr}$  between samples were also small, it was only slightly lower than the core mean.
- Differences in the concentration of  $^{60}\text{Co}$  between samples was seen. There were also differences in the concentrations of structural material elements ( $\text{Fe}+\text{Cr}+\text{Ni}$ ) between batched specimens, and it is thought that it was also easy for this nuclide to associate. (The concentration of  $^{60}\text{Co}$  is also noted using Ni as a benchmark nuclide for the sake of reference)
- $^{137}\text{Cs}$  originated from fuel, but it is assumed that the majority volatilized during the melting process, and a minuscule amount remains in a non-uniform manner within the sample.

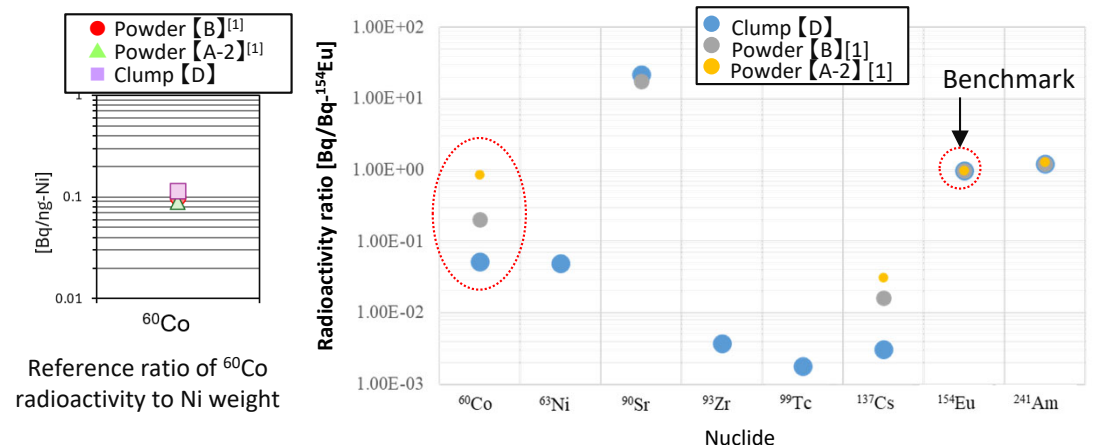


Figure 1 Radioactivity ratio using  $^{154}\text{Eu}$  in the first fuel debris sample as a benchmark

### 1st Fuel debris Clump [D]



Chart 1 Radioactivity concentration assessment results (values on the date of measurement)

Expanded uncertainty: k=2

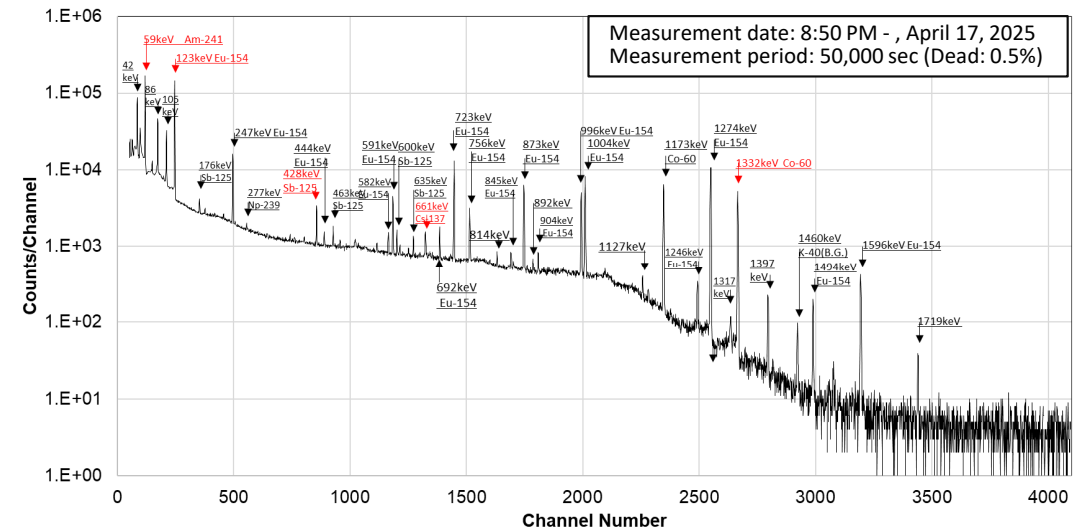
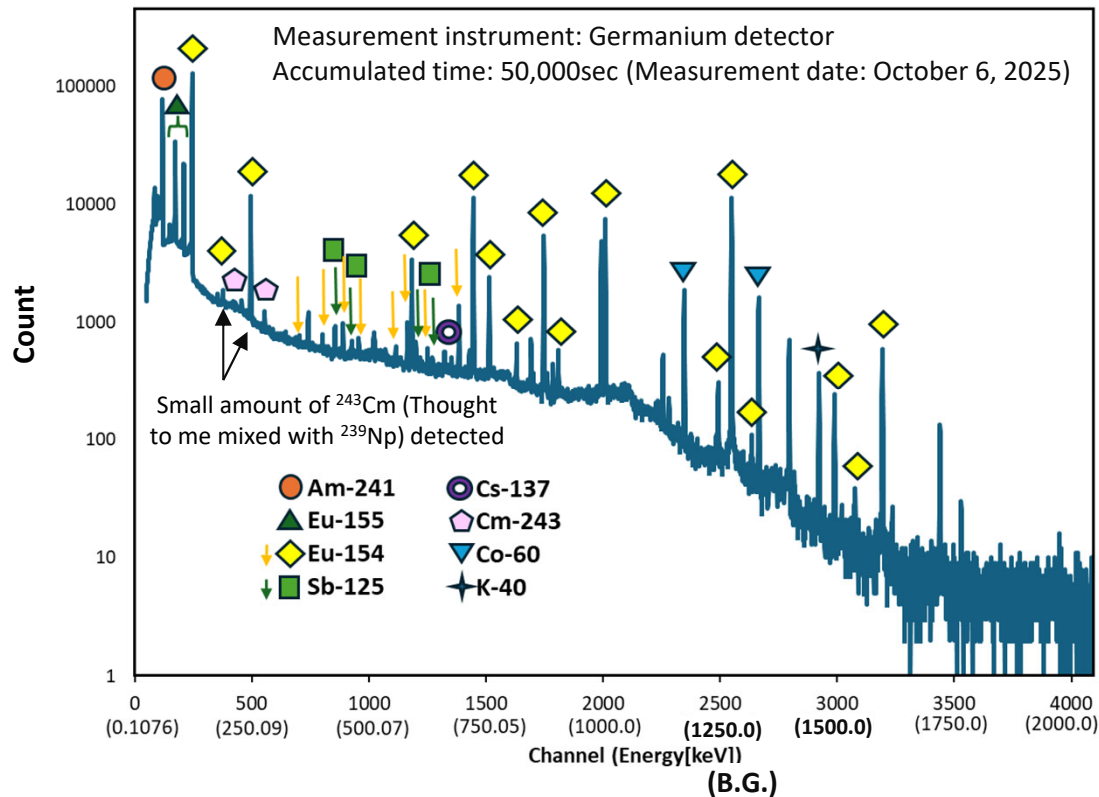
| Nuclide           | Radioactivity concentration [MBq/g specimen] | Lower limit of detection | Measurement date |
|-------------------|----------------------------------------------|--------------------------|------------------|
| $^3\text{H}$      | ND                                           | 0.030                    | 2026/4/21        |
| $^{14}\text{C}$   | ND                                           | 0.015                    | 2026/4/21        |
| $^{36}\text{Cl}$  | ND                                           | $4.4 \times 10^{-4}$     | 2026/4/2         |
| $^{41}\text{Ca}$  | ND                                           | 0.38                     | 2026/5/18        |
| $^{60}\text{Co}$  | $0.86 \pm 0.10$                              | 0.015                    | 2025/10/4        |
| $^{63}\text{Ni}$  | $0.77 \pm 0.16$                              | 0.11                     | 2026/4/3         |
| $^{79}\text{Se}$  | ND                                           | 0.018                    | 2026/4/16        |
| $^{90}\text{Sr}$  | $(3.31 \pm 0.30) \times 10^2$                | 0.25                     | 2026/4/22        |
| $^{93}\text{Zr}$  | $0.0577 \pm 0.0066$                          | $9.5 \times 10^{-4}$     | 2026/5/8         |
| $^{93}\text{Mo}$  | ND                                           | 0.011                    | 2026/5/18        |
| $^{99}\text{Tc}$  | $0.0273 \pm 0.0024$                          | $4.7 \times 10^{-4}$     | 2026/4/2         |
| $^{107}\text{Pd}$ | ND                                           | $4.8 \times 10^{-5}$     | 2026/5/12        |
| $^{126}\text{Sn}$ | ND                                           | $1.4 \times 10^{-3}$     | 2026/4/7         |
| $^{129}\text{I}$  | ND                                           | $4.9 \times 10^{-3}$     | 2026/4/14        |
| $^{137}\text{Cs}$ | $0.0435 \pm 0.0061$                          | $5.1 \times 10^{-4}$     | 2026/4/17        |
| $^{154}\text{Eu}$ | $15.9 \pm 2.5$                               | 0.039                    | 2025/10/4        |
| $^{241}\text{Am}$ | $18.8 \pm 2.9$                               | 0.12                     | 2025/10/4        |

ND: Not detected

Foundational data was attained that will enable us to enhance analysis as well as deliberate the mass relationship between various radionuclides as well as which nuclides should serve as benchmarks when doing so. Going forward we will continue to deliberate the nuclides targeted for analysis while accumulating data.

### Reference $\gamma$ -ray spectrometry results Clump [D] solution

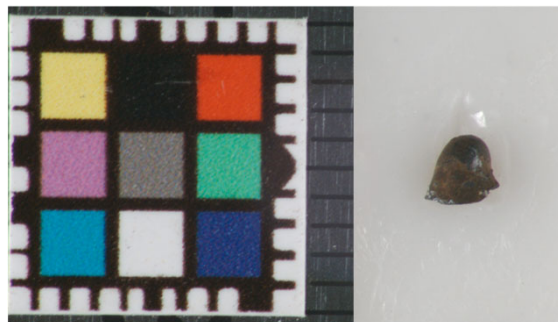
■  $^{241}\text{Am}$ ,  $^{154}\text{Eu}$ ,  $^{155}\text{Eu}$ ,  $^{125}\text{Sb}$ ,  $^{137}\text{Cs}$ ,  $^{60}\text{Co}$  were the primary nuclides detected.



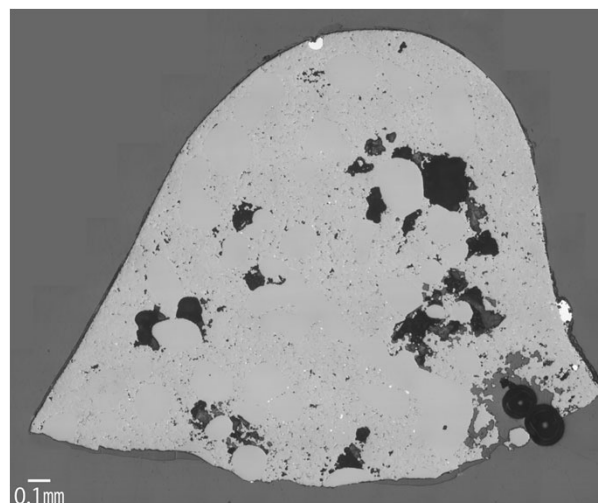
Reference: 1st Fuel debris  
Powder [B] solution  $\gamma$ -ray spectrum

Figure 1st Fuel debris Clump [D] solution  $\gamma$ -ray spectrum

### ◆ Cross-sectional observation results

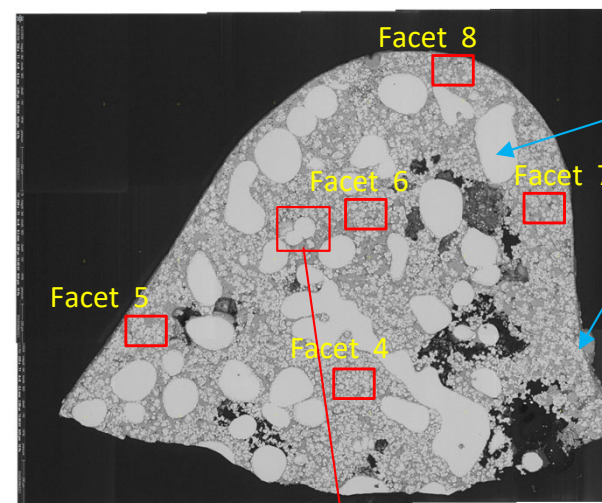


External view of the sample received by the NDC



1mm

Optical microscope image



1mm

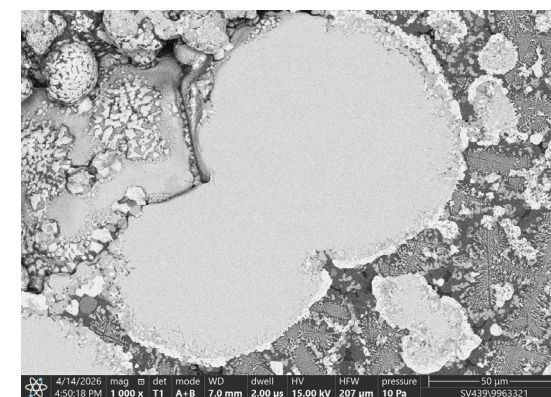
SEM image (BSE image)

Zr-U-O

Fe-Ni metal (Very little in the cross-section)

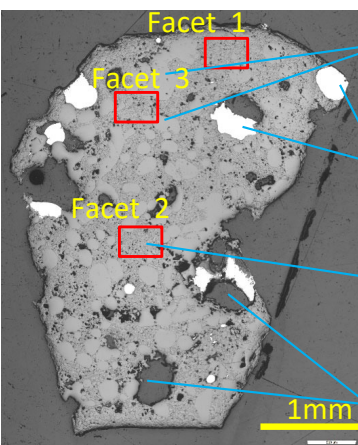
 $Ni/(Fe+Ni)-0.7$ 

Zr-U-O phase



Zr/U-1.1

50μm



Reference Clump [C] Optical microscope image of cross-section

Zr-U-O phase

- The atomic ratio of Zr/U is approx. 2 (Almost constant regardless of grain)

Fe-Ni metal phase

- The atomic ratio of Fe/Ni is approx. 1~3 (Different depending on grain)

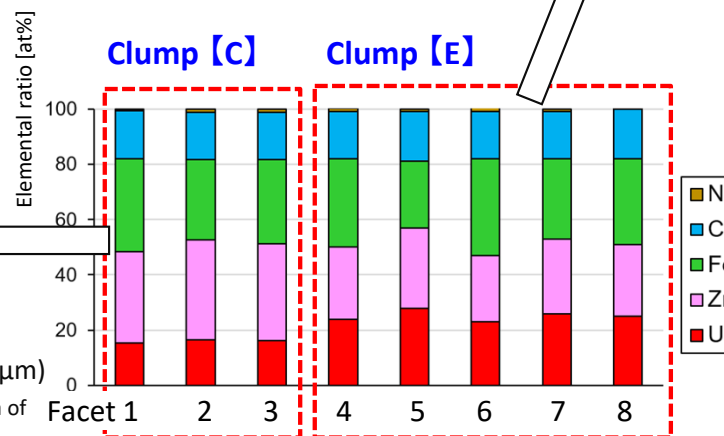
Fine mixed phase

- U-Zr-O, Zr-U-O, Fe-Cr-O, Fe-O mixed phase

Void

(Several μm~several 100 μm)

- Approximately 20% of the area of the cross-section



Fine mixed phase SEM-EDX Surface analysis results

- The protrusions on the first fuel debris sample (Clump [E]) showed the same trends as other areas when it comes to cross-section structure and the breakdown of Fe+Cr+Ni, but differences were also seen, such as a lower Zr/U ratio than that found in other regions.

### ◆ Cross-section observation results

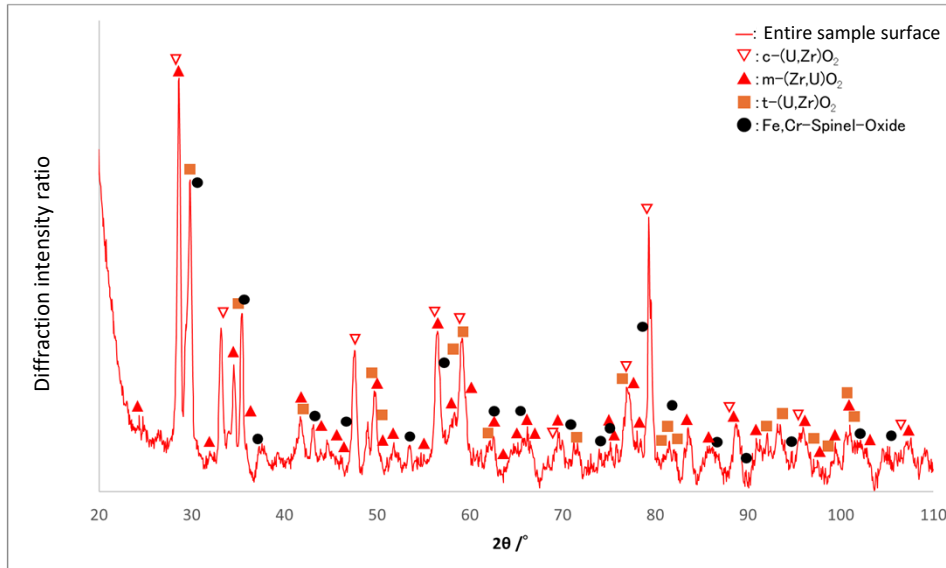


Figure 1 XRD measurement results for the entire cross section

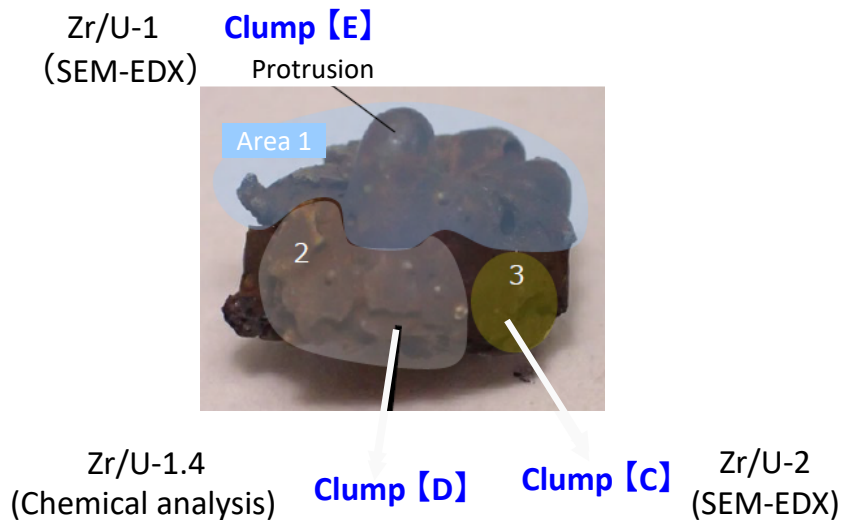
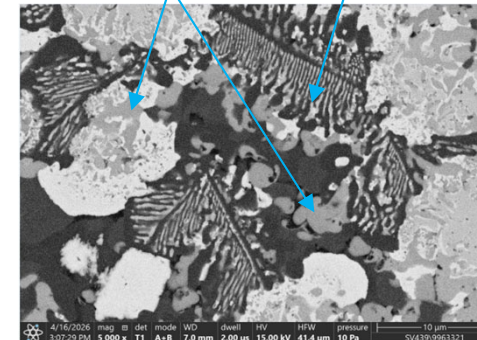
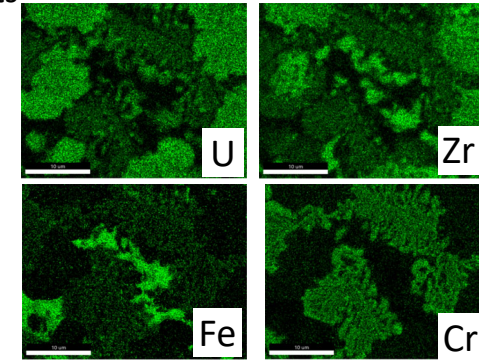


Figure 3 Composition of each region of the first fuel debris sample

Phase with a high concentration of Zr  
Dendritic deposits



10μm SEM image



10μm Primary element distribution

Figure 2 SEM-EDX analysis results for the fine mixed region

- XRD results from the cross-section identified cubic crystals (U,Zr)O<sub>2</sub> also seen through conventional TEM observation, as well as tetragonal crystals (Zr,U)O<sub>2</sub>, Fe-Cr spinel phases, and monoclinic crystals (Zr,U)O<sub>2</sub> (Figure 1)
- Since SEM observations also found phases with high concentrations of Zr and dendritic deposits in the fine mixed phase (Figure 2), it is possible that the tetragonal crystals (Zr,U)O<sub>2</sub> and monoclinic crystals (Zr,U)O<sub>2</sub> identified with XRD correspond to these phases.
- It is assumed that these regions solidified from the same molten material as other regions in the sample, but since the composition is slightly different depending on region (Figure 3) it is possible that solidification occurred at a different time.

- In order to expand the nuclides targeted for analysis, which will contribute to deliberating treatment and disposal, and also further deepen our understanding about the process through which the samples formed, the first fuel debris sample was subjected to additional radioactivity concentration and phase status analyses.
- During the additional radioactivity concentration analysis, nuclides that had not previously been targeted were added, and various analysis methods were applied. As a result, in addition to  $\gamma$ -ray emitting nuclides,  $\beta$ -ray emitting nuclides, such as  $^{63}\text{Ni}$  and  $^{99}\text{Tc}$ , were also detected.
  - Foundational data was obtained that will enable us to enhance analysis as well as deliberate the mass relationship between various radionuclides as well as which nuclides should serve as benchmarks when doing so.
  - During sample analysis in the future we will continue to deliberate the nuclides targeted for analysis while accumulating data.
- During phase status analysis, SEM-EDX was used to observe the cross-section, and XRD was used to assess the crystalline structure of protrusions that had not yet been analyzed. The results showed that whereas the structural phases of the cross section were the same as other regions, the ratio of Zr/U was lower than other regions.
  - It is assumed that these regions solidified from the same molten material as other regions in the sample, but since the composition is slightly different depending on region it is possible that solidification occurred at a different time.
  - Deliberation of the formation process will continue and such information will be reported at academic conferences and in academic papers

Chart Elements and nuclides focused on during solution analysis

|                             | Unit                                                                             | Focused element/nuclide                                                                                                                                                                                                                                                                                                                                                                                                                                                      | Analysis method                                                                                                           |
|-----------------------------|----------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------|
| Element content             | mg/100mg <sub>specimen</sub><br>(Weight of each element per 100mg of the sample) | <ul style="list-style-type: none"> <li>• U (Fuel) , Zr (Cladding tubes/CB)</li> <li>• Fe, Cr, Ni (carbon steel, SUS, etc.)</li> <li>• Si, Ca, Al, Mg (Instrument materials, seawater, concrete, etc.)</li> <li>• B (<sup>10</sup>B), Gd (<sup>155</sup>Gd, <sup>157</sup>Gd) (Neutron poison)</li> <li>• Pb, Zn, Ti (Shielding, paint, etc.)</li> <li>• Mo, Sb (Grease, instruments, FP, etc.)</li> <li>• Other elements found during qualitative analysis or SEM</li> </ul> | <ul style="list-style-type: none"> <li>• ICP-AES</li> <li>• ICP-MS</li> <li>• IDMS</li> </ul>                             |
| Isotope ratio               | Molar ratio [-]                                                                  | <ul style="list-style-type: none"> <li>• U isotope : <math>^{235}\text{U}/^{238}\text{U}</math> ration or <math>^{235}\text{U}/\text{U}_{\text{total}}</math> ratio<br/> <math display="block">\text{U}_{\text{total}} = ^{234}\text{U} + ^{235}\text{U} + ^{236}\text{U} + ^{238}\text{U}</math> </li> </ul>                                                                                                                                                                | <ul style="list-style-type: none"> <li>• TIMS</li> <li>• ICP-MS</li> </ul>                                                |
|                             |                                                                                  | <ul style="list-style-type: none"> <li>• Pu isotope</li> <li>• Nd isotope (Burn rate index)</li> <li>• Gd isotope</li> </ul>                                                                                                                                                                                                                                                                                                                                                 | <ul style="list-style-type: none"> <li>• TIMS (The mass of the element was assessed using IDMS)</li> </ul>                |
| Radioactivity concentration | MBq/g<br>(Radioactivity of each nuclide per sample weight)                       | <ul style="list-style-type: none"> <li>• γ-ray emitting nuclide</li> <li>• α-ray emitting nuclide</li> <li>• <sup>90</sup>Sr</li> </ul>                                                                                                                                                                                                                                                                                                                                      | <ul style="list-style-type: none"> <li>• γ-ray spectrometry</li> <li>• α-ray spectrometry</li> <li>• ICP-MS/MS</li> </ul> |

### ◆ Solution analysis at the JAEA Nuclear Science Research Institute (1/3)

○ **Element content** → Refer to the chart on the right

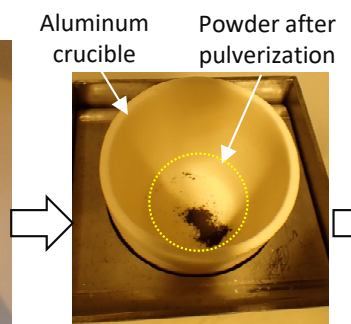
#### Analysis methods (summary)

- 1) Powder B specimen received by the JAEA Nuclear Science Research Institute, was pulverized for 30 minutes in a tungsten carbide (WC) vibratory ball mill.
- 2) The produced powder specimen was melted in an aluminum crucible with alkali flux<sup>※</sup>, and the resulting melted product was dissolved in heated nitric acid.  
<sup>※</sup> Alkali fusion: By heating and melting fuel debris, which is difficult to dissolve in acid, along with alkali flux (sodium peroxide), and converting it into a substance that can be easily dissolved in acid, we can obtain a uniform solution with no undissolved residue.
- 3) The total volume of the solution is visually inspected and diluted to volume of 250mm
- 4) The mass of elements in the solution is measured using the calibration curve method with ICP-AES, and the element content of the sample is assessed by deducting the weight of the specimen under test (43.5mg). Since the solution contains a lot of sodium originating from the alkali flux, during ICP-AES measurement, the matrix matching method is applied by which the same amount of sodium measured in the specimen is added to the control specimen.
- 5) In regards to U and Pu, each element in the solution obtained in 3) above was separated using UTEVA<sup>®</sup> resin and anion exchange resin, and the element volume of the obtained separated solution was measured using IDMS.

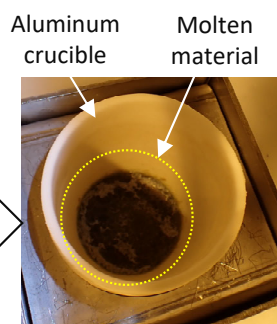
#### Powder B



Specimen received by JAEA Ooarai Nuclear Engineering Research



The powder produced with the ball mill is added to an aluminum crucible



Melted product after alkali fusion



Solution produced from heated nitric acid

Figure Alkali fusion treatment

### Chart Element content assessment results

Weight of the specimen under test: 43.5±0.4mg. Expanded uncertainty of k=2

| Element | Element content <sup>※1</sup><br>[mg/100mg specimen] | Notes                                                   |
|---------|------------------------------------------------------|---------------------------------------------------------|
| U       | 42.82 ± 0.78                                         | Measurement with IDMS                                   |
| Zr      | 23 ± 2                                               |                                                         |
| Fe      | 4.4 ± 0.3                                            |                                                         |
| Cr      | 4.3 ± 0.2                                            |                                                         |
| Ni      | 0.16 ± 0.02                                          |                                                         |
| B       | 0.08 ± 0.01                                          |                                                         |
| Si      | 0.7 ± 0.06                                           | Reference value <sup>※2</sup>                           |
| Ca      | 0.12 ± 0.01                                          | Corrected mass of Ca in the flux                        |
| Al      | N/A                                                  | Not included since it originates from the crucible      |
| Mg      | 0.12 ± 0.02                                          | Reference value <sup>※2</sup>                           |
| Na      | N/A                                                  | Not included since it originates from the flux          |
| S       | 0.11 ± 0.02                                          | Reference value <sup>※2</sup>                           |
| Ti      | 0.03 ± 0.01                                          |                                                         |
| Mn      | 0.41 ± 0.03                                          |                                                         |
| Cu      | 0.11 ± 0.03                                          | Reference value <sup>※2</sup>                           |
| Zn      | 0.08 ± 0.02                                          | Reference value <sup>※2</sup>                           |
| Mo      | <0.01                                                |                                                         |
| Ru      | <0.03                                                |                                                         |
| Pd      | 0.10 ± 0.02                                          |                                                         |
| Ag      | 0.05 ± 0.01                                          |                                                         |
| Sn      | <0.02                                                |                                                         |
| Pu      | 0.237 ± 0.010                                        | Measurement with IDMS                                   |
| Nd      | 0.15 ± 0.02                                          |                                                         |
| Gd      | 0.35 ± 0.02                                          |                                                         |
| Ce      | 0.11 ± 0.04                                          |                                                         |
| Co      | 0.35 ± 0.03                                          | Assumed to originate from the WC used for pulverization |
| W       | 4.1 ± 0.3                                            | Assumed to originate from the WC used for pulverization |

※1 Analysis value uncertainty is a synthesis of the uncertainty associated with ICP measurement, specimen weighing, diluting to volume, calibration standard solution and dilution, and is represented as expanded uncertainty multiplied by a coverage factor (k=2)

※2 The concentration ratio of the procedural blank/solution exceeds 0.1 and is a reference value.

### ◆ Solution analysis at the JAEA Nuclear Science Research Institute (2/3)

○ **Isotope ratios (U, Pu, Gd and Nd)** → Refer to the chart on the right

#### Analysis method (summary)

- 1) A suitable amount of the solution prepared in “Element content” on the previous page was batched and an eluent for each element was prepared using UTEVA® resin and anion exchange resin.
- 2) The prepared eluent was used for TIMS isotope ratio (Ex:  $^{234}\text{U}/^{238}\text{U}$ , etc.) measurement. During measurement, the total evaporation method※ that does not require mass fractionation correction was employed.  
 ※ Total evaporation method: The specimen is painted onto a filament and totally evaporated to detect the isotope ions that are generated during this process. Mass fractionation correction is not required.
- 3) The following formulas were used to calculate isotope composition from the mean of the isotope ratio measurements ( $n = 3$ )

Isotope composition calculation formulas (U used as an example)

$$\left\{ \begin{array}{ll} ^{234}\text{U}/\text{U}[\text{at}\%] = R_{234/238} / A \times 100 & R_{234/238} : ^{234}\text{U}/^{238}\text{U} \text{ isotope ratio (Measurement value)} \\ ^{235}\text{U}/\text{U}[\text{at}\%] = R_{235/238} / A \times 100 & R_{235/238} : ^{235}\text{U}/^{238}\text{U} \text{ isotope ratio (Measurement value)} \\ ^{236}\text{U}/\text{U}[\text{at}\%] = R_{236/238} / A \times 100 & R_{236/238} : ^{236}\text{U}/^{238}\text{U} \text{ isotope ratio (Measurement value)} \\ ^{238}\text{U}/\text{U}[\text{at}\%] = 1 / A \times 100 & \end{array} \right.$$

$$A \equiv R_{234/238} + R_{235/238} + R_{236/238} + 1$$

### Chart TIMS My stove composition analysis results

Weight of the specimen under test:  $43.5 \pm 0.4\text{mg}$ . Expanded uncertainty of  $k=2$

| Element | Nuclide           | isotope ratio ※ <sup>1</sup> [at%] |
|---------|-------------------|------------------------------------|
| U       | $^{234}\text{U}$  | $0.027 \pm 0.001$                  |
|         | $^{235}\text{U}$  | $2.061 \pm 0.001$                  |
|         | $^{236}\text{U}$  | $0.314 \pm 0.001$                  |
|         | $^{238}\text{U}$  | $97.598 \pm 0.001$                 |
| Pu      | $^{238}\text{Pu}$ | $1.264 \pm 0.009$                  |
|         | $^{239}\text{Pu}$ | $67.159 \pm 0.008$                 |
|         | $^{240}\text{Pu}$ | $22.763 \pm 0.003$                 |
|         | $^{241}\text{Pu}$ | $5.072 \pm 0.006$                  |
|         | $^{242}\text{Pu}$ | $3.743 \pm 0.002$                  |
| Nd      | $^{142}\text{Nd}$ | $0.40 \pm 0.01$                    |
|         | $^{143}\text{Nd}$ | $21.18 \pm 0.01$                   |
|         | $^{144}\text{Nd}$ | $31.93 \pm 0.02$                   |
|         | $^{145}\text{Nd}$ | $17.16 \pm 0.01$                   |
|         | $^{146}\text{Nd}$ | $16.51 \pm 0.01$                   |
|         | $^{148}\text{Nd}$ | $8.81 \pm 0.01$                    |
|         | $^{150}\text{Nd}$ | $4.02 \pm 0.01$                    |
| Gd      | $^{152}\text{Gd}$ | $0.11 \pm 0.01$                    |
|         | $^{154}\text{Gd}$ | $2.05 \pm 0.01$                    |
|         | $^{155}\text{Gd}$ | $2.23 \pm 0.01$                    |
|         | $^{156}\text{Gd}$ | $32.96 \pm 0.03$                   |
|         | $^{157}\text{Gd}$ | $1.24 \pm 0.01$                    |
|         | $^{158}\text{Gd}$ | $39.69 \pm 0.01$                   |
|         | $^{160}\text{Gd}$ | $21.73 \pm 0.02$                   |

※<sup>1</sup> Analysis value uncertainty was synthesized in accordance with the law of propagation errors during isotope composition calculation using the standard deviation from three repeated measurements, and is represented as expanded uncertainty multiplied by a coverage factor ( $k=2$ )

※<sup>2</sup> Measurement period: U・Pu: May 2026, Nd/Gd: July 2026

### ◆ Solution analysis at the JAEA Nuclear Science Research Institute (3/3)

#### ○ Radioactivity concentration → Refer to the chart on the right

##### Analysis method (summary)

- $\gamma$ -ray emitting nuclides were measured by taking a 0.1mL of the solution (250mL) prepared in “Element content” on the previous pages, putting it in a 6mL vial with 2.9mL of nitric acid, and subjecting it to  $\gamma$ -ray spectrometry using a high purity germanium detector (Refer to the chart below)
- $^{241}\text{Am}$  and  $^{244}\text{Cm}$  were measured by separating Am and Cm from the obtained solution with TRU resin, and measuring the radioactivity from the obtained separated solution with  $\alpha$ -ray spectrometry.
- $^{90}\text{Sr}$  was measured by taking a portion of the obtained solution and adding a stable isotope Sr as a carrier, after which Sr resin was used to separate Sr and ICP-MS/MS Was used to measure the amount of  $^{90}\text{Sr}$  in the obtained solution.

### Chart Radioactivity concentration assessment results

Weight of the specimen under test:  $43.5 \pm 0.4\text{mg}$ . Expanded uncertainty of  $k=2$

| Analysis method            | Nuclide                          | Radioactivity concentration※ <sup>1</sup><br>[MBq/g specimen] |
|----------------------------|----------------------------------|---------------------------------------------------------------|
| $\gamma$ -ray spectrometry | $^{60}\text{Co}$                 | $0.11 \pm 0.07$                                               |
|                            | $^{125}\text{Sb}$                | $<0.30$                                                       |
|                            | $^{134}\text{Cs}$                | $<0.11$                                                       |
|                            | $^{137}\text{Cs}$                | $0.32 \pm 0.10$                                               |
|                            | $^{154}\text{Eu}$                | $12 \pm 2$                                                    |
|                            | $^{155}\text{Eu}$                | $3.3 \pm 0.6$                                                 |
|                            | $^{241}\text{Am}$                | $17 \pm 2$                                                    |
| $\alpha$ -ray spectrometry | $^{241}\text{Am}$                | $20 \pm 2$                                                    |
|                            | $^{243}\text{Am}$                | $<1.3$                                                        |
|                            | $^{244}\text{Cm}$ ※ <sup>2</sup> | $7.9 \pm 0.8$                                                 |
| ICP-MS/MS                  | $^{90}\text{Sr}$                 | $(1.7 \pm 0.5) \times 10^2$                                   |

※<sup>1</sup> Values as of the measurement date. Unity is a synthesis of the uncertainty pertaining to calibration, counting,  $\gamma$ -ray emission rate, batching, weighing, diluting to volume, and measurement system fluctuations, and is represented as expanded uncertainty multiplied by a coverage factor ( $k=2$ ).

The dates for each analysis method are as follows:

- $\gamma$ -ray spectrometry : June 12, 2026
- $\alpha$ -ray spectrometry : May 18, 2026
- ICP-MS/MS( $^{90}\text{Sr}$ ) : April 8, 2026

※<sup>2</sup>  $^{244}\text{Cm}$  was solely assessed under the assumption that the amount of radioactivity from  $^{244}\text{Cm}$  is sufficiently large even though the  $^{243}\text{Cm}$  peak cannot be separated using  $\alpha$ -ray spectrometry

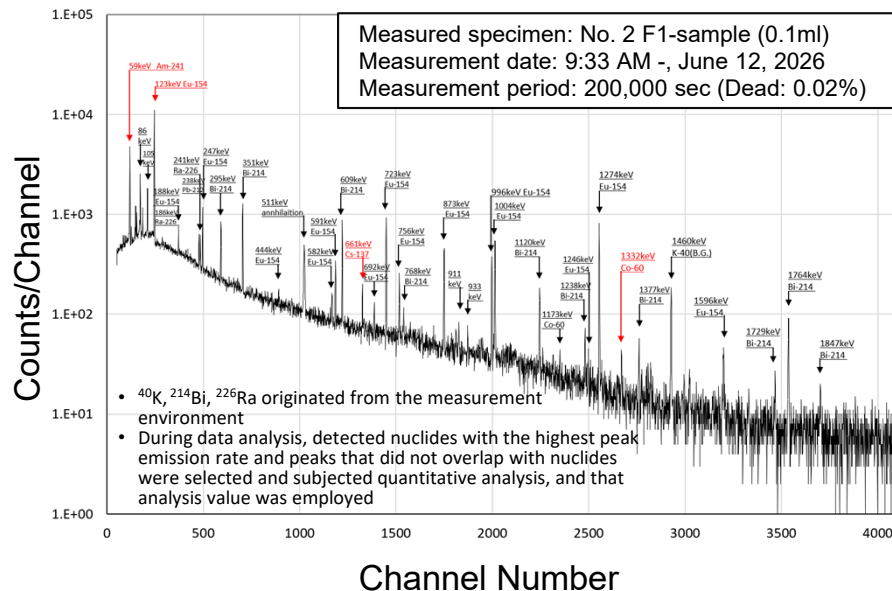


Figure  $\gamma$ -ray spectrum of the solution

### ◆ JAEA Ooarai Nuclear Engineering Research Solution analysis (1/2)

#### ○ Element content → Chart 1

#### Analysis method (summary)

- 1) The analysis specimen (Chunk A-2:11.9mg) was dissolved in heated nitric acid (a very small amount of hydrofluoric acid was also added).
- 2) The solution was filtered through a PC membrane filter (pore diameter: 0.45μm) and the residue on top of the filter was recovered while the filtered liquid was diluted to volume to prepare the solution.
- 3) The calibration curve method was used with ICP-MS to measure the mass of the targeted nuclide in the obtained solution, and nuclide mass was converted to element mass using fuel composition<sup>[1]</sup> and natural isotope composition as references.
- 4) Since approximately 8% of the input mass ended up as undissolved residue (primarily Fe-Cr oxide), the element content was assessed using SEM-WDX/EDX and the mass of elements contained in the undissolved residue was estimated.
- 5) The element content of the sample was assessed by combining the element mass analysis value for the solution with estimated element mass in the undissolved residue.

Chunk A-2



Figure Dissolving with nitric acid and a small amount of hydrofluoric acid

Chart 1 Element content analysis values  
Solution (ICP-MS) + Residue (SEM-WDX/EDX)

Weight of the specimen under test: 11.9mg. Expanded uncertainty of k=2

| Element          | Element content <sup>※1</sup><br>[mg/100mg specimen] | Notes<br>(Measured mass <sup>※2</sup> etc.)     |
|------------------|------------------------------------------------------|-------------------------------------------------|
| U                | 43.2 ± 0.9                                           | Atomic mass: Total of 234, 235, 236, 238        |
| Zr               | 23 ± 2                                               | Measured mass: 90                               |
| Fe               | 9.8 ± 0.6                                            | Measured mass: 57                               |
| Cr               | 1.52 ± 0.08                                          | Measured mass: 52                               |
| Ni               | 0.2 ± 0.1                                            | Measured mass: 60                               |
| Si               | 0.2 ± 0.1                                            | Only detected in residue                        |
| Ca               | 2 ± 2                                                | Measured mass: 44. Below the quantitative limit |
| Al               | 0.07 ± 0.03                                          | Only detected in residue                        |
| Mg               | 0.1 ± 0.1                                            | Only detected in residue                        |
| Ti               | 0.10 ± 0.01                                          | Measured mass: 47                               |
| Mn               | 0.38 ± 0.04                                          | Measured mass: 55                               |
| Mo <sup>※3</sup> | 0.02 ± 0.03                                          | Measured mass: 97                               |
| Nd <sup>※4</sup> | 0.13 ± 0.01                                          | Measured mass: 146                              |
| Gd <sup>※4</sup> | 0.4 ± 0.3                                            | Measured mass: 157                              |

- ※1 Analysis value uncertainty is a synthesis of the uncertainty associated with ICP measurement, specimen weighing, diluting to volume of the solution, calibration standard solution and dilution, and is represented as expanded uncertainty multiplied by a coverage factor (k=2).
- ※2 Mass numbers that have no isobars or mass numbers that have been impacted very little by isobars based on the count before and after were selected.
- ※3 Mo is a reference value since it is assumed that the isotope composition differs from the natural isotope composition
- ※4 Nd and Gd were converted to element mass using fuel composition since the isotope ratio was close to the fuel composition of Unit 2

### ◆ JAEA Ooarai Nuclear Engineering Research Solution analysis (2/2)

#### ○ Isotope ratio (U) → Refer to Chart 1

- The nuclide mass of  $^{234}\text{U}$ ,  $^{235}\text{U}$ ,  $^{236}\text{U}$  and  $^{238}\text{U}$  in the solution batched and diluted from the original solution was measured using the ICP-MS and the calibration curve method, and calculated as a ratio of the total of these isotopes.

#### ○ Radioactivity concentration

- γ-ray spectrometry → Refer to Chart 2
  - $^{60}\text{Co}$ ,  $^{125}\text{Sb}$ ,  $^{137}\text{Cs}$ ,  $^{154}\text{Eu}$  and  $^{241}\text{Am}$  were detected in the solution. (Figure 1 above. The same nuclides as those in the non-destructive analysis results were detected)
  - The same nuclides were detected in the undissolved residue, but the relative intensity of  $^{60}\text{Co}$  was high (Figure 1 below)

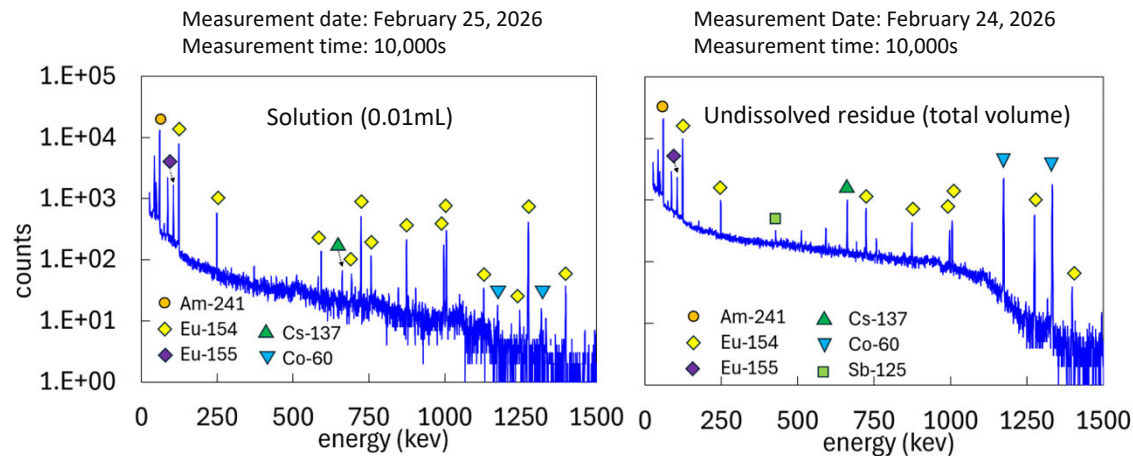


Figure 1 γ-ray spectrum Left: Solution Right: Undissolved residue

### Chart 1 U Isotope composition analysis results

Expanded uncertainty of k=2

| Nuclide          | Isotope composition ※ <sup>1</sup> [at%] |
|------------------|------------------------------------------|
| $^{234}\text{U}$ | $0.027 \pm 0.001$                        |
| $^{235}\text{U}$ | $2.044 \pm 0.004$                        |
| $^{236}\text{U}$ | $0.29 \pm 0.002$                         |
| $^{238}\text{U}$ | $97.6 \pm 0.2$                           |

※<sup>1</sup> Ratio of U =  $^{234}\text{U} + ^{235}\text{U} + ^{236}\text{U} + ^{238}\text{U}$

Analysis value uncertainty is a synthesis of the uncertainty associated with the control liquid and calibration curve method, and is represented as expanded uncertainty multiplied by a coverage factor (k=2).

### Chart 2 Radioactivity concentration assessment results

Weight of the specimen under test: 11.9mg. Expanded uncertainty of k=2

| Nuclide           | Radioactivity concentration ※ <sup>1</sup> [MBq/g specimen] |
|-------------------|-------------------------------------------------------------|
| $^{60}\text{Co}$  | $0.11 \pm 0.03$                                             |
| $^{125}\text{Sb}$ | <0.3                                                        |
| $^{134}\text{Cs}$ | <0.2                                                        |
| $^{137}\text{Cs}$ | $0.19 \pm 0.06$                                             |
| $^{154}\text{Eu}$ | $14 \pm 4$                                                  |
| $^{155}\text{Eu}$ | $4 \pm 1$                                                   |
| $^{241}\text{Am}$ | $20 \pm 5$                                                  |

## Observation location selection

【External appearance attributes used to ascertain the positional relationship】

$p_1 - p_3$  : Pit,  $r_1$  : Ridge

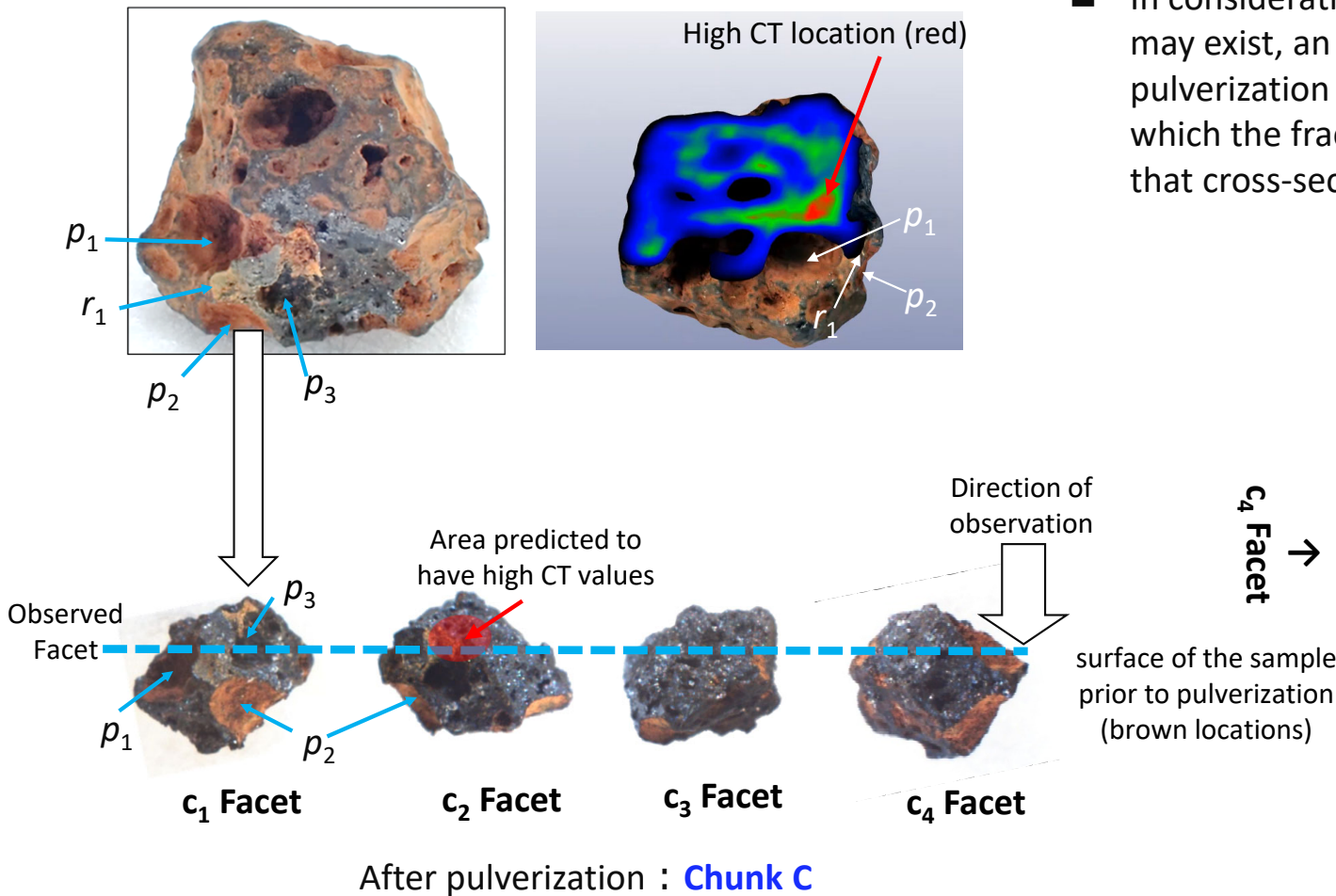


Figure 1 General positional relationships of the second fuel debris sample prior to and after pulverization

- **Chunk C**, for which the positional relationship of the sample was relatively easy to ascertain prior to and after pulverization, was used for cross-sectional observation. (Figure 1)
- In consideration of the possibility that unmelted fuel ( $\text{UO}_2$ ) may exist, an X-ray CT scan of the sample prior to pulverization was used to identify high CT locations, along which the fracture line of the cross-section was created, and that cross-section was observed (Figure 2)

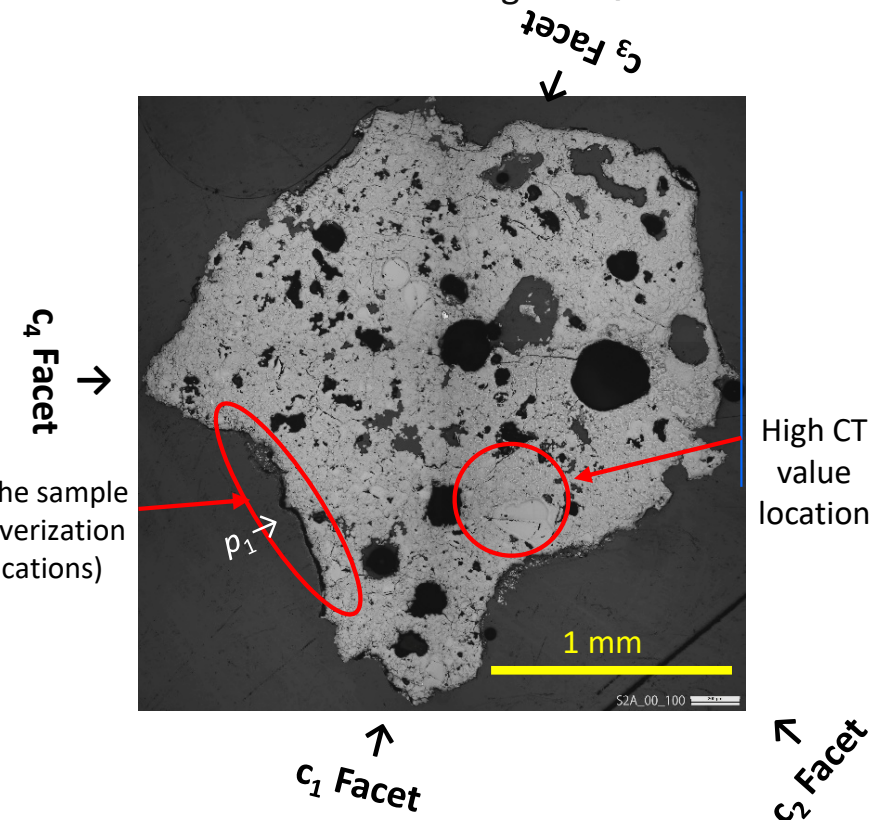
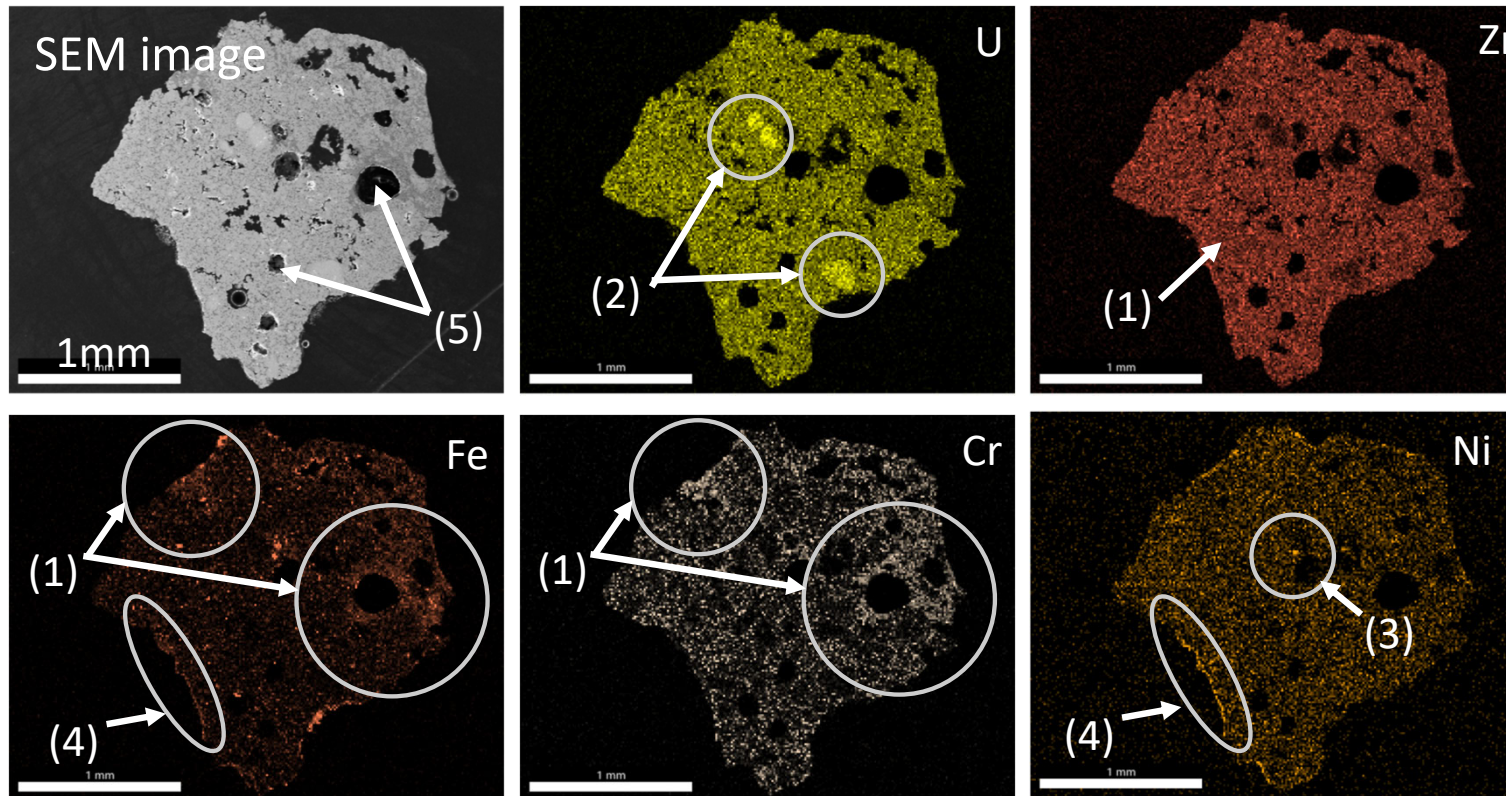


Figure 2 Optical microscope image of a cross-section of **Chunk C**

## General region classifications based on cross-sectional observation and element distribution



➤ Define structure observation was conducted of regions that showed unique element distributions (1)-(5), and assessments were made about formation process of each region (Refer to the following pages)

**Chunk C** Element distribution of the entire cross-section (EDX element mapping)

### 【Classification results】

- (1) Zr, and U are widely distributed throughout the entire sample and there is one Zr-U-O island less than several 10s - 100μm in size. Fe and Cr are distributed in the gap. There is an uneven distribution of Fe and Cr near the void.
- (2) A clear region of highly concentrated U approximately 100μm in size is present in the high CT value area
- (3) There is one small Fe-Ni near the center.
- (4) There are relatively large amounts of Fe and Ni in the area that corresponds to the outermost surface of the sample.
- (5) There are voids of various sizes, shapes, and dimensions distributed throughout the entire cross-section

⇒ Fine mixed region

⇒ Coarse U-Zr-O

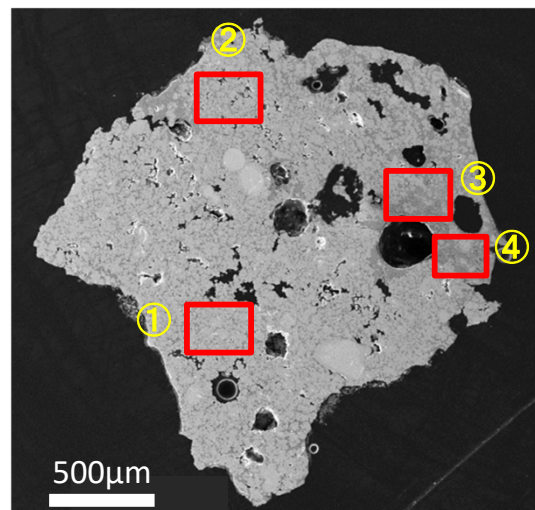
⇒ Fe-Ni

⇒ Surface layer

⇒ Void

### Fine mixed region

Chunk C



SEM image

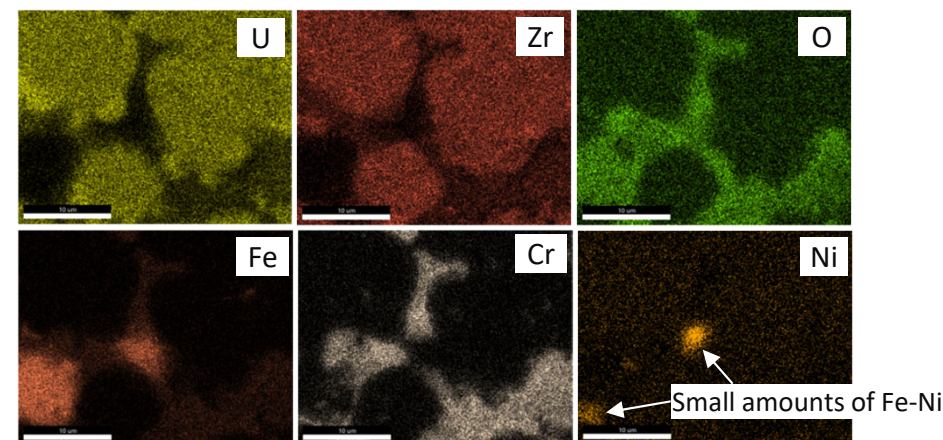
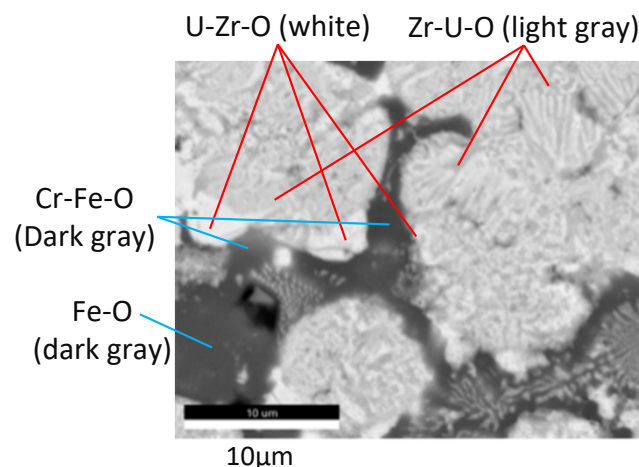
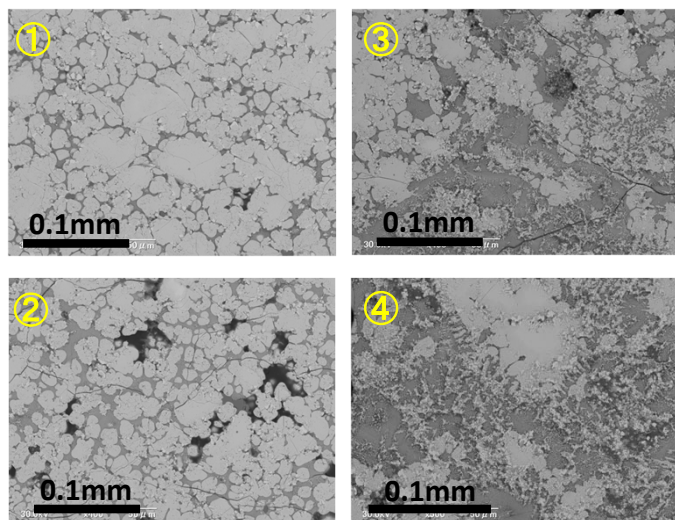


Figure 1 Typical example of fine mixed region element distribution

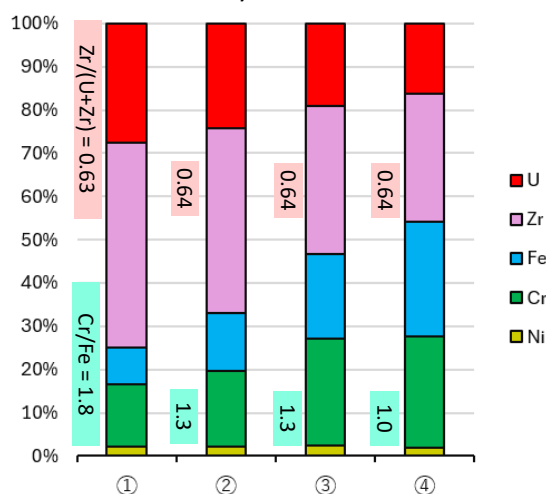
Complete SEM image



Region with a lot of U/Zr  
(①②)

Region with a lot of Fe/Cr  
(③④)

The Zr/(U+Zr) ratio is fairly constant regardless of location, but the Cr/Fe ratio differs by location



Element composition of each visual field  
(SEM-EDX Surface analysis results for each visual field)

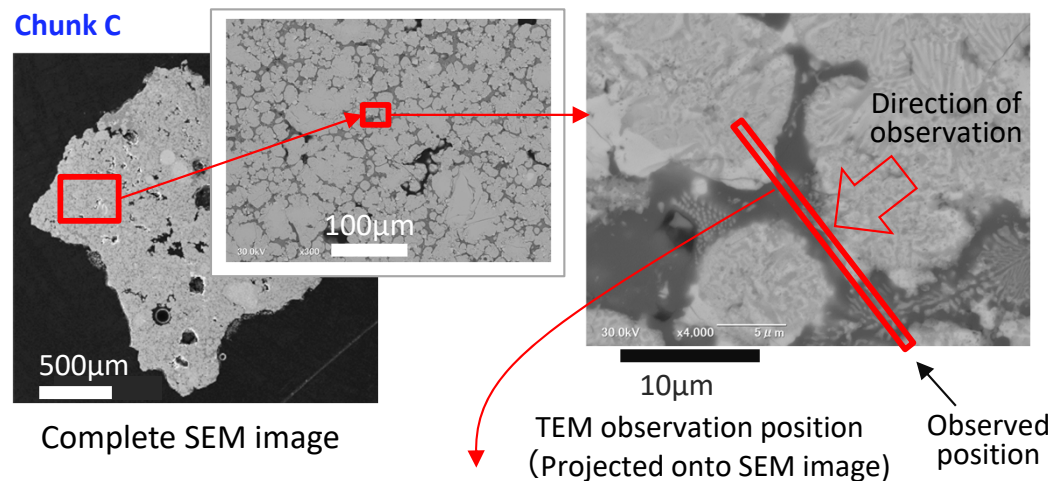
- There are mainly 4 types of phases in the fine mixed region: Zr-U-O, U-Zr-O, Cr-Fe-O and Fe-O, but there is a mixture of various sizes from less than 1μm to several 10μm (Figure 1)
- On a macro scale the sample can be divided into regions with a lot of U/Zr and regions with a lot of Fe/Cr (Figure 2)

➤ Since it is possible that temperature ranges within which each region formed differ, TEM was used to examine the structural phases of each region and make assessments about the differences in the formation process (See the next page)

Figure 2 Composition of each fine mixed region

### Fine mixed region (Region with a lot of U/Zr)

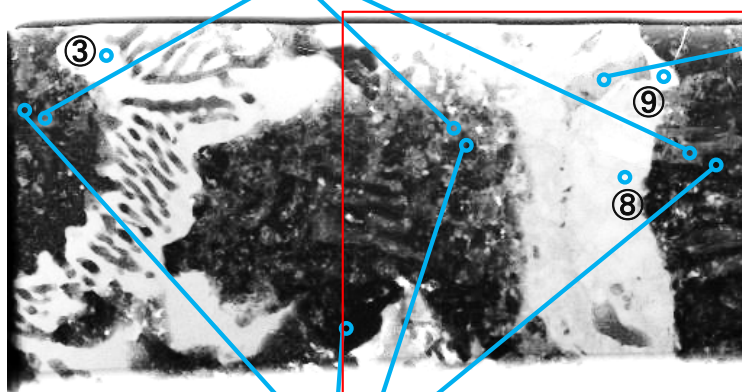
Chunk C



Tetragonal crystal (Zr,U)O<sub>2</sub>

Zr/(U+Zr)=0.64, 0.72, 0.63

②,⑤,⑩



①,④,⑥,⑪ cubic crystal (U,Zr)O<sub>2</sub>

Zr/(U+Zr)=0.23, 0.31, 0.26, 0.29

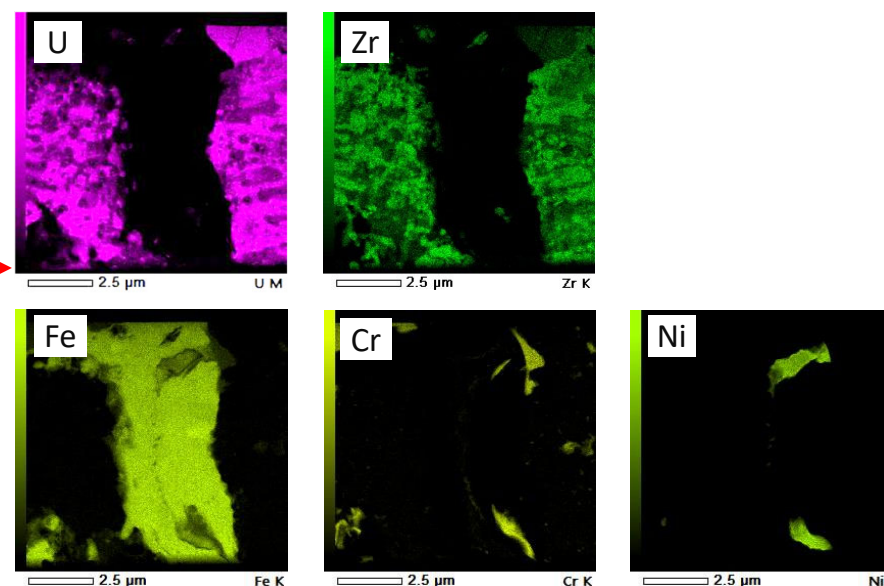
TEM bright-field image

- The Zr-U-O island several 10s µm in size seen under magnification was found through TEM observation to be a mixture of cubic crystals (U,Zr)O<sub>2</sub> and tetragonal crystals (Zr,U)O<sub>2</sub> less than approx. 1µm in size.

➤ The cubic crystal + tetragonal crystal mixed phase, was either formed during solid phase separation or directly deposited from the molten material.

- There are Fe<sub>3</sub>O<sub>4</sub> spinels and FeCr<sub>2</sub>O<sub>4</sub> spinels around the Ni-Fe metal phase

➤ It is assumed that the aforementioned region was formed as Cr and Fe, which are elements in Fe-Cr-Ni structural materials (stainless steel, etc.) and which oxidize easily, oxidized in succession.

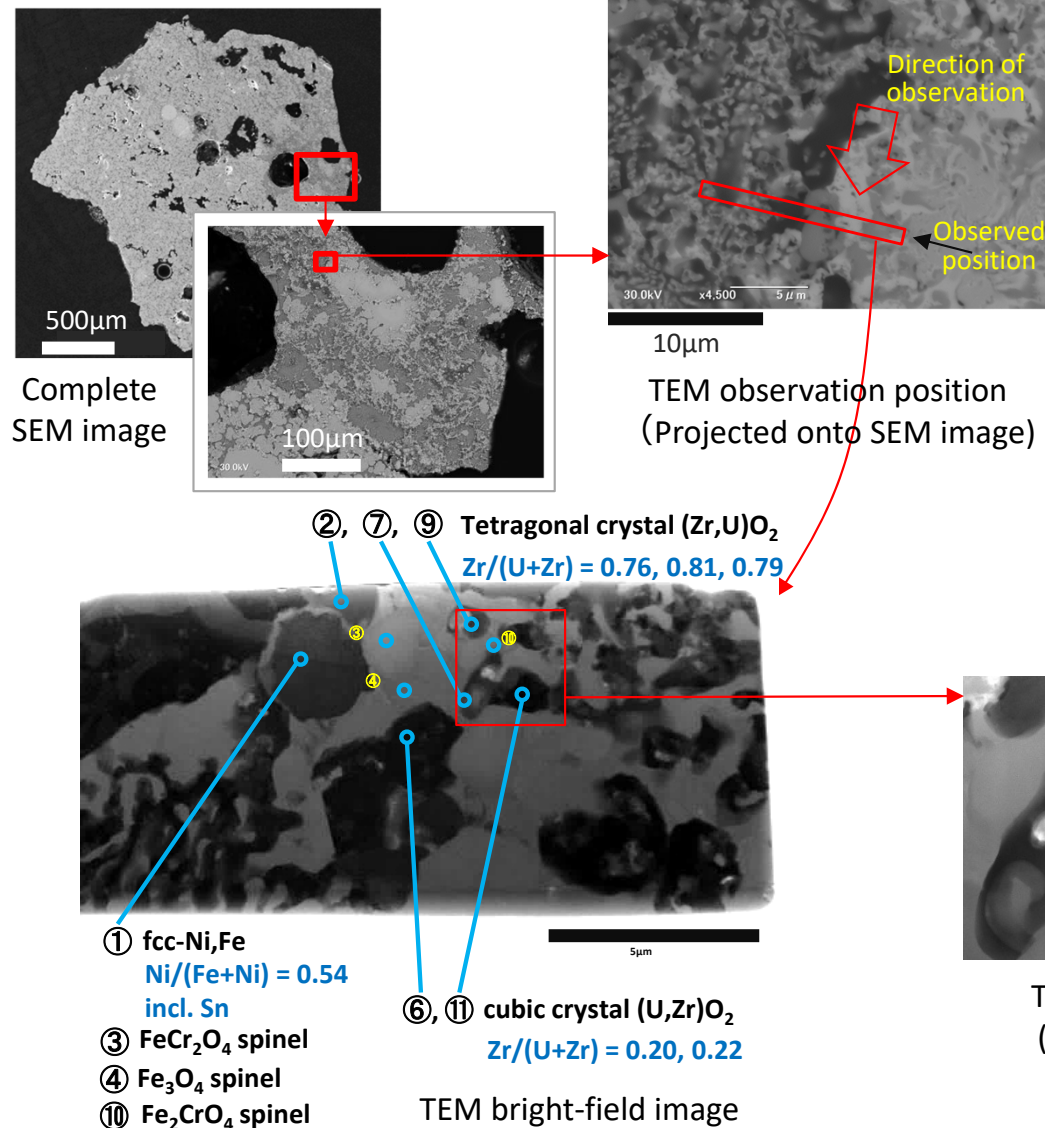


Primary element distribution (STEM-EDX surface analysis)

Figure Fine mixed region (region with a lot of U/Zr) structure phase (TEM analysis results)

### Fine mixed region (Regions with a lot of Fe/Cr)

#### Chunk C



- The identified compounds were for the most part the same as those in areas with a lot of U/Zr (see the previous page) (Figure 1)
- Compared with regions that have a lot of U/Zr (See the previous page), there are large differences in the composition of material between the cubic crystals (U,Zr)O<sub>2</sub> and tetragonal crystals (Zr,U)O<sub>2</sub>
  - ✓ Zr/(U+Zr) ratio of regions with a lot of U/Zr (refer to the previous page)  
Approx. 0.2–0.3 (cubic crystal) | Approx. 0.6–0.7 (tetragonal crystal)
  - ✓ Zr/(U+Zr) ratio of regions with a lot of Fe/Cr (refer to Figure 1)  
Approx. 0.2 (cubic crystal) | Approx. 0.8 (tetragonal crystal)

➤ It is possible that the aforementioned regions were formed in a temperature range that differs from areas with a lot of U/Zr.

#### Enlargement of element distribution (STEM-EDX surface analysis)

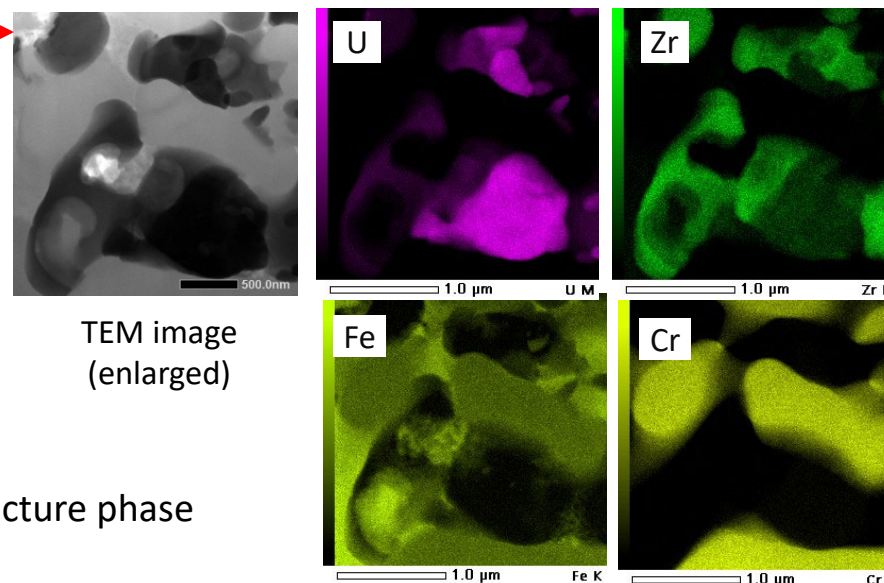


Figure 1 Fine mixed region (region with a lot of U/Zr) structure phase (TEM analysis results)

## Coarse U-Zr-O

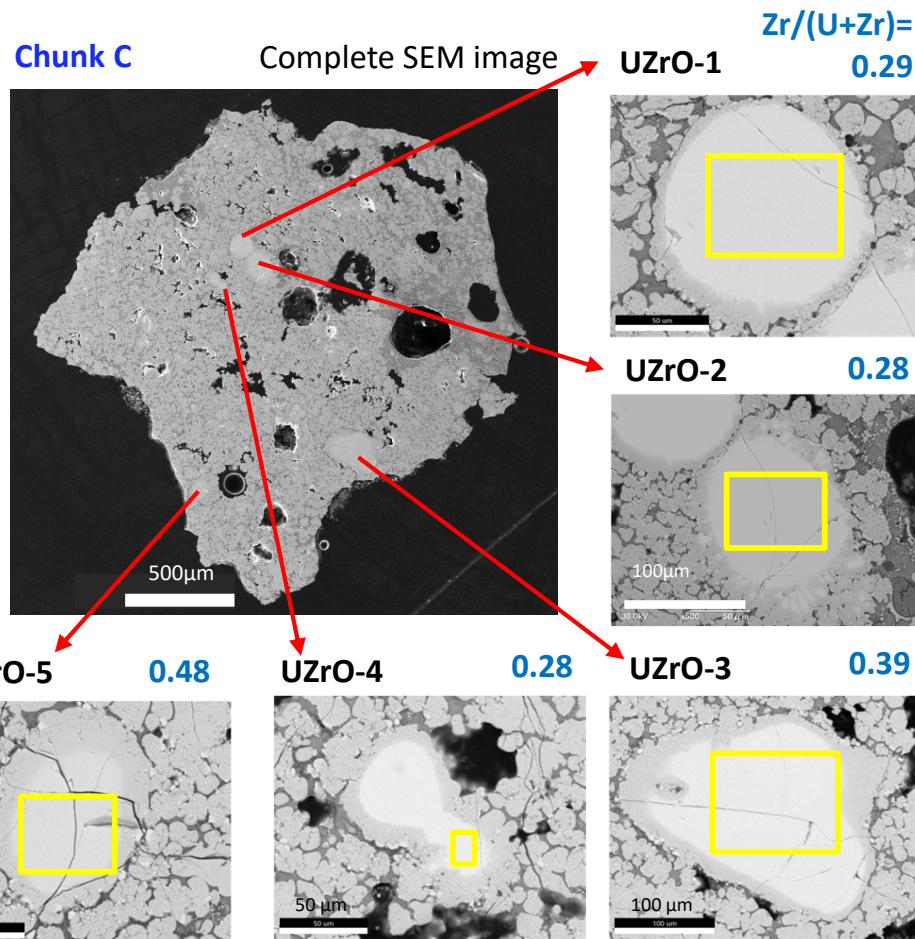


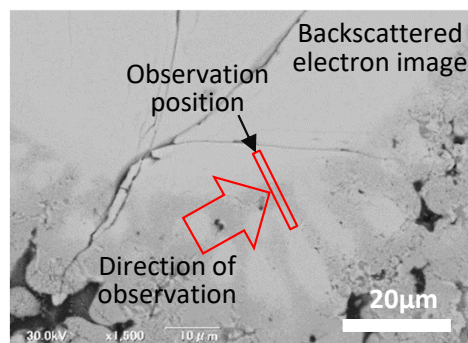
Figure SEM Observation results that focus on the coarse U-Zr-O phase

- There is a densely packed region of coarse U-Zr-O circles approx. 50- more than 100μm in size scattered throughout the fine mixed region
  - ✓ There is a fine mixed region separate from the phase with a high concentration of Zr in the periphery
  - ✓ The concentration of U in the phase is high. The  $Zr/(U+Zr)$  ratio near the center of each particle is between approx. 0.3–0.5, and differs depending on the particle
- It is assumed that when the coarse U-Zr-O formed, the surrounding fine mixed region was still in a liquid phase state.
- The U to Zr concentration ratio within each particle is almost constant, indicating that each particle most likely grew within approximately the same temperature range

➤ In order to examine the state of the molten material when coarse U-Zr-O formed, TEM was used to perform a detailed observation of the area bordering the fine mixed region (See the next page)

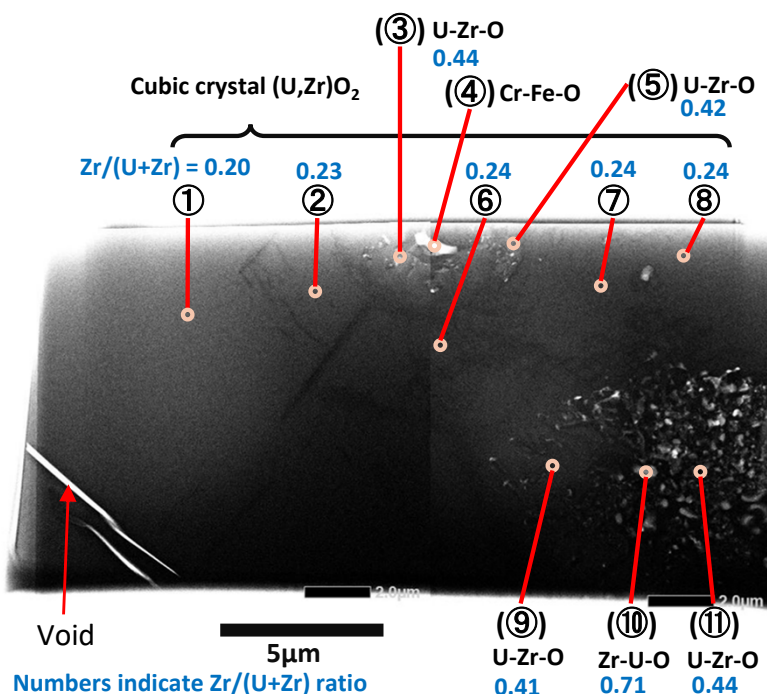
### Coarse U-Zr-O

#### Chunk C



"UZrO-2," which has a unique shape around the border of the fine mixed region, was focused on and a sample was taken near the boundary for TEM observation

TEM observation position  
(Projected onto SEM image)



A dendritic shape can be seen spreading from the inside towards the outer edge

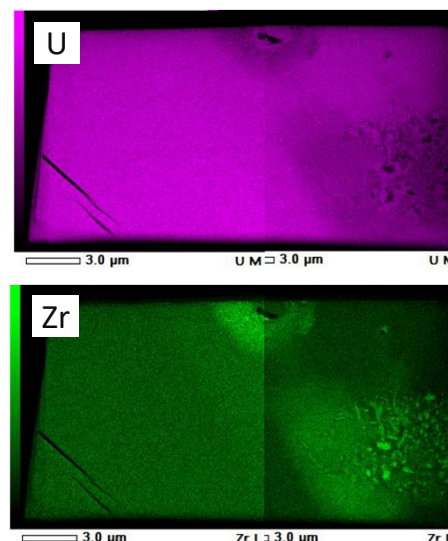


Figure 1 Coarse U-Zr-O structure phase  
(TEM Analysis results)

- A cubic crystal (U,Zr)O<sub>2</sub> phase with a high concentration of U spreads out towards the edge in a dendritic pattern (Figure 1/Figure 2 )
- It is possible that the dendritic part of the inside of coarse U-Zr-O is a monoclinic crystal (Figure 3). Surrounding phases consist of areas with slightly high concentrations of Zr between the dendritic shapes (Figure 1 ③, ④, ⑨~⑪).

➤ It is possible that when the surrounding fine mixed region was melting, the crystal grew into a dendritic shape or there was Zr, etc. intrusion from the liquid phase.

The diffraction patterns of positions ①, ②, ⑥, ⑦, and ⑧ indicate the same crystal system/ crystal direction, and it is possible that these are monoclinic crystals.

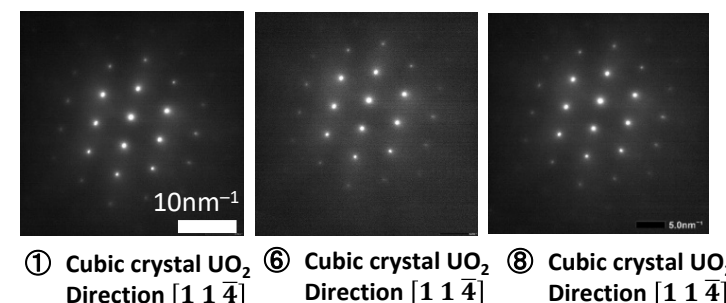
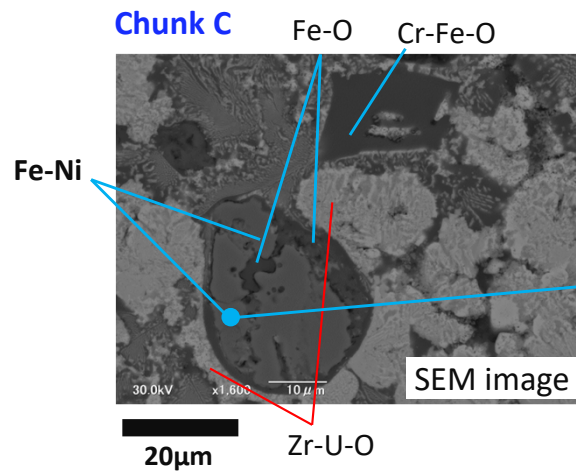


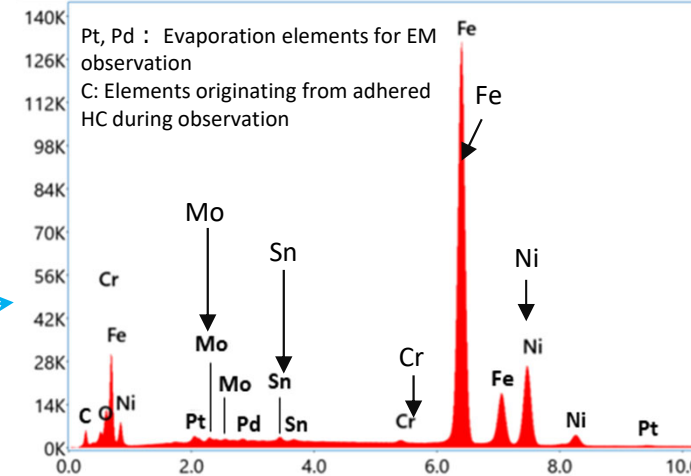
Figure 3 Example of the electron diffraction pattern

Figure 2 U/Zr distribution  
(STEM-EDX surface analysis)

## Fe-Ni metal phase



EDX point analysis spectrum from above the metal phase



- Fe-Ni metal grains several  $\mu\text{m}$ -approx. 20 $\mu\text{m}$  in size are scattered throughout the flying mixed region and surrounded by Fe-O and Cr-Fe-O.
- Each grain has a different composition

Figure 1 SEM observation results from near the Fe-Ni metal phase

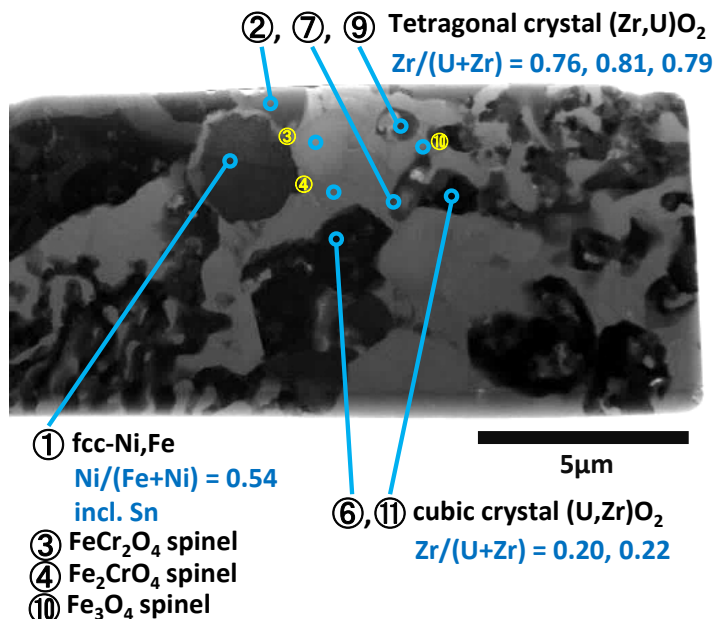
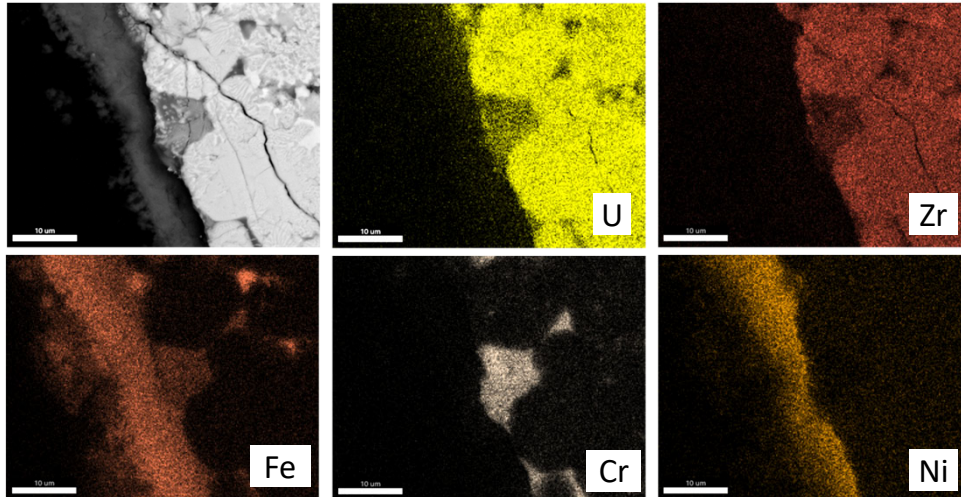


Figure 2 Fine mixed region TEM observation results  
(Figure 1 is of area near a different metal phase)

- It is assumed that during the melting and solidification process, Cr and Fe, which are elements originating from structural materials (Fe, Cr, Ni), oxidized first thereby leaving a metal phase with a high concentration of Ni.

## Surface layer

### Chunk C



- The outermost surface of the sample when received was formed from layers and deposits approximately approx. 10 - 50μm thick.

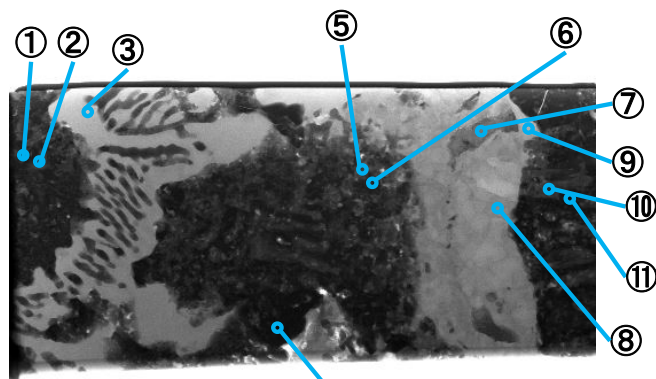
- After most of the sample was formed through melting and solidification, dissolved constituents in the cooling water were deposited on the surface of the sample thereby altering the surface

Figure 1 SEM observation from near the surface of the sample

### ◆ Assessment of the element composition of each phase using STEM-EDX

#### Fine mixed region (regions with a lot of U/Zr)

The composition of each phase from STEM-EDX point analysis ※



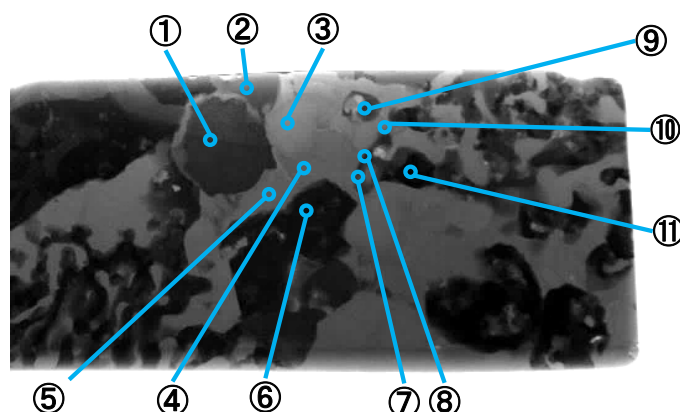
TEM observation image

(①~⑪ correspond to the analysis locations in the left graph)

| at% | O  | Al     | Cr     | Fe   | Ni   | Zr     | Sn     | Pb     | U    | U/(U+Zr)at% | Classification |
|-----|----|--------|--------|------|------|--------|--------|--------|------|-------------|----------------|
| ①   | 63 | n.d.   | 1      | n.d. | n.d. | 8      | n.d.   | n.d.   | 28   | 77          | U-Zr-O         |
| ②   | 62 | n.d.   | 1      | n.d. | n.d. | 24     | n.d.   | n.d.   | 13   | 36          | Zr-U-O         |
| ③   | 58 | L.O.Q. | 30     | 12   | n.d. | L.O.Q. | L.O.Q. | n.d.   | n.d. |             | Cr-Fe-O        |
| ④   | 71 | n.d.   | L.O.Q. | n.d. | n.d. | 9      | n.d.   | n.d.   | 20   | 69          | U-Zr-O         |
| ⑤   | 76 | n.d.   | L.O.Q. | n.d. | n.d. | 17     | n.d.   | n.d.   | 7    | 28          | Zr-U-O         |
| ⑥   | 74 | n.d.   | n.d.   | n.d. | n.d. | 7      | n.d.   | L.O.Q. | 19   | 74          | U-Zr-O         |
| ⑦   | 3  | n.d.   | n.d.   | 30   | 67   | n.d.   | n.d.   | n.d.   | n.d. |             | Ni-Fe          |
| ⑧   | 64 | n.d.   | n.d.   | 36   | n.d. | n.d.   | n.d.   | n.d.   | n.d. |             | Fe-O           |
| ⑨   | 61 | n.d.   | 27     | 12   | n.d. | n.d.   | n.d.   | n.d.   | n.d. |             | Cr-Fe-O        |
| ⑩   | 76 | n.d.   | L.O.Q. | n.d. | n.d. | 15     | n.d.   | n.d.   | 9    | 37          | Zr-U-O         |
| ⑪   | 73 | n.d.   | n.d.   | n.d. | n.d. | 8      | n.d.   | n.d.   | 19   | 71          | U-Zr-O         |

The composition of each phase from STEM-EDX point analysis ※

#### Fine mixed region (regions with a lot of Fe/Cr)



TEM observation image

(①~⑪ correspond to the analysis locations in the left graph)

| at% | O      | Si   | Cr     | Fe   | Ni   | Zr     | Sn   | U      | U/(U+Zr) | Classification |
|-----|--------|------|--------|------|------|--------|------|--------|----------|----------------|
| ①   | L.O.Q. | n.d. | n.d.   | 45   | 54   | n.d.   | 1    | n.d.   |          | Ni-Fe          |
| ②   | 82     | n.d. | L.O.Q. | 1    | n.d. | 13     | n.d. | 4      | 24       | Zr-U-O         |
| ③   | 70     | n.d. | 19     | 11   | n.d. | L.O.Q. | n.d. | n.d.   |          | Cr-Fe-O        |
| ④   | 76     | 2    | L.O.Q. | 20   | n.d. | 2      | n.d. | L.O.Q. |          | Fe-O           |
| ⑤   | 75     | 2    | L.O.Q. | 21   | n.d. | 2      | n.d. | L.O.Q. |          | Fe-O           |
| ⑥   | 75     | n.d. | L.O.Q. | n.d. | n.d. | 5      | n.d. | 20     | 80       | U-Zr-O         |
| ⑦   | 85     | n.d. | L.O.Q. | 6    | n.d. | 7      | n.d. | 2      | 19       | Zr-Fe-U-O      |
| ⑧   | 87     | n.d. | L.O.Q. | 1    | n.d. | 9      | n.d. | 3      | 24       | Zr-Fe-U-O      |
| ⑨   | 84     | n.d. | L.O.Q. | 1    | n.d. | 12     | n.d. | 3      | 21       | Zr-U-O         |
| ⑩   | 72     | n.d. | 6      | 20   | n.d. | 2      | n.d. | n.d.   |          | Fe-Cr-O        |
| ⑪   | 76     | n.d. | L.O.Q. | n.d. | n.d. | 5      | n.d. | 19     | 78       | U-Zr-O         |

※ With the exception of elements for which it is noted n.d. or LOQ, the numbers reflect the percentage of each element from 100%.

- n.d.: Indicates that no significant amounts were detected in the spectrum ("not detected")
- L.O.Q.: Lower limit of quantification. Whereas a small peak was seen in the spectrum, it was determined that quantity was below the lower limit of quantification which is less than 0.5at%

### ◆ TEM electron diffraction pattern measurements and crystalline structure identification through Raman spectroscopy

- A TEM electron diffraction pattern was obtained for each phase position seen in the fine mixed region and the crystalline system was identified. Furthermore, estimates were made about the chemical form based upon the composition analysis results for each position mentioned on the previous page (Figure 1)
- In addition to the electron diffraction, Raman spectroscopy (Figure 2) was used as necessary to improve the accuracy of crystalline structure identification.

STEM image of the fine mixed region  
(Right half of the region shown on the previous page)

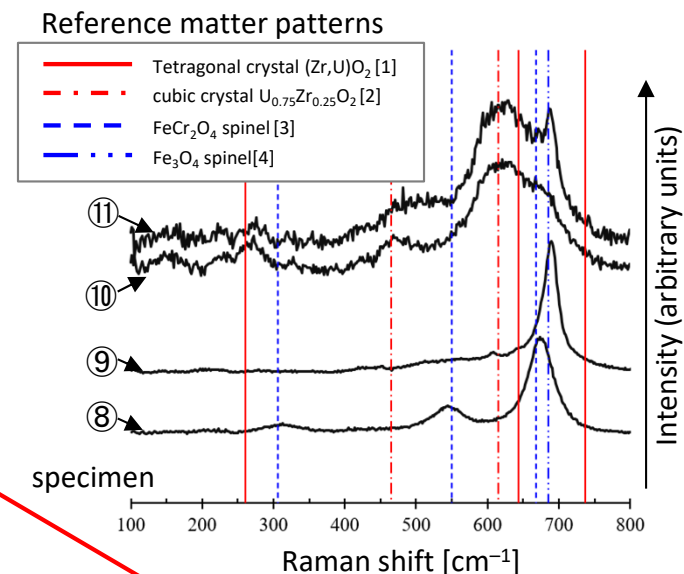
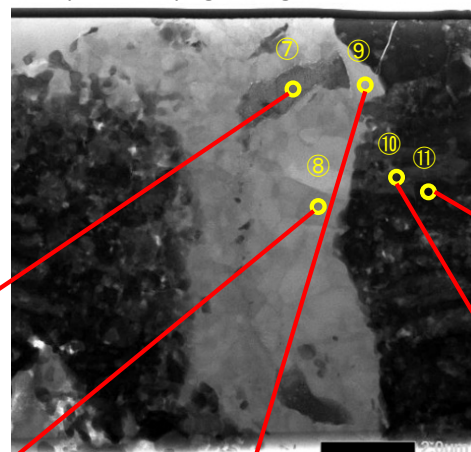


Figure 2 Raman spectroscopy results for each phase

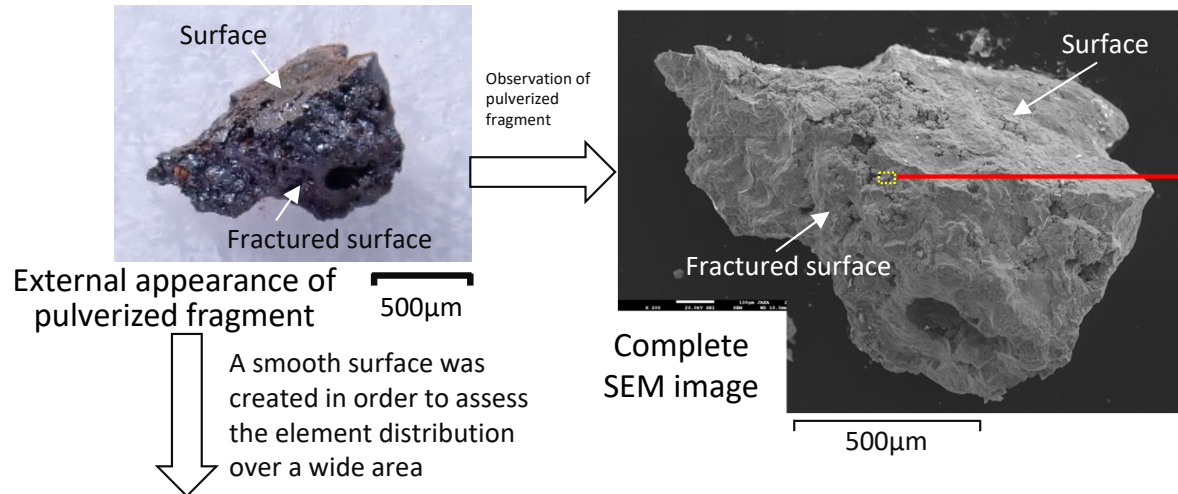
| Position                                                | ⑦Ni-Fe                                            | ⑧Fe-O                                                                    | ⑨Cr-Fe-O                                                                   | ⑩Zr-U-O                                                                  |                                                                          | ⑪U-Zr-O                                                            |
|---------------------------------------------------------|---------------------------------------------------|--------------------------------------------------------------------------|----------------------------------------------------------------------------|--------------------------------------------------------------------------|--------------------------------------------------------------------------|--------------------------------------------------------------------|
| TEM Electron diffraction pattern                        |                                                   |                                                                          |                                                                            |                                                                          |                                                                          |                                                                    |
| Crystal system identification result                    | <b>fcc-(Ni,Fe)</b><br>Direction $[0\ \bar{1}\ 1]$ | <b>Fe<sub>3</sub>O<sub>4</sub> spinel</b><br>Direction $[1\ \bar{1}\ 2]$ | <b>FeCr<sub>2</sub>O<sub>4</sub> spinel</b><br>Direction $[0\ 1\ \bar{1}]$ | <b>Tetragonal crystal ZrO<sub>2</sub></b><br>Direction $[\bar{3}\ 3\ 1]$ | <b>Tetragonal crystal ZrO<sub>2</sub></b><br>Direction $[1\ \bar{4}\ 2]$ | <b>Cubic crystal UO<sub>2</sub></b><br>Direction $[0\ 1\ \bar{3}]$ |
| Of chemical form made based on the composition analysis | <b>fcc-(Ni,Fe)</b>                                | <b>Fe<sub>3</sub>O<sub>4</sub> spinel</b>                                | <b>FeCr<sub>2</sub>O<sub>4</sub> spinel</b>                                | Tetragonal crystal (Zr,U)O <sub>2</sub>                                  |                                                                          | cubic crystal (U,Zr)O <sub>2</sub>                                 |

Figure 1 TEM Electronic diffraction pattern for each analysis position and estimates of the chemical form based on composition analysis

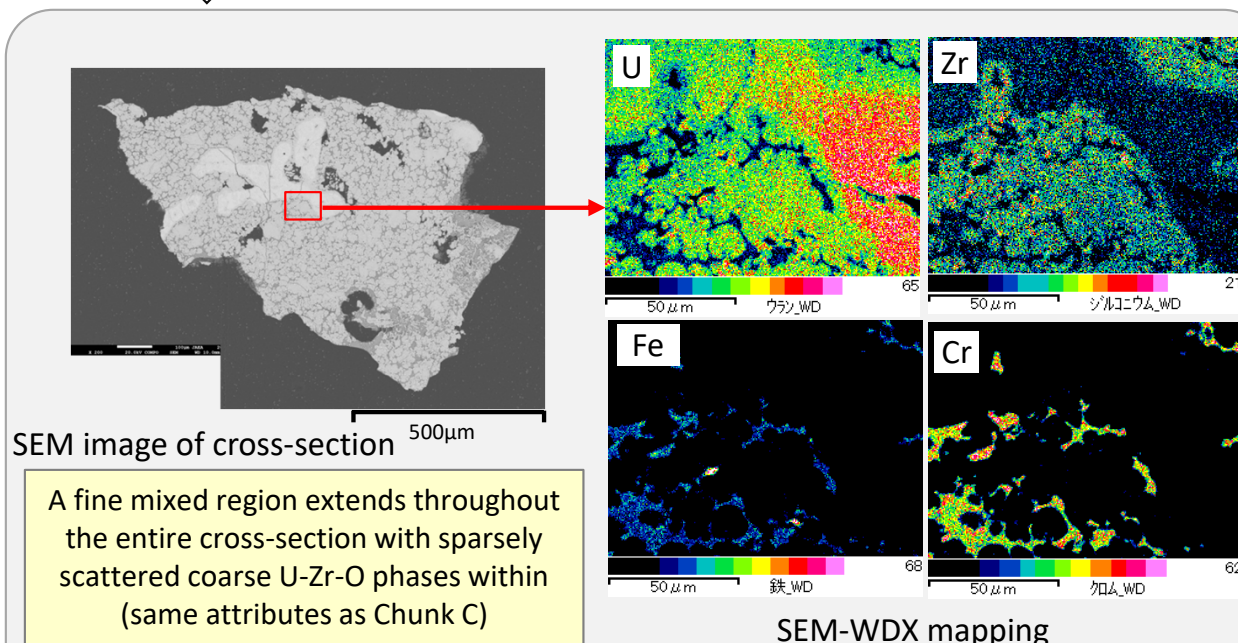
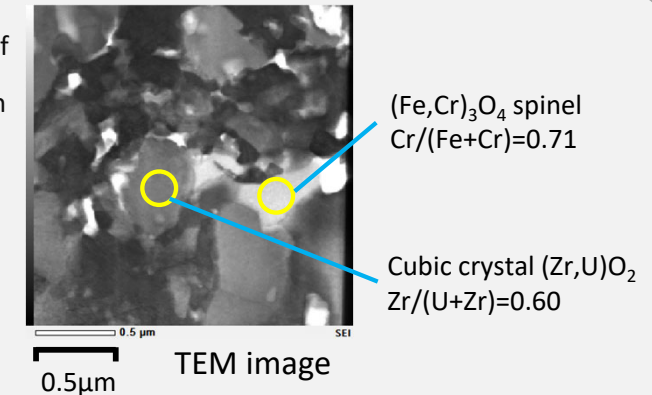
### Comparison of multiple batched specimens

SEM and TEM Used to perform a detailed observation of not just Chunk C but the other batched specimens as well, and it was confirmed that they have the same element distribution and crystalline structure.

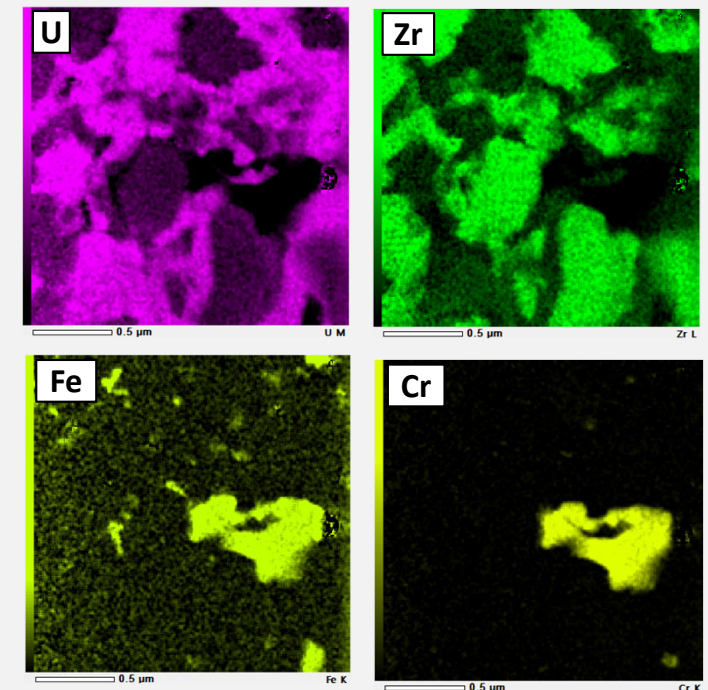
#### Chunk A-1



A thin film was fabricated from part of the fractured surface to prepare a specimen for TEM observation



Element distribution in cross section of pulverized fragment (SEM-WDX)



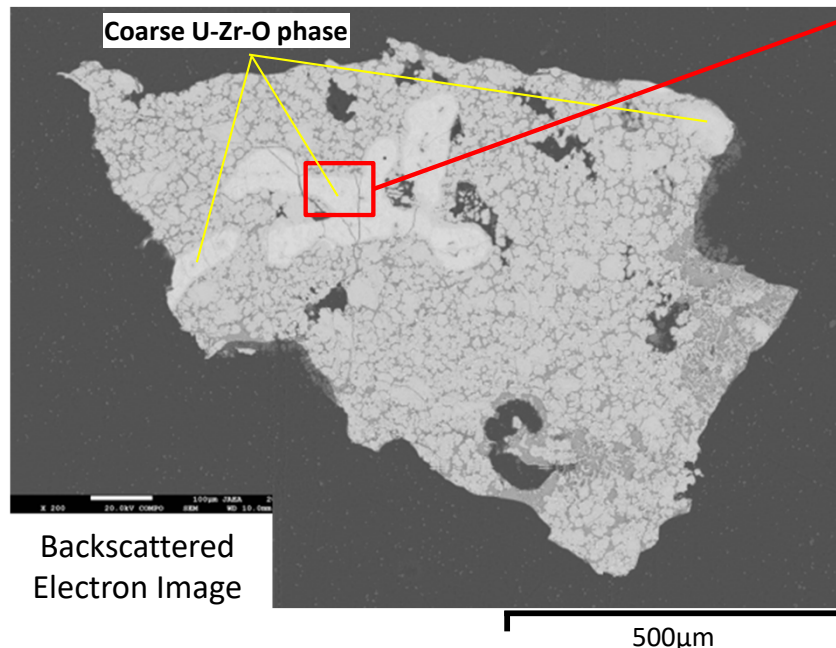
STEM-EDX mapping

TEM observation results from thin film of cross-section

### ◆ SIMS assessment of localized U isotope ratio

SIMS was used to assess localized U isotope ratios in order to contribute to deliberations over the formation mechanism of the coarse U-Zr-O phase during the formation process of the second fuel debris sample. (Because if there is a remarkable difference between the isotope ratios of coarse U-Zr-O phase and the fine mixed region, it could be direct evidence that they have different origins)

- No remarkable difference in the ratio of  $^{235}\text{U}/^{238}\text{U}$  was found between the coarse U-Zr-O phase and the fine mixed region (both were approx. 2at%)
- No direct evidence about the origins of the coarse U-Zr-O phase was found at the measurement location, but we will continue to take measurements at other locations and analyze the sample in order to ascertain trends of localized isotope ratios.



Chunk A-1 cross-section SEM image

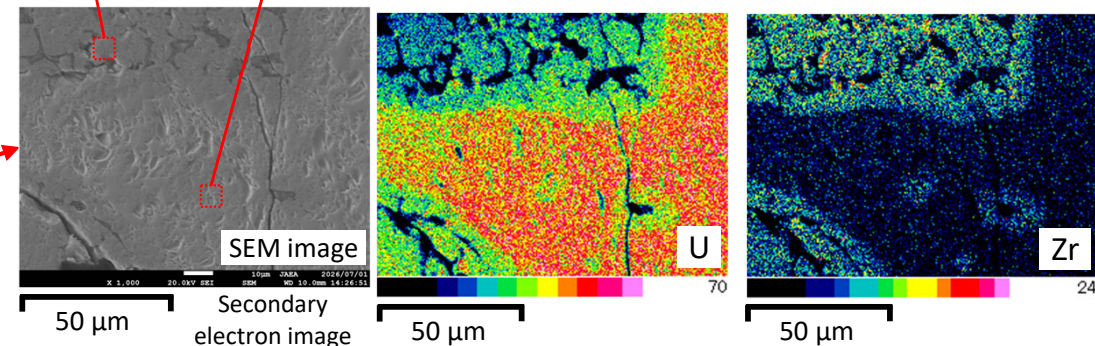


ChunkA-1

Relatively small are in the fragment, but as with Clump C, the Coarse U-Zr-O phase is mixed with the Fine mixed region.

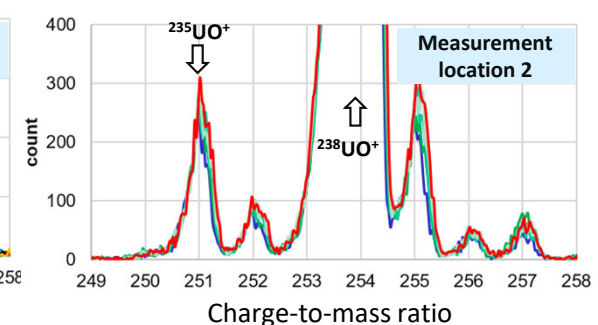
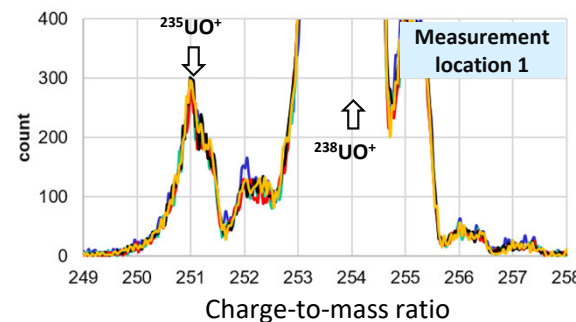
Measurement location 2:  
Fine mixed region

measurement location 1:  
Coarse U-Zr-O phase



Approximate location of SIMS measurements and element distribution in the surrounding (SEM-WDX)

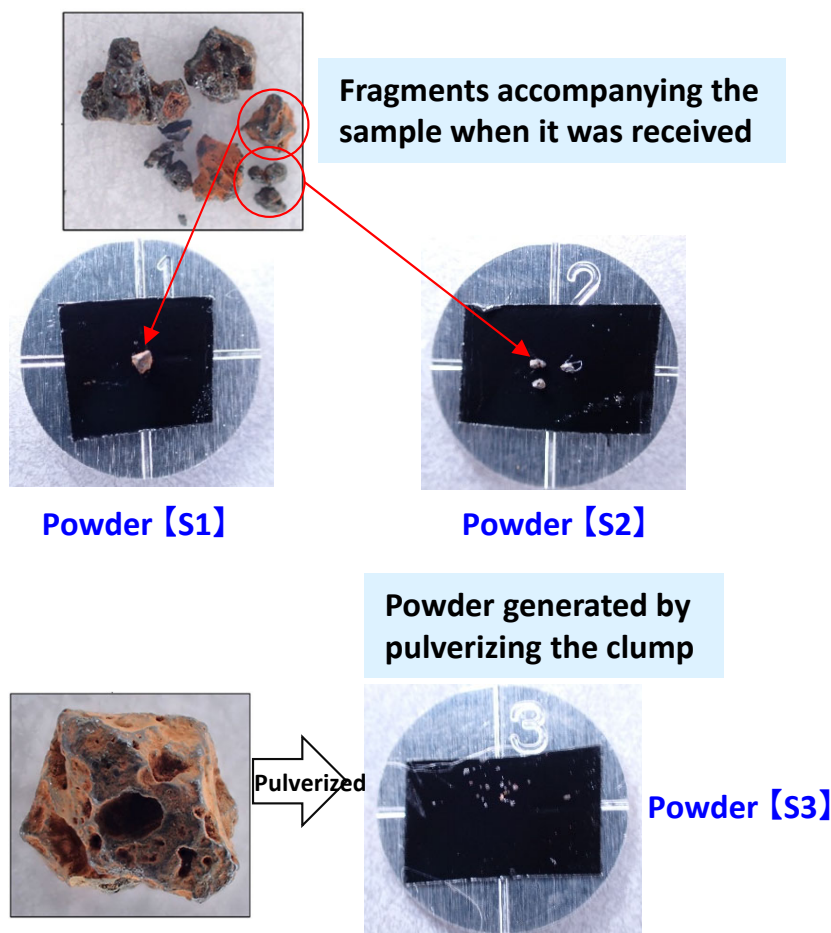
※ Calculated from the intensity ratio of  $^{235}\text{UO}^+$  and  $^{238}\text{UO}^+$   
(Measurements repeated five times)



Mass spectrum for each measurement location (SIMS measurement results)

## ◆ The aim of synchrotron radiation analysis:

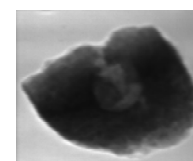
To use  $\mu$ -XRF/XRD/XAFS analysis that employs the High Intensity Synchrotron Radiation of Spring-8 to ascertain the primary constituent elements of the fuel debris sample as well as the distribution of those elements, as well as the chemical state of fuel elements, and crystalline structures



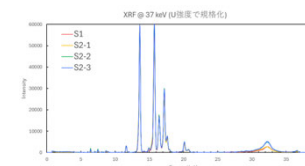
## Synchrotron radiation analysis (SPring-8) flow

Entire specimen analyzed using unfocused beam (2mm square)  
( X-ray transmission image, XRF, XRD, XAFS )

- ✓ General understanding of the attributes of the entire specimen  
(Fine structure, constituent elements, crystalline structure, chemical state)



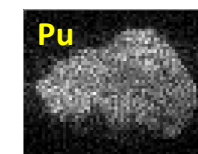
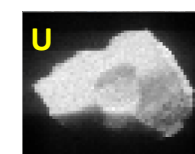
X-ray transmission image Powder [S2-2]



XRF example from unfocused beam

Constituent element of void distribution examined with  $\mu$ -beam  
(1 $\mu$ m square) (XRF mapping)

- ✓ Constituent element void distribution examined
- ✓ Association between U and trace elements (Pe, etc.) found



Powder [S2-2] XRF mapping example

Crystalline structure and chemical state analyzed at specific specimen positions ( $\mu$ -XRD,  $\mu$ -XAFS point analysis)

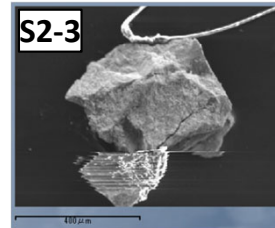
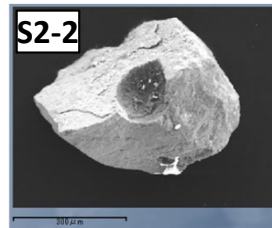
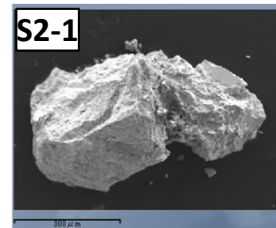
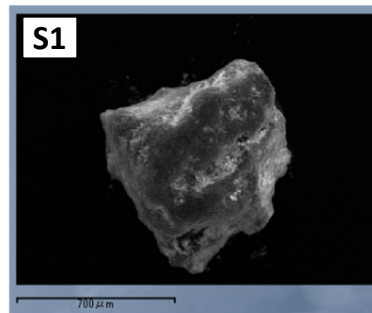
- ✓ Ascertain crystalline structure and chemical state at specific specimen positions
- ✓ Ascertain phase differences between the surface and internal

Figure The sample used for synchrony radiation analysis

(※Use of imaging XAFS, and XRD mapping being deliberated)

## ◆ Ratio of primary elements in each sample

Fragments accompanying the sample when it was received



Powder generated during pulverization of the clump

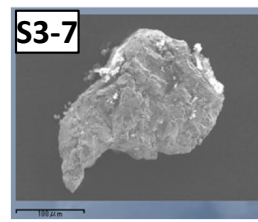
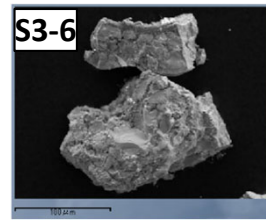
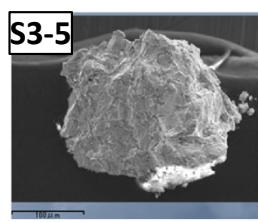
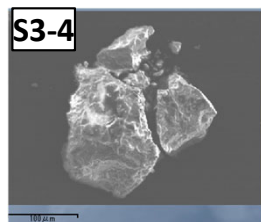
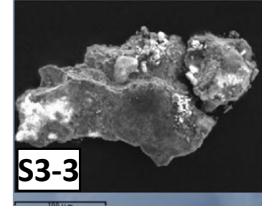
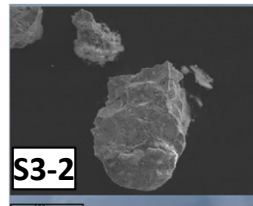
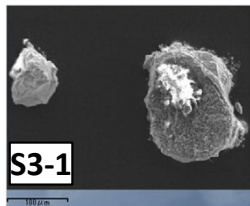
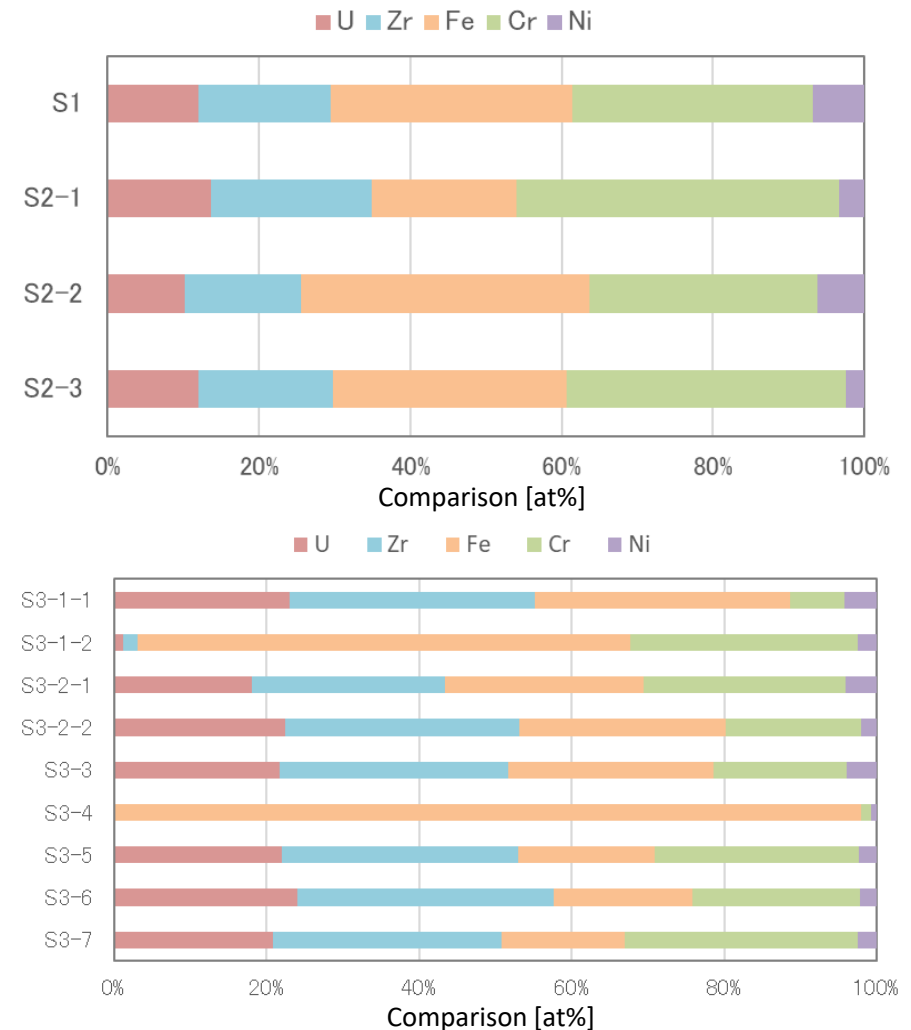


Figure 1 SEM image of sample used for synchrotron radiation analysis

The Zr/U ratio between samples generally fluctuated between 1.4 and 1.8 (Refer to Figure 2). It is assumed that the clump and fragments sampled as part of the second fuel debris sample contain a mixture of U originating from fuel and Zr originating from cladding tubes, etc.



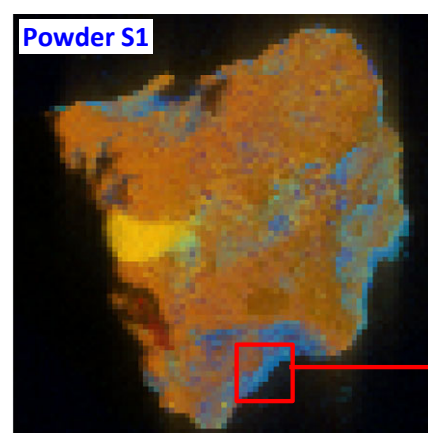
※ Calculated by comparison the measurement results at 18.2 keV and 8.5 keV with the Control specimen  
Data to a depth of several 10s μm from the surface is reflected in μ-XRF

Figure 2 Ratio of primary elements in each sample (XRF measurement results)

### Assessment of the chemical state near the surface

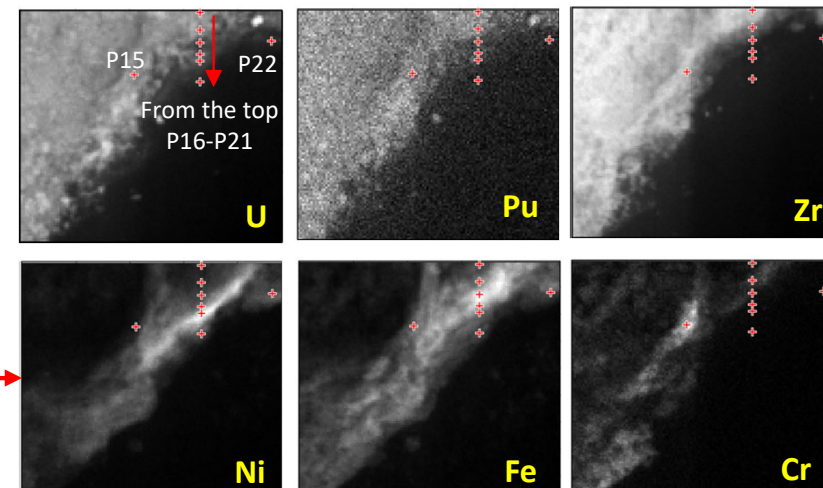
assessment of the element distribution, crystalline structure and valence, etc., of Powder S1, which has a clear position of relationship between the surface and the internal areas of the sample, were performed to examine the chemical state from the inside regions to the surface

- Regions with high concentrations of Fe/Ni exist near the surface (Figure 1). XRD analysis revealed a pattern resembling Fe oxide (Figure 2, P19 and P20), indicating that it may have formed after the accident.
- U valence at the same location is slightly high (figure 3, P19 and P20), and whereas XRD analysis revealed changes, such as a broadening of the cubic crystal  $\text{UO}_2$  peak, higher order oxides such as  $\text{U}_3\text{O}_8$  were not found.



Yellow: U Red: Zr Blue: Fe Light blue: Ni

Element distribution over the entire surface



Enlargement of the area near the surface (measurement locations P15-P21)

Figure 1  $\mu$ -XRF element distribution assessment

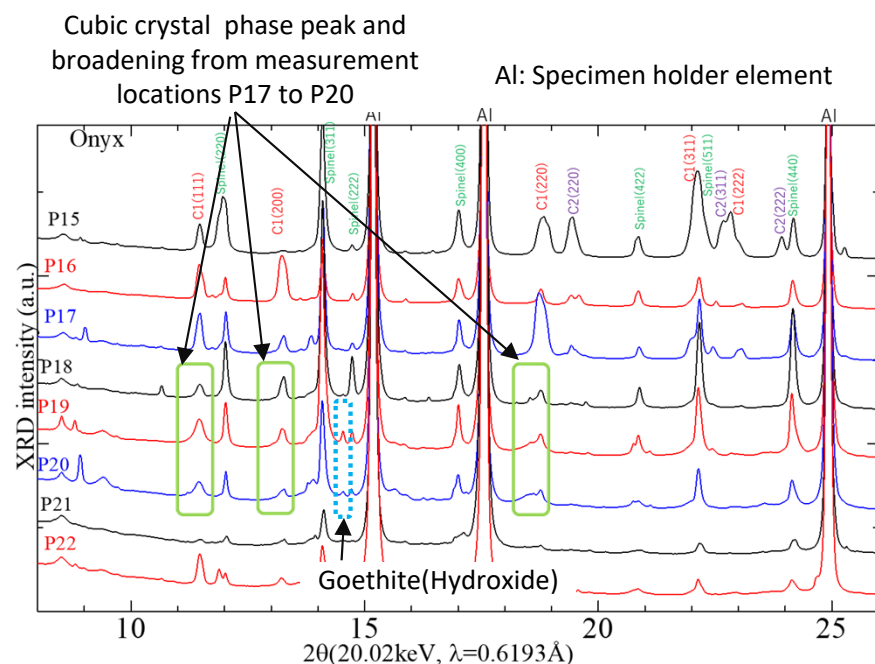


Figure 2  $\mu$ -XRD crystalline structure analysis

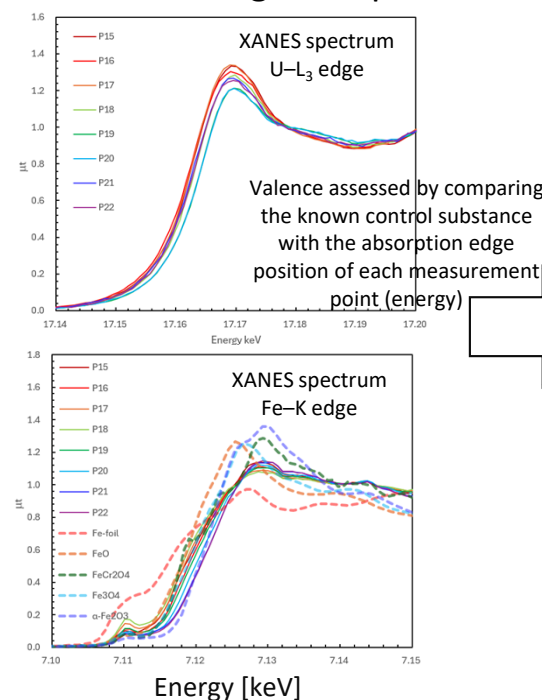
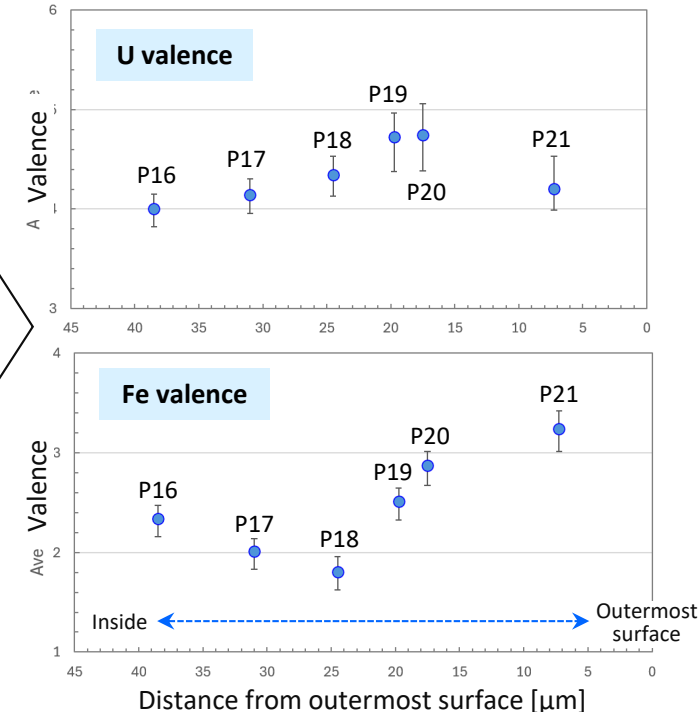
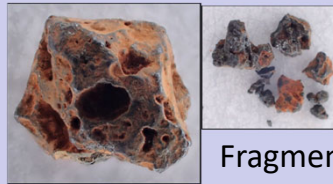
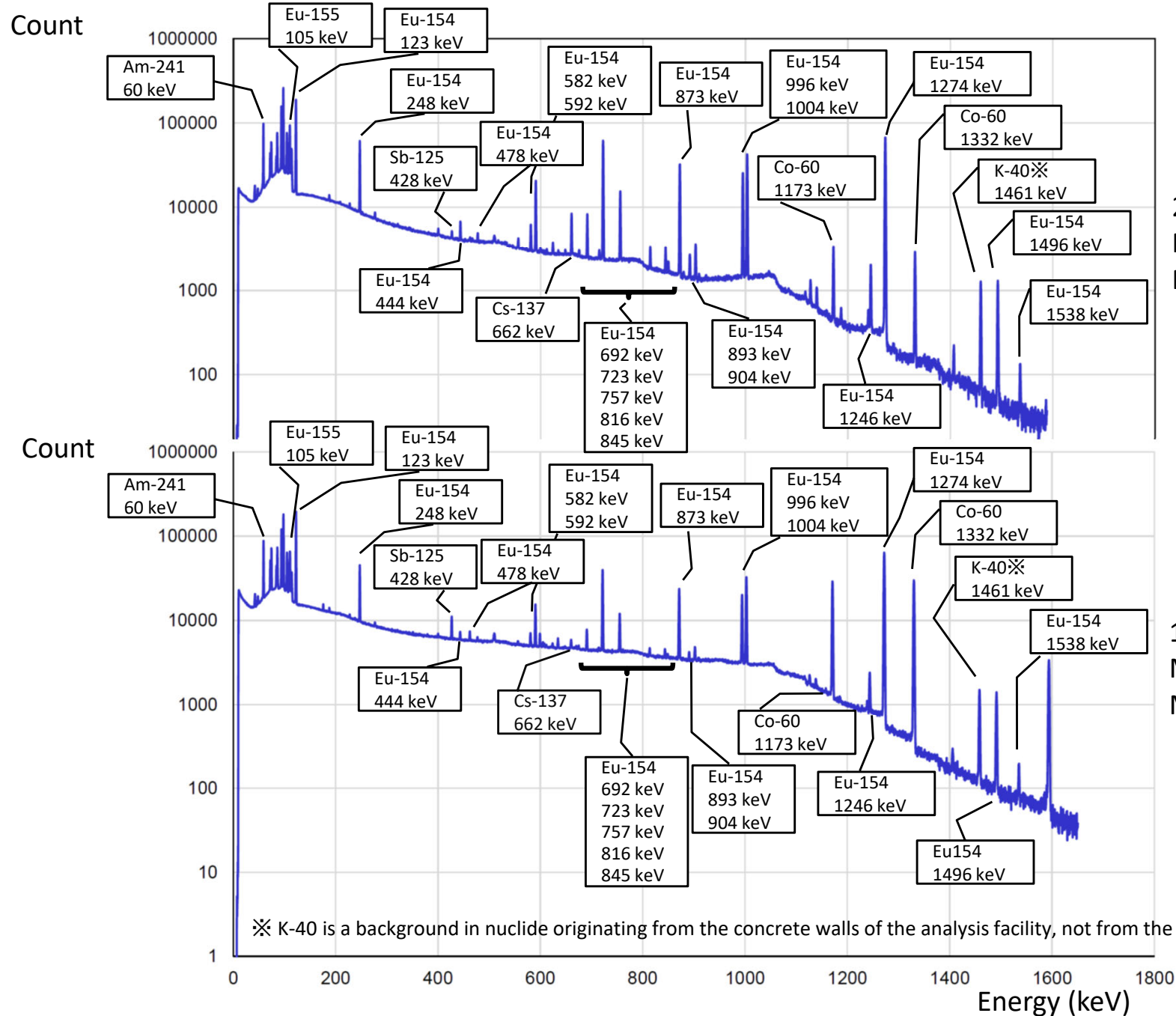


Figure 3  $\mu$ -XAFS oxidation state assessment



| Items                           | 1st Fuel debris                                                                                                                                             | 2nd Fuel debris                                                                                                                                                                    | Notes                                                                                                                                             |
|---------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------|
| External appearance             |  <p>Reddish brown overall with black and glossy regions on the surface</p> |  <p>Clump<br/>Fragments</p> <p>Brownish overall with black regions and holes on the surface</p> | The areas of the 2 <sup>nd</sup> sample that are brown (outermost surface layer) contain hardly any U, which may be why the color looks different |
| Mass                            | 0.693g                                                                                                                                                      | 0.187g (Total mass of the sample)                                                                                                                                                  | —                                                                                                                                                 |
| Size                            | Approx. 9mm×Approx. 7mm                                                                                                                                     | Approx. 5mm×Approx. 4mm (Clump)                                                                                                                                                    | —                                                                                                                                                 |
| Dose rate                       | Approx. 8mSv/h (γ-ray)                                                                                                                                      | Approx. 0.3mSv/h (γ-ray)                                                                                                                                                           | —                                                                                                                                                 |
| γ spectrometry                  | <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 values are non-uniform<br>Distribution of low density areas assumed to be voids<br>Volume: Approx. 0.1cm <sup>3</sup>                                    | CT values are non-uniform<br>Distribution of low density areas assumed to be voids<br>Volume: Approx. 0.026cm <sup>3</sup>                                                         | Same results                                                                                                                                      |
| Surface observations<br>SEM-WDX | Si, Ca, Al, etc. detected in addition to U, Zr, Fe, Cr, Ni, O                                                                                               | U, Zr, Fe, Cr, Ni, O detected.<br>Si, Ca, Al, etc. not detected                                                                                                                    | Second sample tends to be more homogeneous                                                                                                        |
| Batching status                 | Pulverized by hand with a stainless steel rod                                                                                                               | Pulverized by hand with a stainless steel rod                                                                                                                                      | —                                                                                                                                                 |

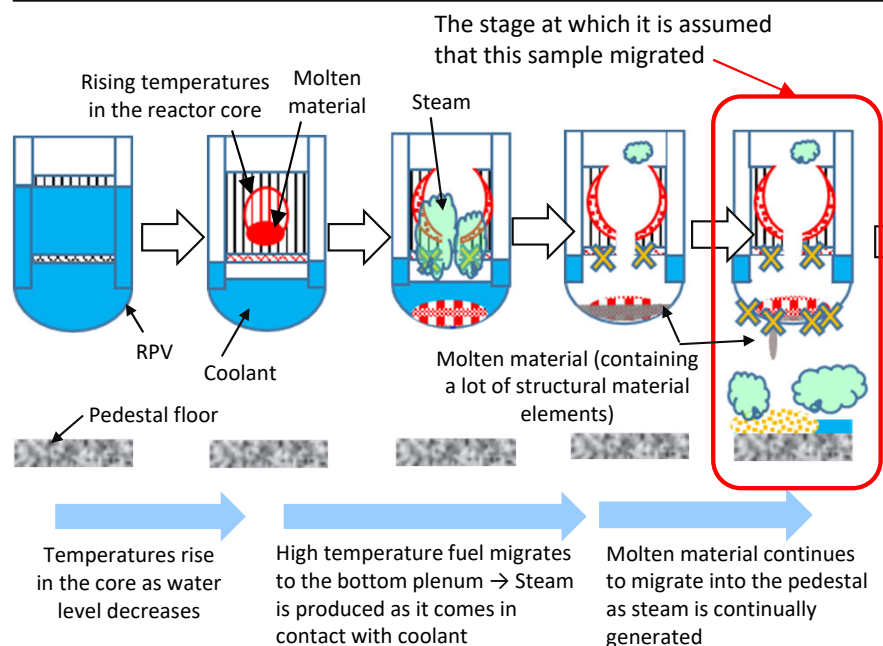


|                                                                             |                                                            | 1st Fuel debris                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | 2nd Fuel debris                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |
|-----------------------------------------------------------------------------|------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Hypotheses about the cross sectional structure and sample formation process |                                                            | <p><b>Clump [C] cross-section</b></p> <p>Approx. 7mm<br/>Approx. 9mm<br/>Approx. 9mm</p> <p>External appearance of sample</p> <p>Optical microscope image</p> <p>1mm</p> <p><b>[Overview of compositional phases]</b></p> <ul style="list-style-type: none"> <li>Coarse Zr-U-O phase</li> <li>Fe-Ni metal phase</li> <li>Fine mixed region <ul style="list-style-type: none"> <li>U-Zr-O, Zr-U-O, Fe-Cr-O, Fe-O mixed phase</li> </ul> </li> <li>Void <ul style="list-style-type: none"> <li>Size: Several - several 100s μm</li> </ul> </li> </ul> <ul style="list-style-type: none"> <li>✓ It is assumed that Cr and Fe out of the structural material elements (Fe, Cr, Ni) oxidized first.</li> <li>✓ It is assumed that the sample formed inside the RPV as it solidified from a molten state (More than approx. 1900°C)</li> </ul> | <p><b>Chunk [C] Cross-section</b></p> <p>Approx. 4mm<br/>Approx. 5mm</p> <p>External appearance of sample</p> <p>Optical microscope image</p> <p>1mm</p> <p><b>[Overview of compositional phases]</b><br/>(Backscattered electron image)</p> <ul style="list-style-type: none"> <li>Coarse U-Zr-O phase</li> <li>Void</li> <li>Fe-Ni phase (Very little)</li> <li>Fine mixed region <ul style="list-style-type: none"> <li>U-Zr-O, Zr-U-O, Fe-Cr-O, Fe-O mixed phase</li> </ul> </li> <li>Void</li> </ul> <p>50μm SEM image</p> <ul style="list-style-type: none"> <li>✓ It is assumed that Cr and Fe out of the structural material elements (Fe, Cr, Ni) oxidized first.</li> <li>✓ It is assumed that the sample formed inside the RPV as it solidified from a molten state (More than approx. 2200°C)</li> </ul> |
| Analysis results                                                            | Element content                                            | <ul style="list-style-type: none"> <li>The percentage of structural material elements (Fe, Cr, Ni) is large compared to the core composition.</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                 | <ul style="list-style-type: none"> <li>The percentage of structural material elements (Ni, in particular) is larger than the 1<sup>st</sup> fuel debris sample.</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
|                                                                             | U isotope ratio                                            | <ul style="list-style-type: none"> <li>Uranium enrichment: Approx. 1.9 wt% (<sup>235</sup>U/U)</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                | <ul style="list-style-type: none"> <li>Uranium enrichment: Approx. 2.0 wt% (<sup>235</sup>U/U)</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |
|                                                                             | Radioactivity concentration                                | <ul style="list-style-type: none"> <li>The concentration of Cs is low compared to the fuel composition prior to the accident, and the contribution from γ-ray emitting nuclides <sup>154</sup>Eu and <sup>60</sup>Co is high.</li> <li>The concentrations of <sup>154</sup>Eu and <sup>244</sup>Cm are approximately the same as the fuel composition prior to the accident, and association with U was found.</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                | <ul style="list-style-type: none"> <li>The concentration of Cs is low compared to the fuel composition prior to the accident</li> <li>The concentrations of <sup>60</sup>Co and <sup>125</sup>Sb are lower than the 1<sup>st</sup> fuel debris sample.</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |
|                                                                             | Element and compound distribution                          | <ul style="list-style-type: none"> <li>Comprised of primarily Coarse Zr-U-O phases, Fe-Ni metal phases, Fine mixed regions and Voids.</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                         | <ul style="list-style-type: none"> <li>Comprised primarily of Fine mixed regions, Coarse U-Zr-O phase and Voids. There are fewer Fe-Ni metal phases than the 1<sup>st</sup> fuel debris sample.</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
|                                                                             | Crystal structure and composition of phases that contain U | <ul style="list-style-type: none"> <li>Coarse Zr-U-O phase : cubic crystal (Zr,U)O<sub>2</sub></li> <li>Fe-Ni metal phase : Face-centered cubic structure Fe-Ni</li> <li>Fine mixed region : Cubic crystal (U,Zr)O<sub>2</sub>, cubic crystal (Zr,U)O<sub>2</sub>, Tetragonal crystal (Zr,U)O<sub>2</sub> phase, spinel-shaped (Fe,Cr)<sub>3</sub>O<sub>4</sub>, and FeO cubic crystals exist</li> <li>An amorphous phase of Ni-U-Fe-O systems has been deposited on the sample surface</li> </ul>                                                                                                                                                                                                                                                                                                                                       | <ul style="list-style-type: none"> <li>Fine mixed region : Cubic crystal (U,Zr)O<sub>2</sub> phase, tetragonal crystal (Zr,U)O<sub>2</sub> phase, spinel-shaped (Fe,Cr)<sub>3</sub>O<sub>4</sub> exist.</li> <li>Coarse U-Zr-O phase : Cubic crystal (Zr,U)O<sub>2</sub></li> <li>Fe-Ni metal phase</li> <li>There are Ni-Fe-O system phases adhered to the surface of the sample (they contain very little U)</li> </ul>                                                                                                                                                                                                                                                                                                                                                                                            |

In conjunction with deliberations to date, estimating how the accident progressed will enable us to ascertain conditions inside the reactor, such as fuel debris distribution, etc., and this information will be leveraged when deliberating fuel debris retrieval and internal investigations.

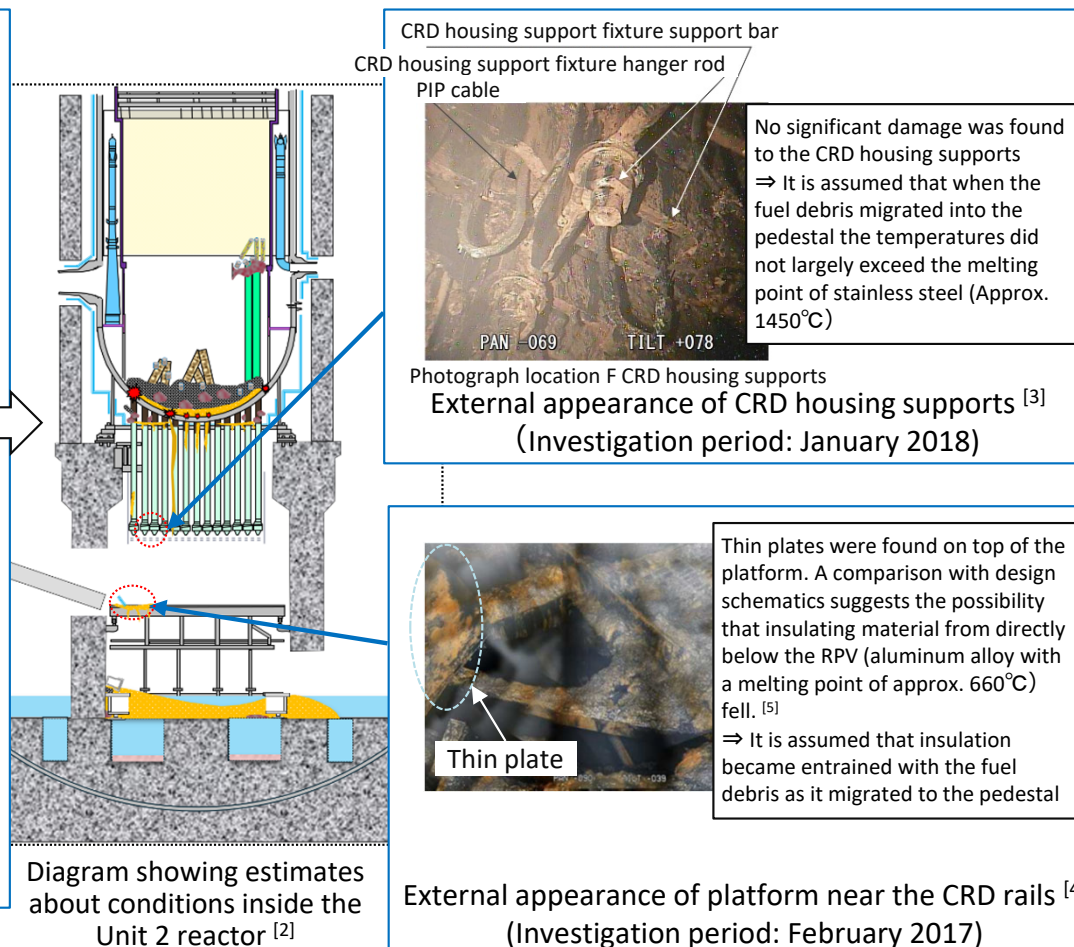
### 【Positioning of the sample analysis results】

From analysis of this fuel debris sample, we estimate that both the first and second fuel debris samples formed as structural material elements oxidized due to the steam created in the reactor pressure vessel as the material migrated into the pedestal



Unit 2 accident progression hypothesis <sup>[1]</sup>

### 【Knowledge gained from internal investigations】



[1] Figure source: Excerpts from Yamashita et al. Annals of Nuclear Energy, 173 (2022) 109129. Additions were made

[2] JAEA, Decommissioning, Contaminated Water and Treated Water Management Project (Development of Analysis and Estimation Technology for Ascertaining the Attributes of Fuel Debris (Developing Technology for Estimating damage inside the RPV and the behavior of fuel debris as it migrated inside the PCV)) subsidized by METI Agency for Natural Resources and Energy that commenced in FY2025, FY2026 Final Report, October 2025

[3] TEPCO HD, IRID, Unit 2 Primary containment vessel internal investigation results. Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (50<sup>th</sup>), Document 3-3

[4] TEPCO HD, IRID, Unit 2 Primary containment vessel internal investigation. Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (39<sup>th</sup>), Document 3-3

[5] JAEA, Decommissioning, Contaminated Water and Treated Water Management Project (Development of Analysis and Estimation Technology for Ascertaining the Attributes of Fuel Debris (Developing Technology for Estimating damage inside the RPV and the behavior of fuel debris as it migrated inside the PCV)) subsidized by METI Agency for Natural Resources and Energy that commenced in FY2022, FY2022 Final Report, September 2023

- Materials that might have been caught up in the creation 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 creation process.

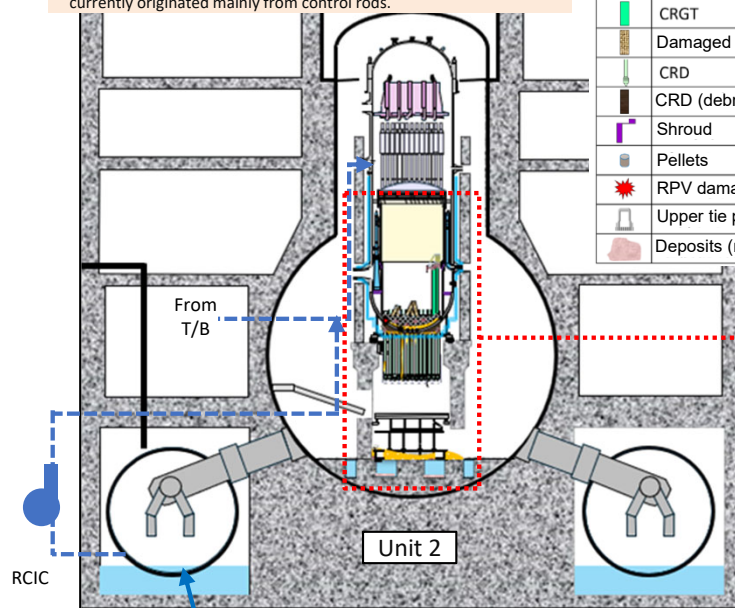
## Seawater

- 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.

|  |                                    |
|--|------------------------------------|
|  | 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)       |



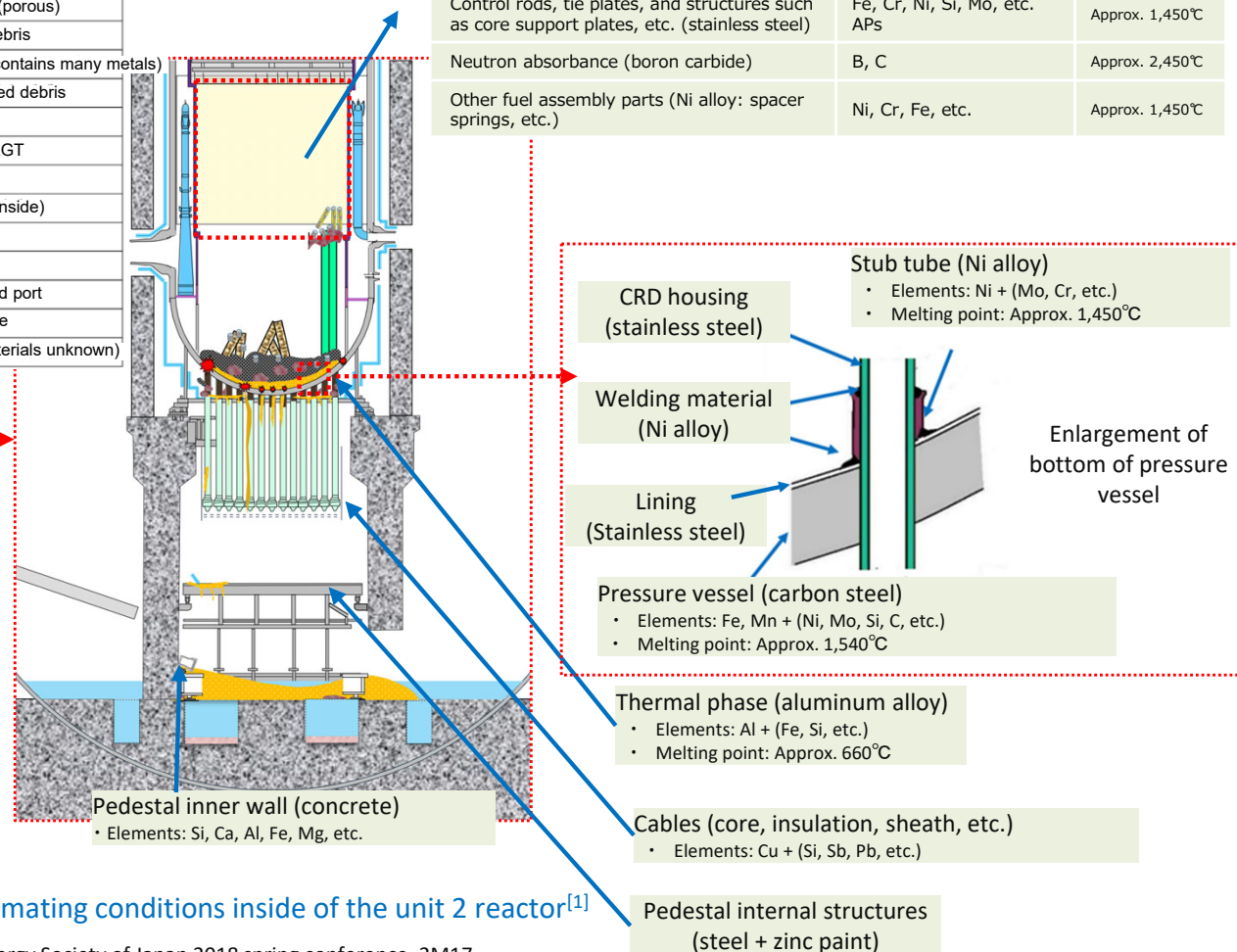
## Inorganic zinc paint

- Elements: Zn, Si, Al + (Cl, K, etc.) <sup>2)</sup>
- Element elution during the heating of water ( $140^\circ\text{C} \times 30\text{h}$ ) <sup>2)</sup>

## Epoxy sealants

- Elements: Ti, Ba, Si, Al, S, etc. <sup>3)</sup>

| Major core materials                                                                         | Major elements               | Singular melting point        |
|----------------------------------------------------------------------------------------------|------------------------------|-------------------------------|
| Uranium fuel                                                                                 | U, Gd, Pu, FPs, MAs          | Approx. $2,800^\circ\text{C}$ |
| Cladding tubes/CB (Zirconium alloy)                                                          | Zr, Sn, etc. APs             | Approx. $1,760^\circ\text{C}$ |
| Control rods, tie plates, and structures such as core support plates, etc. (stainless steel) | Fe, Cr, Ni, Si, Mo, etc. APs | Approx. $1,450^\circ\text{C}$ |
| Neutron absorbance (boron carbide)                                                           | B, C                         | Approx. $2,450^\circ\text{C}$ |
| Other fuel assembly parts (Ni alloy: spacer springs, etc.)                                   | Ni, Cr, Fe, etc.             | Approx. $1,450^\circ\text{C}$ |



estimating conditions inside of the unit 2 reactor<sup>[1]</sup>

[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, Deliberations of TEPCO Fukushima Daiichi Nuclear Power Station Accident Analysis (28th meeting) Document 4-1. February 28, 2022 (SEM-EDX results)

4) IRID, JAEA, (39th) Secretariat of the Team for Countermeasures for Decommissioning, Contaminated Water and Treated Water Treatment, metrial3-4-4, February 23, 2017. (Analysis results of stagnant water in the PCV)

[1] JAEA, Decommissioning/contaminated water/treated water countermeasure project beginning in 2023 (development of analysis/estimating technologies for ascertaining the attributes of still debris) 3. The development of technologies for estimating RPV damage and the behavior of migrating 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 whether the location of the sample after fluctuation can be obtained.

[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, TEPCO HD, Fuel debris Sample Non-destructive analysis results, Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (138<sup>th</sup>), 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.                  |

[3] JAEA, TEPCO HD, Fuel debris Sample Non-destructive analysis results, Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (140<sup>th</sup>), July 31, 2025.

| 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.                                                                                                                   |

[3] JAEA, TEPCO HD, Fuel debris Sample Non-destructive analysis results, Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment (140<sup>th</sup>), July 31, 2025.



## ① Element content

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- ◆ In order to identify and quantify the elements contained in the fuel debris [block \[D\]](#) and [particles \[B\]](#) obtained through crushing, the specimens were fractionated, dissolved (approx. 0.1g each specimen), and analyzed.

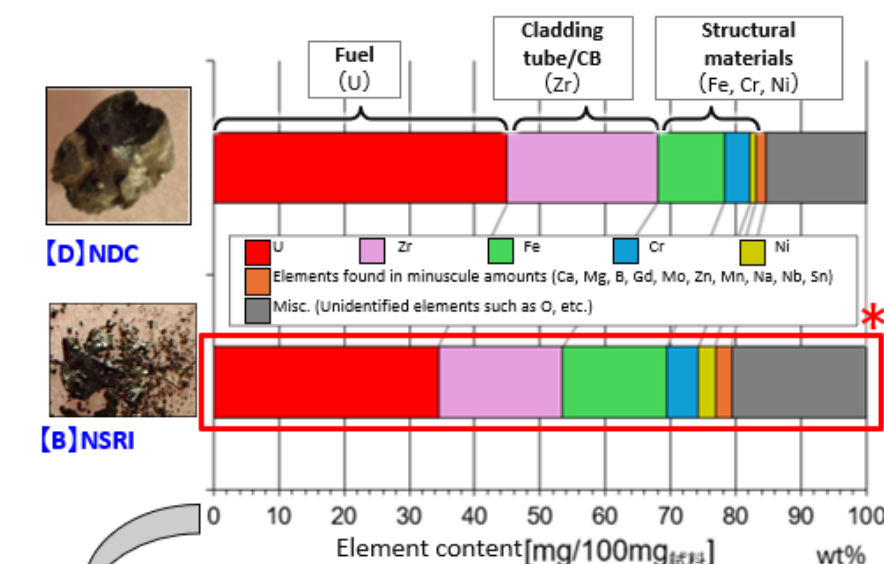


Figure 1 Elemental composition of the sample (ICP-AES and ICP-MS analysis results)

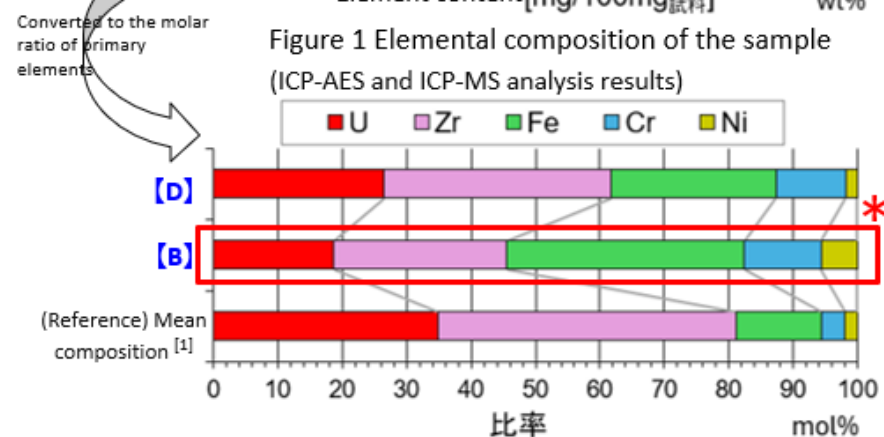


Figure 2 Ratio of major elements (U, Zr, Fe, Cr, Ni)

[1] Sato et al., Nucl.Eng.Des., 404, 112205. Converted to element mass based on Table1 fuel, cladding tube/CB and control rod blade material composition

- U, Zr, Fe, Cr and Ni were the primary elements in all samples. The ratio of U was highest compared to sample mass. (Figure 1)
- The ratio of structural material elements (Fe+Cr +Ni) was higher than the initial mean composition of fuel, cladding tubes/CB, and control rod blades in Unit 2. (Figure 2)

⇒ It is possible that after the fuel and cladding tubes, etc. melted in the core, these elements were generated as other materials got caught up in the molten material as it migrated to the PCV.

- The following elements were also found in minuscule amounts: Ca, Mg, B, Gd, Mo, Zn, Mn, Na, Nb, Sn. (All at amounts less than 1wt% of the sample mass)

⇒ In light of the structural materials inside the PCV and RPV as well as conditions during the accident, it is possible that these elements originate from seawater elements, as well as structural elements and the paint, etc. they were covered with.

- The ratio of fuel elements (uranium) differed in the parts of the sample that remained in block [\[D\]](#) and the parts that were turned to particles [\[B\]](#) during crushing (Figure 2).

⇒ [\[B\]](#) suggests a larger ratio of the micro-mixed phase (Considering ④ Element, compound distribution)

Note: CB: Channel box, SUS: Stainless steel

\* The graph was updated based on a re-evaluation of the analytical value for U on August 27, 2026.

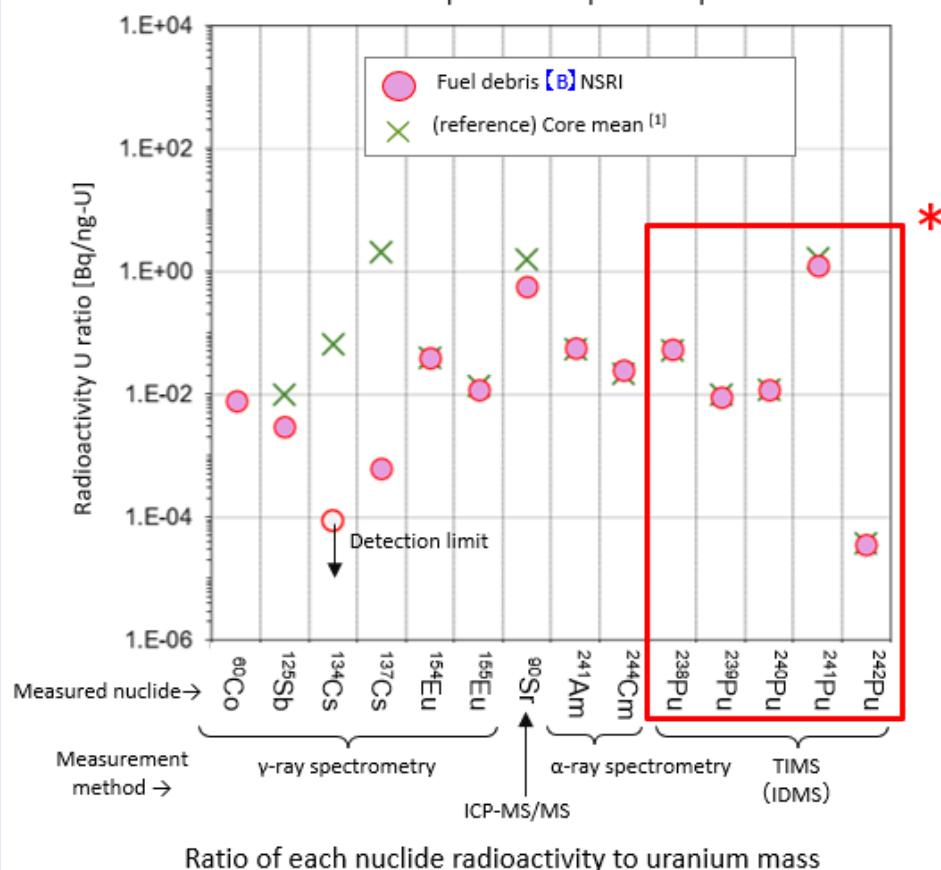


### ③ Radioactivity concentration

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- ◆ In order to ascertain primary radioactive nuclides, fractionated specimens of **particles [B]** obtained through crushing were dissolved and the concentration of radioactivity in the solution was measured using  $\gamma$ -ray spectrometry and  $\alpha$ -ray spectrometry, etc. In order to assess the association with uranium, which is the primary element in fuel, the ratio of nuclide radioactivity to uranium mass was calculated and compared with past samples and core means.



#### ○ Major radioactive nuclides

- $^{90}\text{Sr}$ ,  $\alpha$  nuclides ( $^{241}\text{Am}$ ,  $^{244}\text{Cm}$ , and Pu isotopes),  $^{154}\text{Eu}$  and  $^{60}\text{Co}$  contribute greatly to the radioactivity in fuel debris samples. (Left figure)
- The contribution of  $^{134}\text{Cs}$  and  $^{137}\text{Cs}$  is small, and less than core means.

⇒ It is possible that during the accident, radioactive Cs volatilized due to the high temperatures and formed fuel debris with low  $\gamma$  dose rates.

#### ○ Accompanied condition of U

- The ratio of Sr, Eu, Pu, Am, Cm to U is very similar to core means.

⇒  $^{154}\text{Eu}$  and  $^{244}\text{Cm}$ , which are searched\* for to detect fuel debris, were found to associate with U.

\* Because they are known to associate with U after normal nuclear reactor operation.

\* The graph was updated based on a re-evaluation of the analytical values for U and Pu on August 27, 2026.



## [Reference] Liquid analysis results

— Element content ratio/isotope ratio/radioactivity concentration —

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## ◆ JAEA NSRI liquid analysis

## ○ Element content ratio → Chart 1

- Approx. 0.1g of [particles\[B\]](#) was dissolved in an alumina crucible with alkaline flux<sup>※</sup>, and the resulting molten material was dissolved in hot nitric acid.
  - ※ Alkali melting: By taking fuel debris, which is difficult to dissolve in acid, and heating/dissolving it in alkaline flux (sodium peroxide) to convert it to a substance that is easily dissolved in acid, we can obtain a uniform solution without undissolved residue.
- The amount of the elements contained in the obtained solution was measured using ICP-AES to assess the element content ratio of the sample.
- U, Pu, Nd and Gd were separated from the solution using UTEVA<sup>®</sup> resin and anion exchange resin after which IDMS was used to measure the amount of the elements in the separated solutions.

## ○ Isotope ratio (U, Pu, Gd and Nd) → Chart 2

- The separated solutions of each element obtained in "Element content ratio" above were then measured using TIMS.

## ○ Radioactivity concentration → Chart 3

- For γ-ray emitting nuclides, a small amount was fractionated from the obtained solution to measure radioactivity with γ-ray spectrometry.
- For <sup>241</sup>Am and <sup>244</sup>Cm, TRU resin was used to separate Am and Cm from the obtained solution and α-ray spectrometry was used to measure the radioactivity of the separated solution.
- For <sup>90</sup>Sr, Sr resin was used to separate Sr from the obtained solution and ICP-MS/MS was used to measure the amount of <sup>90</sup>Sr isotopes in the obtained solution.

Chart 1 Element content ratio assessment results

k=2 Expanded uncertainty

| Element          | Element content ratio<br>[mg/100mg Specimen] | Notes                                          |
|------------------|----------------------------------------------|------------------------------------------------|
| U                | 34.48 ± 0.64 *                               | IDMS measurement                               |
| Zr               | 19 ± 2                                       |                                                |
| Fe               | 16 ± 1                                       |                                                |
| Cr               | 4.8 ± 0.2                                    |                                                |
| Ni               | 2.6 ± 0.1                                    |                                                |
| Si <sup>※1</sup> | 0.05 ± 0.01 <sup>※1</sup>                    | Reference value <sup>※1</sup>                  |
| Ca               | 0.29 ± 0.02                                  | Revised value for the amount of Ca in the flux |
| Al               |                                              | Excluded since it originates from the crucible |
| Mg <sup>※1</sup> | 0.16 ± 0.01 <sup>※1</sup>                    | Reference value <sup>※1</sup>                  |
| B <sup>※1</sup>  | 0.05 ± 0.01 <sup>※1</sup> *                  | Reference value <sup>※1</sup>                  |
| Gd               | 0.294 ± 0.012 *                              | IDMS measurement                               |
| Mo               | 0.21 ± 0.01                                  |                                                |
| Sb               |                                              | Undetected by ICP-AES qualitative analysis     |
| Pb               |                                              | Undetected by ICP-AES qualitative analysis     |
| Zn               | 0.08 ± 0.01                                  |                                                |
| Ti               | 0.02 ± 0.01                                  |                                                |
| Mn               | 0.43 ± 0.02                                  |                                                |
| Na               |                                              | Excluded since it originates from the flux     |
| Nb               |                                              | Undetected by ICP-AES qualitative analysis     |
| Sn               | 0.13 ± 0.01 *                                |                                                |
| Pu               | 0.201 ± 0.008                                | IDMS measurement                               |
| Nd               | 0.114 ± 0.005                                | IDMS measurement                               |

※1 In regards to the element concentration in the solution the concentration ratio of operational gaps to solution is 0.1 or higher, reference value.

\* The table was updated based on a re-evaluation of the analytical values for U, Pu, Nd, and Gd determined by IDMS on August 27, 2026.



## [Reference] Liquid analysis results

— Element content ratio/isotope ratio/radioactivity concentration —

TEPCO

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## ◆ JAEA Oarai liquid analysis (cont.)

## ○ Radioactivity concentration

- $\gamma$ -ray spectrometry
  - $^{60}\text{Co}$ ,  $^{125}\text{Sb}$ ,  $^{137}\text{Cs}$ ,  $^{154}\text{Eu}$  and  $^{241}\text{Am}$  were detected. (Figure 1 top. Same nuclides that were detected during non-destructive analysis)
  - The same nuclides were detected in the undissolved residue, but the intensity of  $^{60}\text{Co}$  was relatively high. (Figure 1, bottom)
- $\alpha$ -ray spectrometry
  - $^{242}\text{Cm}$ ,  $^{243}\text{Cm}+^{244}\text{Cm}$ ,  $^{241}\text{Am}+^{238}\text{Pu}$  and  $^{239}\text{Pu}+^{240}\text{Pu}$  were detected in the solution

Chart 3 Radioactivity concentration assessment results

k=2 Expanded uncertainty

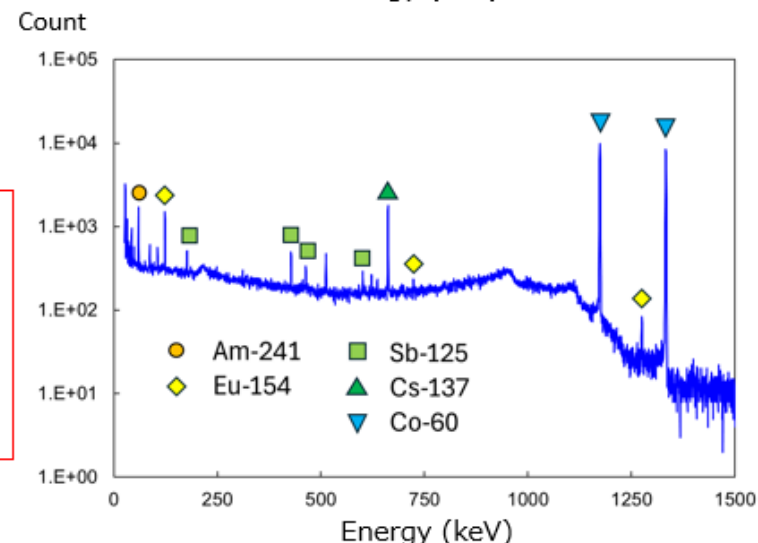
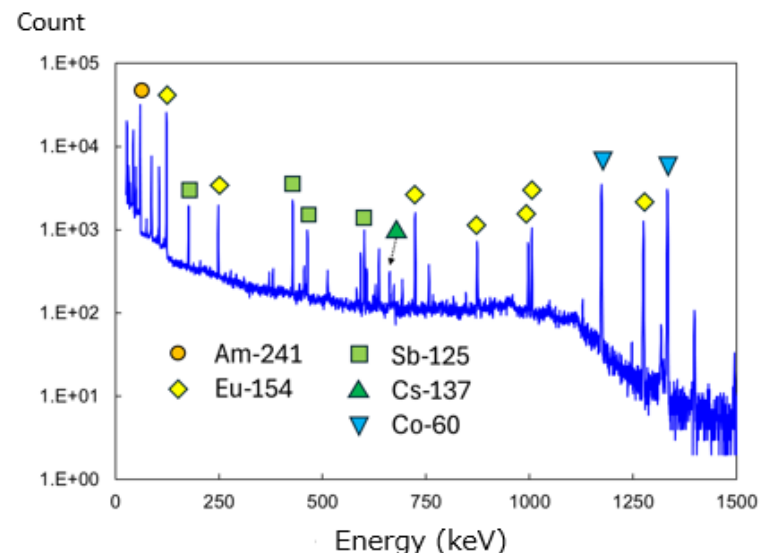
| Nuclide                           | Radioactivity concentration <sup>※1</sup><br>[MBq/g-specimen] |
|-----------------------------------|---------------------------------------------------------------|
| $^{60}\text{Co}$                  | $9 \pm 2$                                                     |
| $^{125}\text{Sb}$                 | $6 \pm 1$                                                     |
| $^{137}\text{Cs}$                 | $0.30 \pm 0.09$                                               |
| $^{154}\text{Eu}$                 | $10 \pm 3$                                                    |
| $^{241}\text{Am}$                 | $13 \pm 3$                                                    |
| $^{242}\text{Cm}$                 | $0.04 \pm 0.03$                                               |
| $^{243}\text{Cm}+^{244}\text{Cm}$ | $8 \pm 2$                                                     |
| $^{241}\text{Am}+^{238}\text{Pu}$ | $25 \pm 5$                                                    |
| $^{239}\text{Pu}+^{240}\text{Pu}$ | $6 \pm 1$                                                     |

\* The table was updated based on a re-evaluation of the radioactivity concentration values obtained by gamma-ray spectrometry and alpha spectrometry on August 27, 2026.

※1 Values as of the date of measurement

\* For  $^{60}\text{Co}$ ,  $^{125}\text{Sb}$ ,  $^{137}\text{Cs}$ ,  $^{154}\text{Eu}$  and  $^{241}\text{Am}$ , small samples were fractionated from the obtained solution and radioactivity was measured using  $\gamma$ -ray spectrometry. (Measurement date: April 17, 2025)

\* For  $^{242}\text{Cm}$ ,  $^{243}\text{Cm}+^{244}\text{Cm}$ ,  $^{241}\text{Am}+^{238}\text{Pu}$  and  $^{239}\text{Pu}+^{240}\text{Pu}$ , small amounts were taken from the obtained solution and smeared on a SUS plate after which  $\alpha$ -ray spectrometry was used to measure the radioactivity of each nuclide. (Measurement date: May 13, 2025)


Figure  $\gamma$ -ray spectrometry Top: Solution, Bottom: Undissolved residue

Top: Measurement date: March 17, 2025, measurement time: 5,000 seconds,  
bottom: measurement date: March 18, 2025, measurement time: 10,000 seconds



## [Reference] Liquid analysis results — Comparison with PCV penetration (X-6) sample —

**TEPCO**

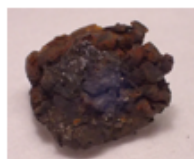
35

Material Adhesions to X-6 penetration investigation device  
(Obtained in 2020)



Smear samples taken from the surface of the end of the investigation device. A portion of the smear paper was cut into batches and subjected to liquid analysis.

Fuel debris sample



(Obtained in FY2024)

Sampled during first trial retrieval. Crushed, fractionated and subjected to liquid analysis.

Obtained sample



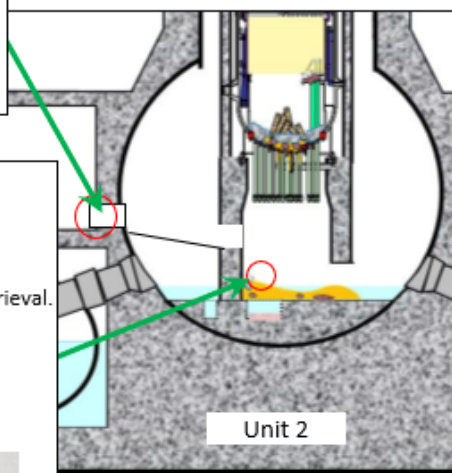
Particles [B]



Block [D]



Particles [A-2]



Unit 2

- It is presumed that the PCV penetration (X-6 penetration) contains substances originating from fuel that migrated from the pressure vessel to the pedestal during the accident. Uranium isotopic composition and element ratio were compared for reference when generating a hypothesis about the process by which the fuel debris sample was created.
- U isotopic composition: Same as the core mean (refer to page 8) and the X-6 penetration sample. (see the chart below)
- Zr/U ratio (atomic ratio): Percentage of Zr is higher than the core mean (approx. 1.3. Refer to page 7) and the X-6 penetration sample. (see the chart below)
- Gd/U ratio: Core mean is **approx. 0.85wt%** of U weight while the measurement results from the fuel sample show approx. around 0.80wt%. (No quantitative data for the X-6 penetration sample)

Liquid analysis results (example of comparison between fuel debris sample and X-6 penetration sample)

| Sample                                                                      |                      | U isotopic composition [at%] |                    |                    | Zr/U ratio<br>(Atomic ratio) | Gd/U ratio<br>[wt%]                       |
|-----------------------------------------------------------------------------|----------------------|------------------------------|--------------------|--------------------|------------------------------|-------------------------------------------|
|                                                                             |                      | <sup>234</sup> U/U           | <sup>235</sup> U/U | <sup>236</sup> U/U |                              |                                           |
| Fuel debris sample                                                          |                      |                              |                    |                    |                              |                                           |
|                                                                             | Particles [B]        | 0.027±0.001                  | 1.943±0.002        | 0.342±0.001        | 1.4±0.2                      | 0.85±0.04                                 |
|                                                                             | Block [D]            | 0.026±0.002                  | 1.9±0.1            | 0.33±0.002         | 1.33±0.08                    | 0.9±0.1                                   |
|                                                                             | Particles [A-2]      | 0.027±0.002                  | 1.93±0.01          | 0.35±0.01          | 1.6±0.3                      | 0.75±0.04                                 |
| Material adhered to X-6 penetration investigation device (Data source: [1]) |                      |                              |                    |                    |                              |                                           |
|                                                                             | Material adhered - 2 | 0.03±0.03                    | 1.91±0.03          | 0.34±0.03          | 1.03±0.02                    | No data since results Gd was not measured |

\* The evaluation results of the Zr/U and Gd/U ratios were updated based on a re-evaluation of the analytical values for U and Gd on August 27, 2026.



# [Reference] Solid analysis results

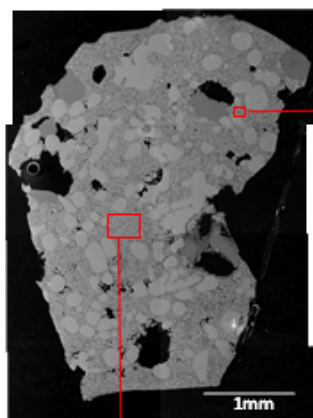
## — Cross-section observations/composition assessment —

TEPCO

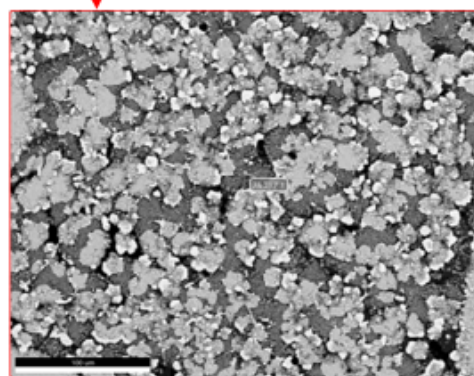
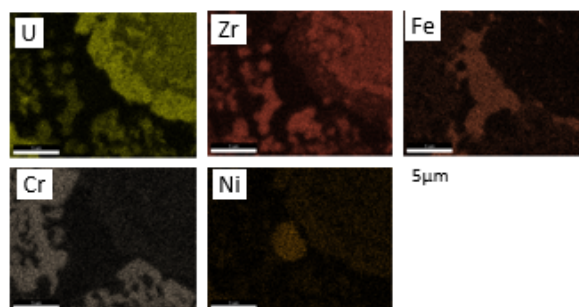
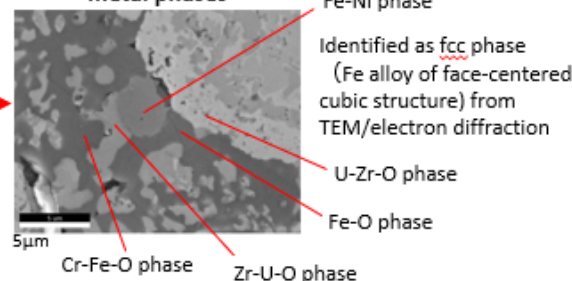
42

Block [C] : NFD

(C)Micro-mixed phase



Example of the inclusion of microscopic Fe-Ni metal phases

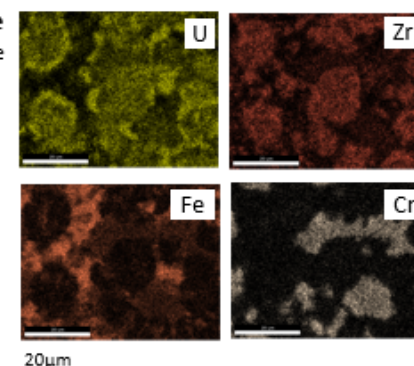
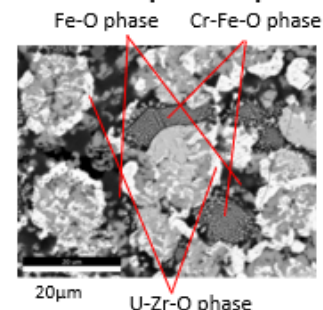


100μm

SEM-EDX facet analysis results of micro-mixed phase

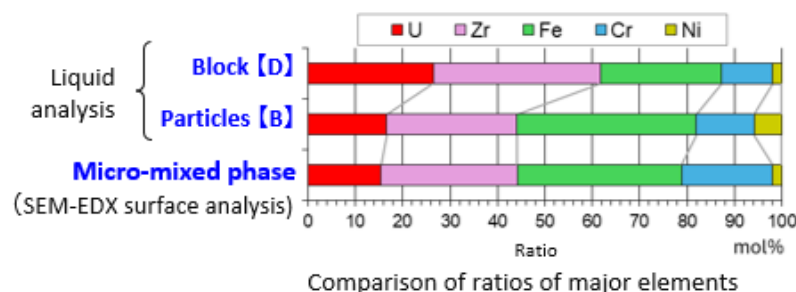
| Major elements | Facet analysis                |
|----------------|-------------------------------|
| O              | 74                            |
| Cr             | 4                             |
| Fe             | 8                             |
| Ni             | <0.5 (Exist in small amounts) |
| Zr             | 9                             |
| U              | 4                             |

Example of microscopic oxide phase



- U-Zr-O phases, Cr-Fe-O phases, and Fe-O phases found mixed into micro-mixed phases in addition to small (~several μm) phases of Zr-U-O and Fe-Ni.

⇒ It is hypothesized that precipitation of the Zr-U-O phase continued and turned into molten oxide material with high concentrations of Fe and Cr, after which each phase split apart, cooled, and solidified.



- The U:Zr:Fe:Cr:Ni ratio of the micro-mixed phase is close to the composition of particles [B] after pulverization, indicating the possibility that powder was easily obtained through crushing.

\* The table and graph were updated based on the re-evaluation of the U analytical result for Powder [B] and the semi-quantitative result for Lump [C] on August 27, 2026.

