This report is related to the FY2023 Subsidized Project of Decommissioning, Contaminated Water and Treated Water Management (Development of Analysis and Estimation Technology for Characterization of Fuel Debris).

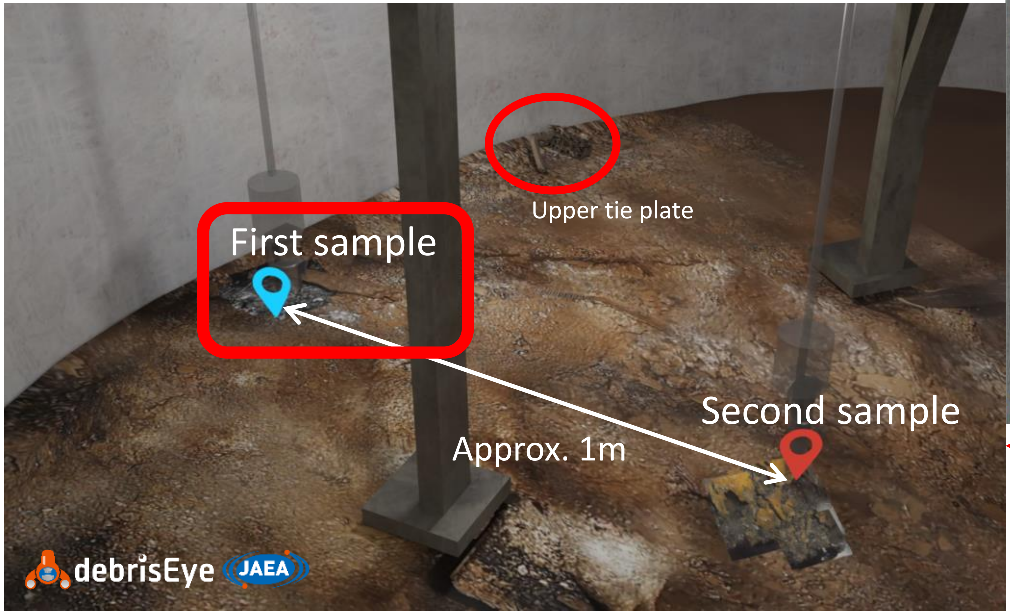
Analysis results of the first fuel debris sample (Follow-up report)

November 27, 2025

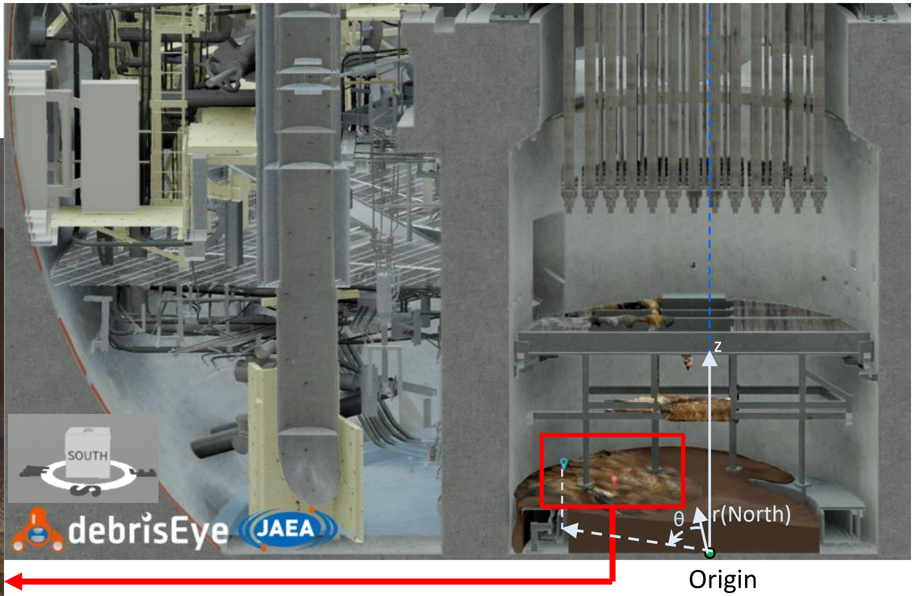
Japan Atomic Energy Agency
Tokyo Electric Power Company Holdings, Inc.

Background information

- On November 12, 2024, the first fuel debris sample taken during trial fuel debris retrieval was received at the JAEA Oarai Nuclear Engineering Institute’s Irradiated Fuel Monitoring Facility (FMF).
- To date, reports on major analysis have been given during Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting / Secretariat Meetings on December 26, 2024, January 30, 2025 and July 31, 2025. [1][2][3]
- At this meeting, we will report on the major results of detailed analysis that have been obtained since then (follow-up on estimation of formation process of fuel debris).



Fuel debris sampling locations on the floor inside the Unit 2 pedestal



Estimates about the sampling locations

	r (Horizontal distance from the center)	Θ(Angle from the north)	Z(Height from the floor)
First sample	Approx. 2.4 m	Approx. 54°	Approx. 0.9 m
Second sample	Approx. 1.4 m	Approx. 42°	Approx. 0.8 m

※ Expressed on a cylindrical coordinate system with the center of the floor of the pedestal as the origin.
※ There may be discrepancies in the height of the fuel debris deposit since it was measured by creating a model based on how much the deposits hide the legs of the platform.

[1] 133rd Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting / Secretariat Meeting 3-3

[2] 134th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting / Secretariat Meeting 3-3

[3] 140th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting / Secretariat Meeting 3-3

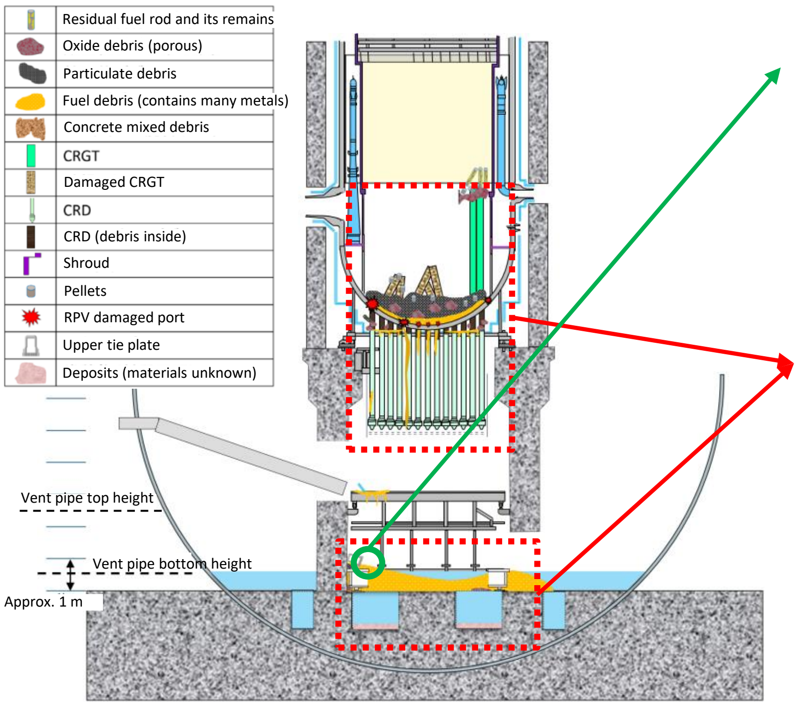
Analysis purpose of the fuel debris sample

- By analyzing the obtained sample, grasp the condition of the sampled area to estimate the formation process of the fuel debris.

⇒ More precise estimation of the condition inside the core will become the basis for review of full-scale fuel debris retrieval to safely retrieve fuel debris and realize thoroughly managed stable storage.

<Example of incorporating “estimation of the condition inside the core” into “review of fuel debris retrieval methods”>

- ➢ Estimate hardness of fuel debris → select retrieval methods and tools
- ➢ Possibility of criticality of fuel debris → review safety measures and storage methods



Estimated drawing of the condition inside the core [1]
(View from southwest to northeast)

1. Grasping the condition of the sampled area (Grasping the condition of fuel debris samples)

- 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)

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[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 (Development of Technology for Estimating RPV damage status and fuel debris migration behavior inside the PCV)).

Analysis items and evaluation details of the fuel debris sample

1. 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 ・External appearance, weight, dose rate, density distribution	・Exterior, weight, dose rate measurement ・Imaging plate (IP) ・X-ray CT	Organization of basic information	Basic information to review retrieval (existence and mount of pores, etc.)
Element content (elemental composition)	・ICP-MS, ICP-AES	Content of fuel components Origin of major components	Basic information to review safety measures at retrieval, such as criticality evaluation, and storage methods
Isotope ratio	・TIMS ・SIMS	U isotope ratio	
Radioactive concentration	・γ-ray spectrometry ・α-ray spectrometry	Accompanied condition of U with focal nuclides	Information to review technology development for non-destructive measurement at fuel debris retrieval
Element and compound distribution	・SEM-EDX、SEM-WDX ・TEM-EDX	Evaluation of distribution of elements and compounds (including pores)	Basic information to review retrieval methods and tools (estimation of hardness, toughness, etc.)

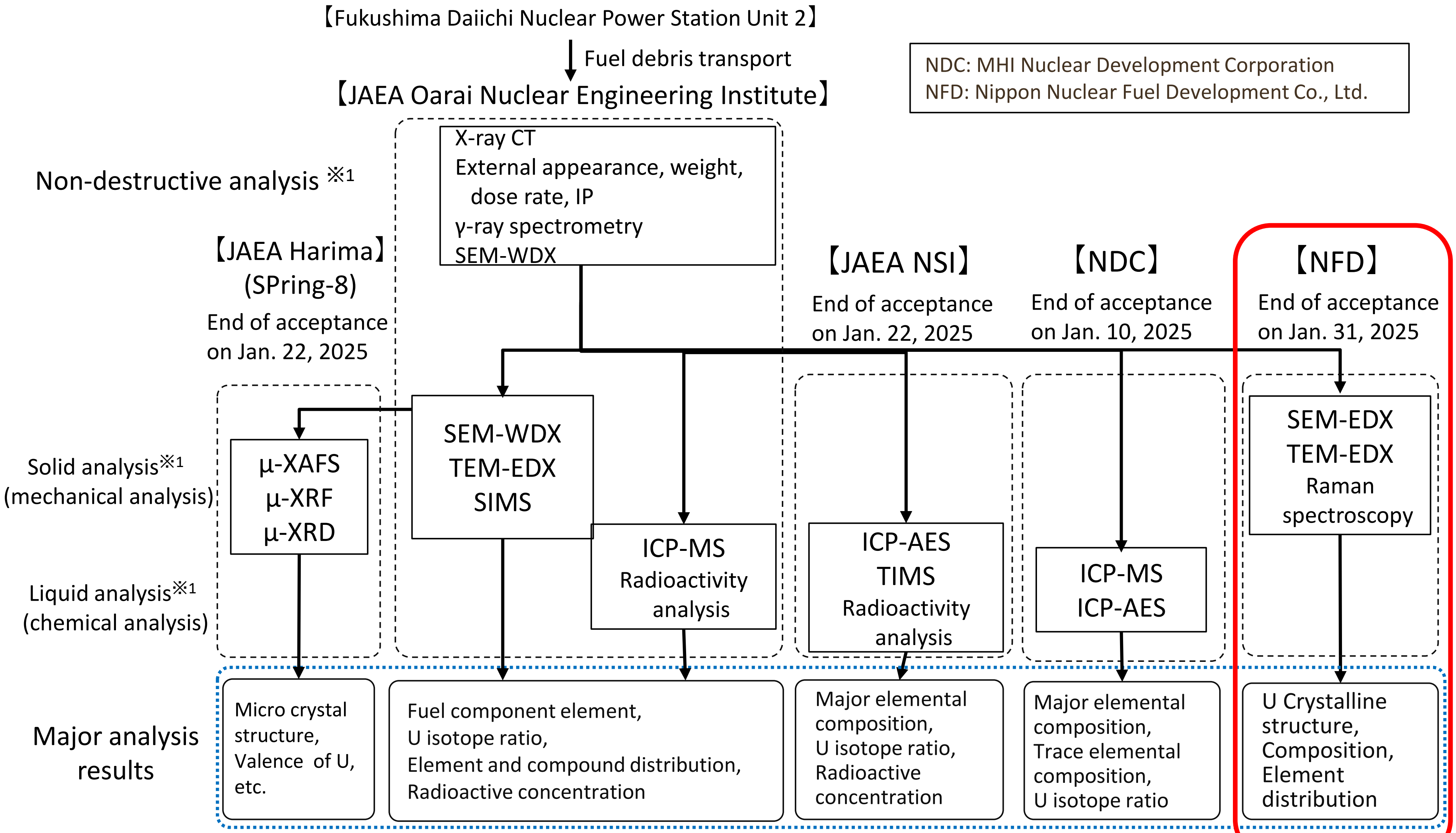
2. Estimation of formation process of fuel debris

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

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

Overall flow of the analysis of the fuel debris sample

- The following is a report on the hypothesis about the mechanism by which fuel debris was created based on analysis results to date as well as SEM-EDX, TEM-EDX, and Raman spectroscopy (composition and crystalline structure estimates) that have been continuously conducted.



【Fukushima Daiichi Nuclear Power Station Unit 2】

Fuel debris transport

【JAEA Oarai Nuclear Engineering Institute】

NDC: MHI Nuclear Development Corporation
NFD: Nippon Nuclear Fuel Development Co., Ltd.

Non-destructive analysis ※1

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

【JAEA Harima】 (SPring-8)
End of acceptance on Jan. 22, 2025

【JAEA NSI】
End of acceptance on Jan. 22, 2025

【NDC】
End of acceptance on Jan. 10, 2025

【NFD】
End of acceptance on Jan. 31, 2025

Solid analysis※1 (mechanical analysis)

Liquid analysis※1 (chemical analysis)

μ-XAFS
μ-XRF
μ-XRD

SEM-WDX
TEM-EDX
SIMS

ICP-MS
Radioactivity analysis

ICP-AES
TIMS
Radioactivity analysis

ICP-MS
ICP-AES

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

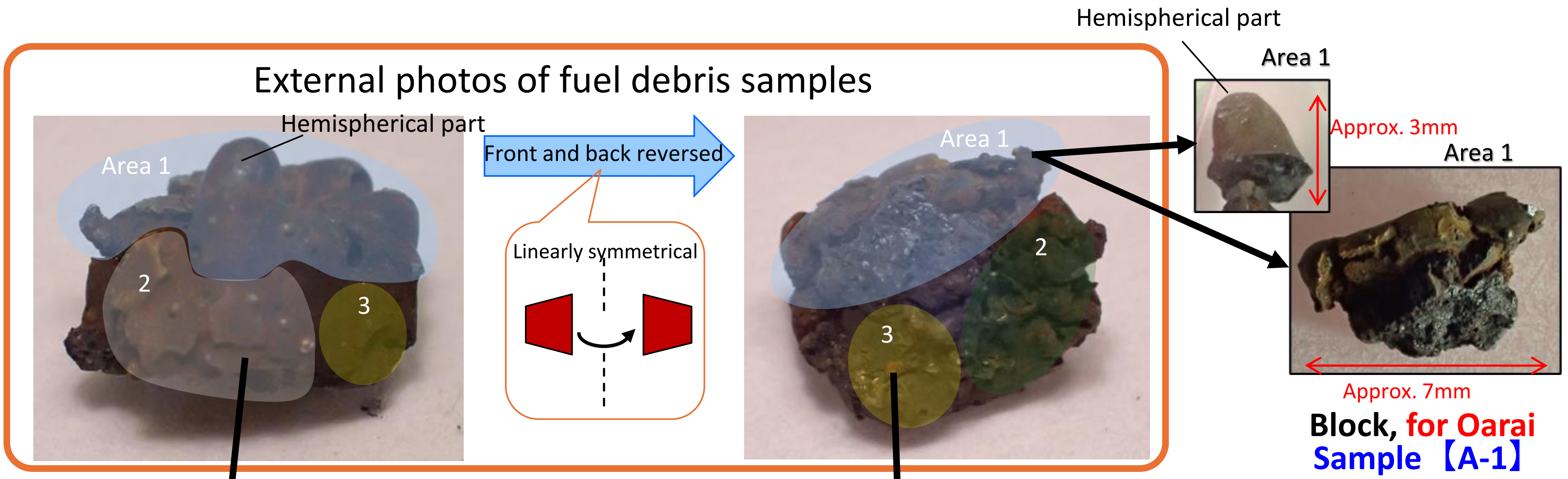
Major elemental composition, U isotope ratio, Radioactive concentration

Major elemental composition, Trace elemental composition, U isotope ratio

U Crystalline structure, Composition, Element distribution

※1 refer to the reference materials for the outline and purpose of each analysis

Fuel debris sample fractionation and transported samples [1]



Block, for Oarai
Sample【A-1】

Block, for NDC
Sample 【D】

Block + Particles, for Nuclear Science Research Institute
Sample【B】

Block, for NFD
Sample【C】

Block + Particles,
For Oarai Nuclear Engineering Institute + SPring-8
Sample【A-2】

- The received sample was reddish brown overall and non-uniform. Part of the surface was black and had shiny regions.
- Mass: 0.693g, Dose rate: (γ rays): Approx. 8mSv/h.
- Hit and crushed with a stainless rod (approx. 250g) and fractionated.

[1] Document 3-3 from the 134th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting / Secretariat Meeting

Estimation of the sample formation process based on analysis results

- Already reported estimation[1] and the focus points.

Fuel and cladding tubes, etc. melt in the core

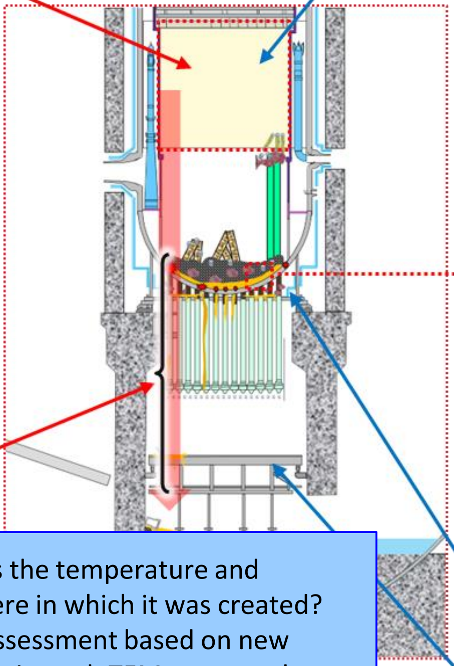
Uranium fuel, zirconium metal and stainless steel, etc., melt together

→Zr-U-O phase, U-Zr-O phase, Fe-Cr-O phase, Fe-O phase, Fe-Ni phase observed

What was the original composition of the molten material?

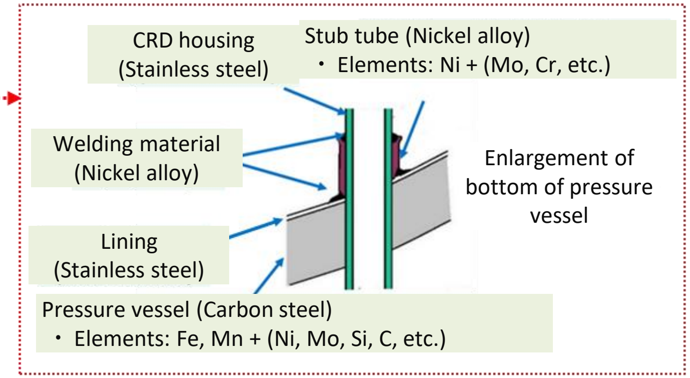
→ 【P8】 shows a further detailed assessment of the elemental composition already reported [1]

Major core materials	Major elements
Uranium fuel	U, Gd, Pu, FPs, MAs
Cladding tubes/CB (Zirconium alloy)	Zr, Sn, etc. APs
Control rods, tie plates, and structures such as core support plates, etc. (stainless steel)	Fe, Cr, Ni, Si, Mo, etc. APs
Neutron absorbance (boron carbide)	B, C
Other fuel assembly parts (Ni alloy: spacer springs, etc.)	Ni, Cr, Fe, etc.



Various materials get mixed in as the molten material migrates and falls into the PCV

- ・CRD housing (stainless steel)
- ・Welding material, stub tubes, (Ni alloy)
- →The percentages of the structural material elements Fe+Cr+Ni are high
- Fe-Ni phase observed
- ・Thermal phase（Al alloy）
- →Al detected



CRD housing
(Stainless steel)

Stub tube (Nickel alloy)
・ Elements: Ni + (Mo, Cr, etc.)

Welding material
(Nickel alloy)

Lining
(Stainless steel)

Enlargement of bottom of pressure vessel

Pressure vessel (Carbon steel)
・ Elements: Fe, Mn + (Ni, Mo, Si, C, etc.)

Insulative layer (Aluminum alloy)
・ Elements: Al + (Fe, Si, etc.)

Pedestal internal structures
(Steel + zinc paint)

What was the temperature and atmosphere in which it was created?

→ An assessment based on new analysis, such TEM, etc. can be found on 【P9～12】

Fuel debris analysis results used to provide more detail on the figure thereby estimating conditions inside the core.

Abbreviations: FPs: Fission Products, Mas: Minor Actinides, Aps: Activation Products

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[1] Edited from document 3-3 from the 140th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting / Secretariat Meeting

Assessment of the major elemental composition of the molten material（Liquid analysis results）

Since the sample is a block, we assumed that the composition is relatively similar to the original molten material from which it formed【D】. So, we compared the percentage of the primary five elements (U, Zr, Fe, Cr, Ni) （Figure 1[1]） with the composition of core internals to estimate the origin.

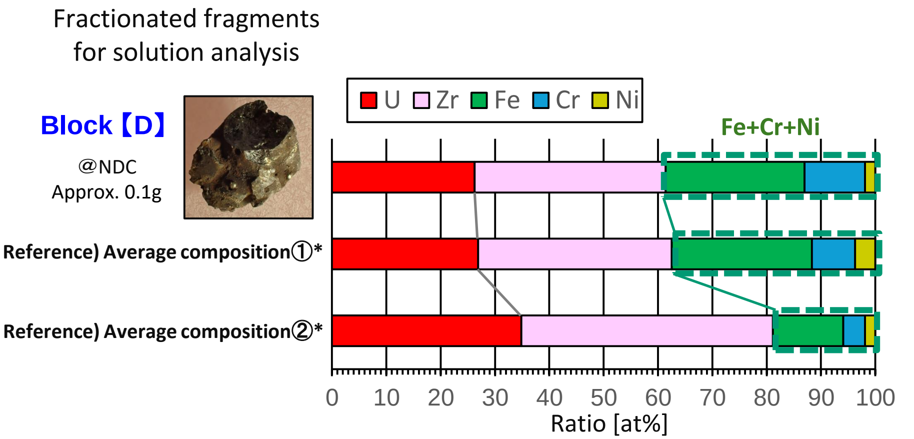
- Percentage of structural elements: Out of the primary five elements, the percentage of Fe+Cr+Ni almost matches the average composition of structures at the bottom of the core.（Refer to the dotted lines in Figure 1 and Figure 2)

⇒ It is estimated that as molten material migrated into the PCV, it mixed with the structural elements of internals at the bottom of the core.

- Zr and U breakdown: The atomic ratio Zr/U in the sample is close to the Zr/U ratio of the core cladding tubes and CB. (Figure 3)

⇒ It is assumed that in addition to the cladding tubes, Zr originating from the CB was entrained in the RPV resulting in molten material that has a higher concentration of Zr than U.

【Estimated mechanism】
- Inside the RPV, molten material with a high concentration of Zr and entrained with the CB, etc. was created, and migrated to the PCV where it mixed with structural material elements of core internals at the bottom.



* Average composition① = ② + core lower SUS (Bottom tie plate, core support plate, speed limiter, etc.) [2]

Average composition② = UO_2＋Zr (Cladding tube, CB, WR)+SUS (control rod blade) [2]

Figure 1 Ratio of the primary five elements based on solution analysis clump【D】[1]

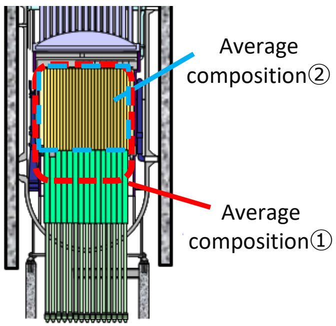
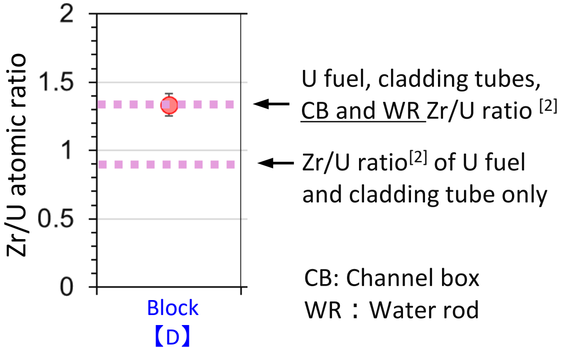


Figure 2 Scope of targeted average composition (Concept diagram)



CB: Channel box

WR：Water rod

Figure 3 Block【D】Zr/U ratio comparison [1]

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[1] Created based on document 3-3 from the 140th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting / Secretariat Meeting

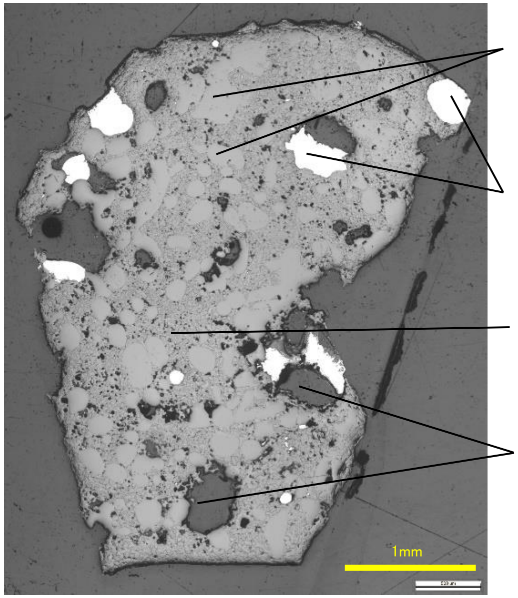
[2] Calculated based on Sato et al., Nucl.Eng.Des., 404, 112205. Table 1

Sample internal formation process (1/4) Cross-section observation results (SEM-EDX analysis results)

- The majority of the internal portion of the sample is comprised of U-Zr oxides, Fe-Cr oxides, and Fe-Ni metals, etc. (refer to the cross-section observation results shown below), and it is assumed to have been formed by oxidization of the structural materials, such as zirconium alloy and stainless steel, etc., after the melting and mixing of these materials with fuel. [1]
- SEM-EDX and TEM-EDX analysis of the unique fine structure and elemental distribution seen during cross-section observation was used to identify the composition of the oxide phases ((A) and (C)) and crystalline structure, and consider the formation process by which each part was formed. （Next pages）

【C】 NFD

External appearance of cross-section
（Refer to the reference materials for info on the cross-section)



Optical micrograph of the cross-section after cutting and polishing

Cross-section observation results [1]

[Classification and summary of constituent phases]

※ The elements contained in each phase is based on SEM-EDX analysis results

(A)Zr-U-O phase （several tens ~ several hundreds μm）

- Zr/U atomic ratio: Approx. 2(Almost consistent regardless of particle)

(B)Fe-Ni metal phase （several ~ several hundreds μm）

- Fe/Ni atomic ratio: Approx. 1～3（Differs depending on particle）

(C)Micro-mixed phase

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

(D)Pore （several μm ~ several hundreds μm）

- Approx. 20% of the cut surface area

Area ratio of each part [%]

(A)Zr-U-O	(B)Fe-Ni	(C)Fine phase	(D)Pore
20	4.4	56	19

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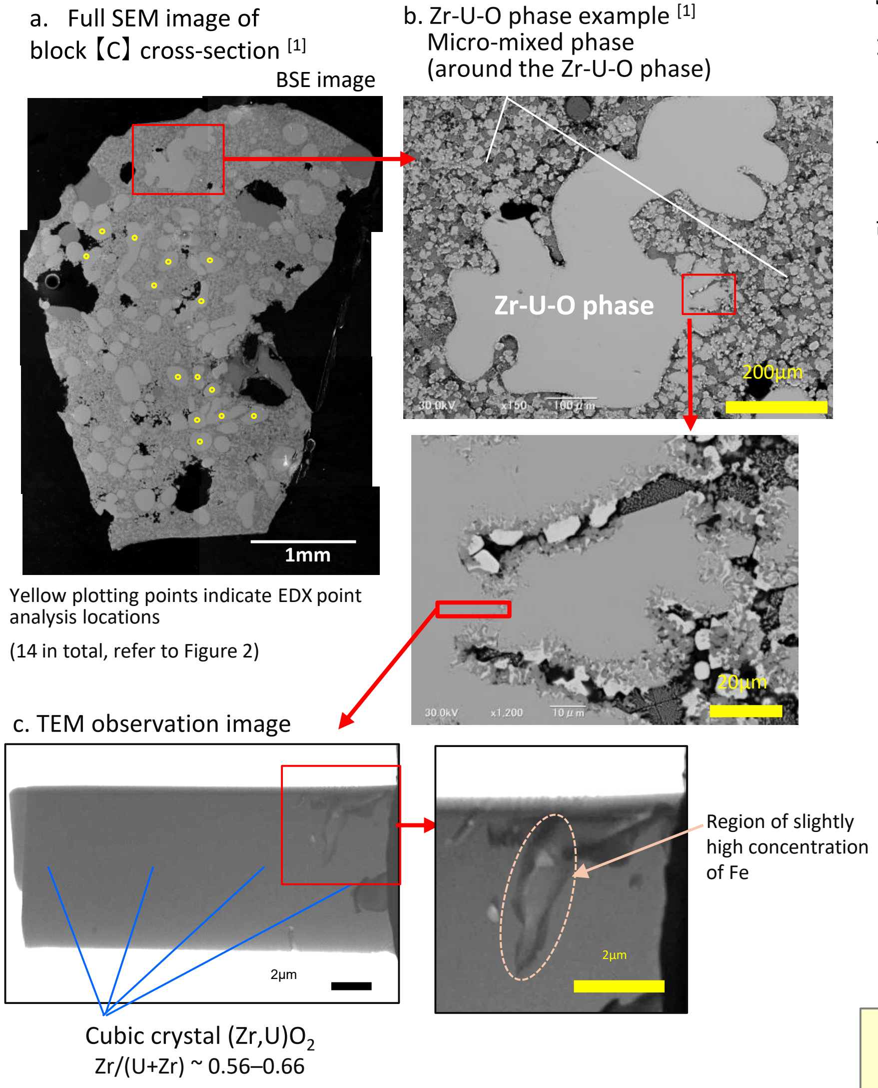
[1] 140th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting / Secretariat Meeting 3-3

Sample internal formation process (2/4)
(A) SEM-EDX analysis and TEM analysis results of Zr-U-O phase

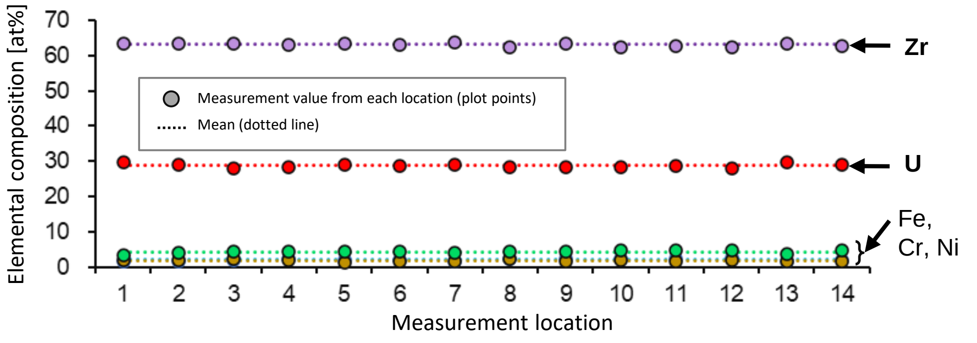
(A)Zr-U-O phase （several tens ~ several hundreds µm）



Yellow plotting points indicate EDX point analysis locations

(14 in total, refer to Figure 2)

Figure 1 Zr-U-O phase SEM and TEM analysis results



Note) Measurement locations 1~14 show the results of EDX point analysis near the center of each different Zr-U-O phase (Refer to Figure 1a). The total of Cr, Fe, Ni, Zr, and U, excluding O, from the detected elements is noted as 100at%

Figure 2 Breakdown of the primary metal elements in the Zr-U-O phase (SEM-EDX)[1]

【Zr-U-O phase attributes】

- Coarse particles of various shapes are distributed throughout the entire cross-section (Figure 1a). And the difference in composition between multiple particles is very small (Figure 2). [1]
  - ⇒ It is possible that they were deposited and grew in around the same temperature range
- The Zr-U-O phase is surrounded by a micro-mixed phase with a high concentration of Fe/Cr. (Figure 1b)
  - ⇒ The Zr-U-O phase has a higher melting point and melts/solidifies quicker. As a result, it is assumed that the amount of Zr-U-O in the liquid portion (mixture of Zr-U-Fe-Cr-O) decreases, leading to higher concentrations Fe and Cr.
- The majority of the inside of the phase is comprised of cubic crystals (Zr,U)O_2, and there are areas of slightly high concentrations of Fe near the outer side. (Figure 1c)
  - ⇒ There is molten material (liquid phase that serves as the origin of the micro-mixed phase) with high concentrations of Fe/Cr around the large cubic crystals (solid phase) and it is possible that the solid phase reacted with the liquid phase at the phase boundaries. (It is assumed that a solid-liquid mixed state was formed)

【Estimated mechanism】

- It is assumed that the Zr-U-O phase formed and grew as the primary crystal from the molten material and coexisted with the liquid phase high Fe-Cr concentration thereby becoming a solid-liquid mixed state.

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[1] 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment, Document 3-3

Sample internal formation process (3/4)
(C) SEM-EDX analysis and TEM analysis results of the micro-mixed phase

(B) Fe-Ni phase

(C) Micro-mixed phase

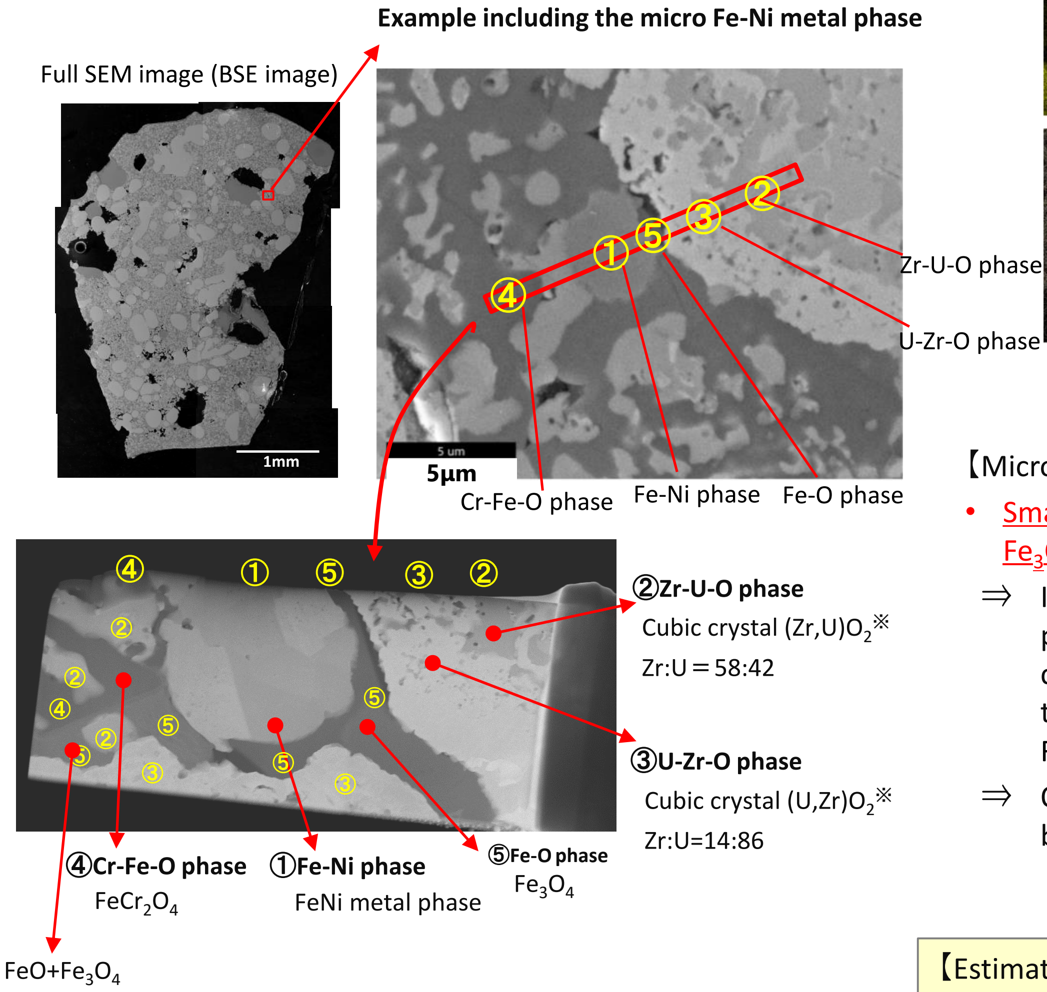


Figure 1 SEM and TEM analysis results of micro-mixed phase

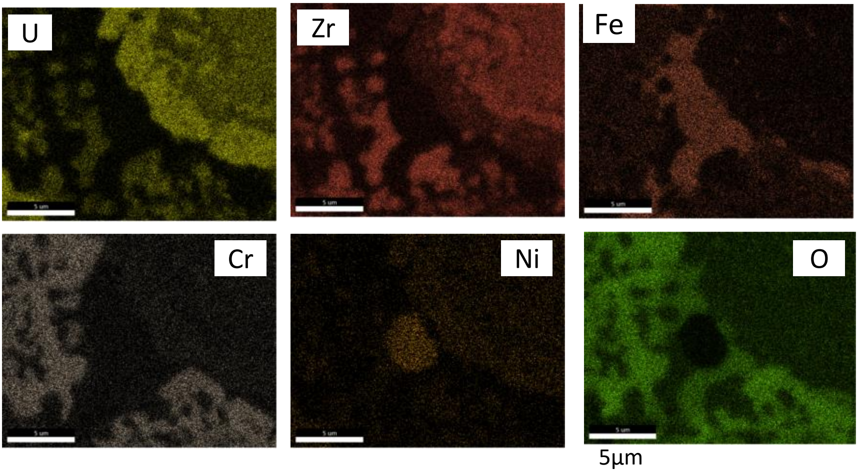


Figure 2 SEM-EDX mapping of micro-mixed phase

【Micro-mixed phase attributes】

- Small (~several μm) particles of $(Zr,U)O_2$, $(U,Zr)O_2$, FeNi metal, Fe_3O_4 and $FeCr_2O_4$ are mixed together. (Refer to Figures 1 and 2)

⇒ It is assumed that after (A) was deposited, these multiple solid phases formed from the liquid phases high in Fe/Cr concentration **【P37】**. It is also assumed that they solidified at a temperature lower than the melting points of Fe_3O_4 and $FeCr_2O_4$ (Approx. 1,600–2,100℃[1,2]).

⇒ Oxide phases containing a lot of Fe were also found at the grain boundary of fuel debris from TMI-2 (lower head) **【P22,23】**

【Estimated mechanism】

- It is assumed that the Zr, U, Fe, Cr oxide phases and the Fe-Ni metal phase formed simultaneously at a temperature lower than the (A)Zr-U-O phase, thereby forming a micro-mixed phase.

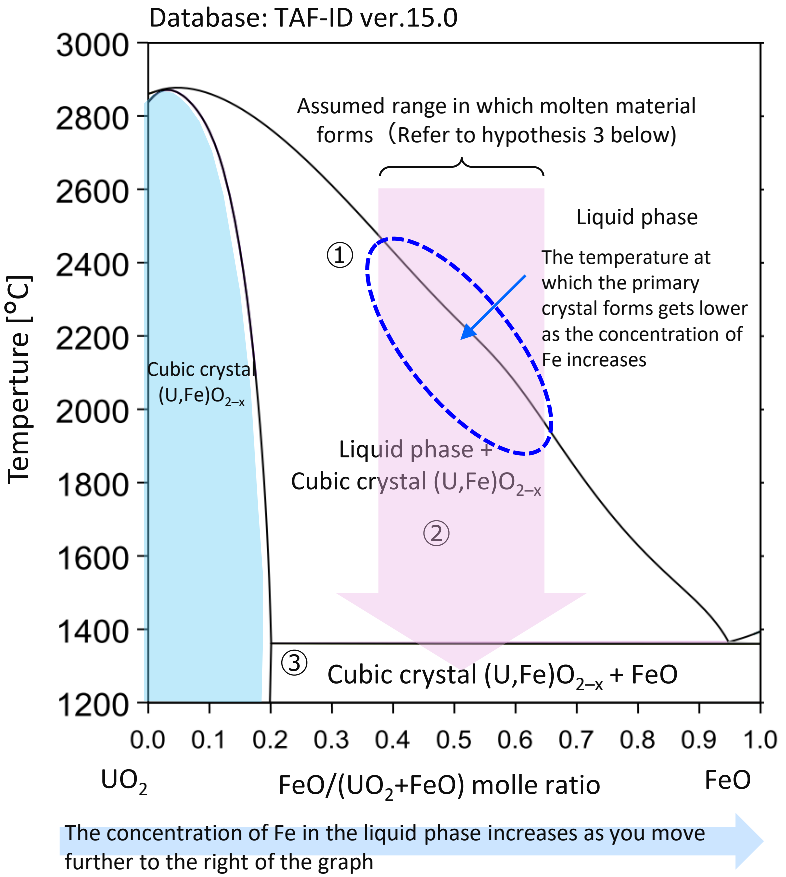
[1] Darken and Gurry, J.Am.Chem.Soc. 68, (1946), 798-816.

[2] Kjellqvist et al., Calphad, 32, (2008), 577-592.

Sample internal formation process (4/4) Estimation of the temperature at which crystal phases identified by the phase diagram formed

- We used a simplified UO_2–FeO phase diagram※ to perform a simple assessment of the temperature at which each phase seen in the analysis results formed.

※ Phase diagram: A graph that shows the stable states of a material under various temperatures and pressure conditions



The concentration of Fe in the liquid phase increases as you move further to the right of the graph

UO_2–FeO pseudo-binary phase diagram

① Approx. 1,900～2,400℃

A cubic crystal forms as the primary crystal from the liquid phase

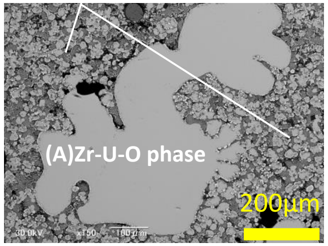
⇒ Addresses (A)Zr-U-O phase (Cubic crystal (Zr,U)O_2) formation

② Solid and liquid phases coexist until approx. 1,350℃

⇒ (A)Zr-U-O phase growth and solid-liquid mixed state

⇒ (C) Addresses U-Zr-Oand Zr-U-O（Cubic crystal (Zr,U)O_2, cubic crystal (U,Zr)O_2) in micro-mixed phase

(C) Micro-mixed phase



Example of Zr-U-O phase and surrounding micro-mixed phase

③ FeO formed at lower than approx. 1350℃

⇒ (C) Addresses Fe-Cr-O and Fe-O（$FeCr_2O_4$, Fe_3O_4, FeNi) in micro-mixed phase

The phase states inside the sample can be qualitatively explained

✓ The assessment above is only tentative and a more thorough investigation will be implemented based on the dissolution of UO_2 to ZrO_2 and the oxide state of Fe

【Simplified hypothesis】

1. It is assumed that Zr is replaced by U, and exists as UO_2
2. It is assumed that Cr and Ni are replaced by Fe, and exist as FeO (The formation of FeO in the phase diagram has been deemed to be the formation of metal Fe and Fe_3O_4(including $FeCr_2O_4$)
3. It is assumed that the percentage of FeO is approx. 40-70at% within the range of Fe+Cr+Ni out of the primary five elements in the sample.

Sample surface conditions
TEM analysis results for layered structure region near the surface

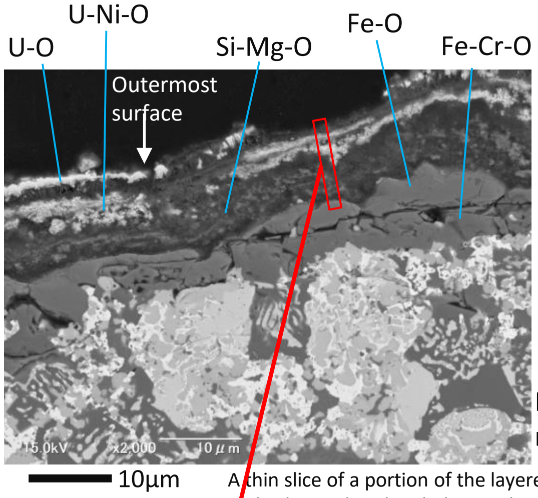


Figure 1 SEM image of area near the surface of the sample [1]

A thin slice of a portion of the layered structure region was produced and subjected to detailed TEM observation

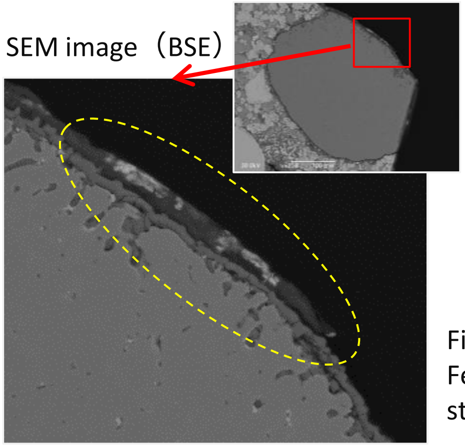
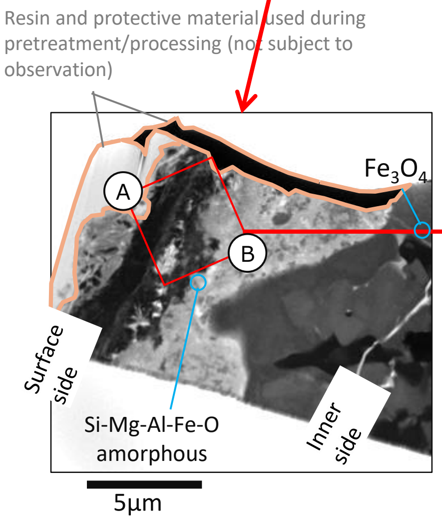


Figure 3
Fe-Ni metal phase surface layered structure (SEM observation results)



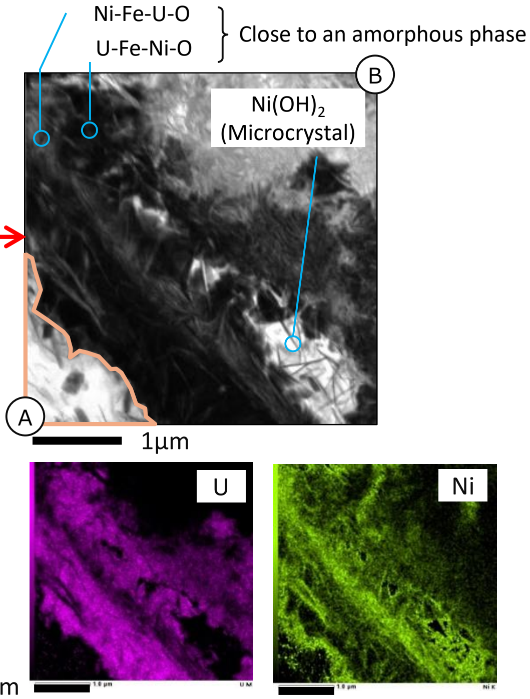


Figure 2 TEM analysis results of layered structure near the surface

- The layered structure region on the sample surface is formed from amorphous layers of varied compositions and microcrystal aggregates (Figures 1, 2)
- A similar layered structure was found on the surface of the Fe-Ni metal phase that contains little U (Figure 3) . However, a similar structure was not seen in the fractured surface (inside original sample)

It is assumed that after the majority of the sample was created through melting and solidification, the layered structure regions on the surface were created through various process.

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[1] 140th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting , Document 3-3

Estimation of the sample formation process based on analysis results

① Melting/mixing of fuel in the core (More than approx. 1,900℃)

- Composition with a high ratio Zr/U **【p.8】**
  → It is assumed that the channel boxes were entrained in addition to the cladding tubes.
- The isotope composition of fuel elements (U、Pu、Nd、Gd) and the ratio of U to ^{154}Eu, ^{241}Am, and ^{244}Cm are approximately the same as the core mean **【p.33,34】**
  → This suggests the possibility that a relatively wide range of fuel was mixed in.
- The chemical composition of the sample requires a temperature of at least more than approximately 1,900℃ to melt **【p.8,12】**

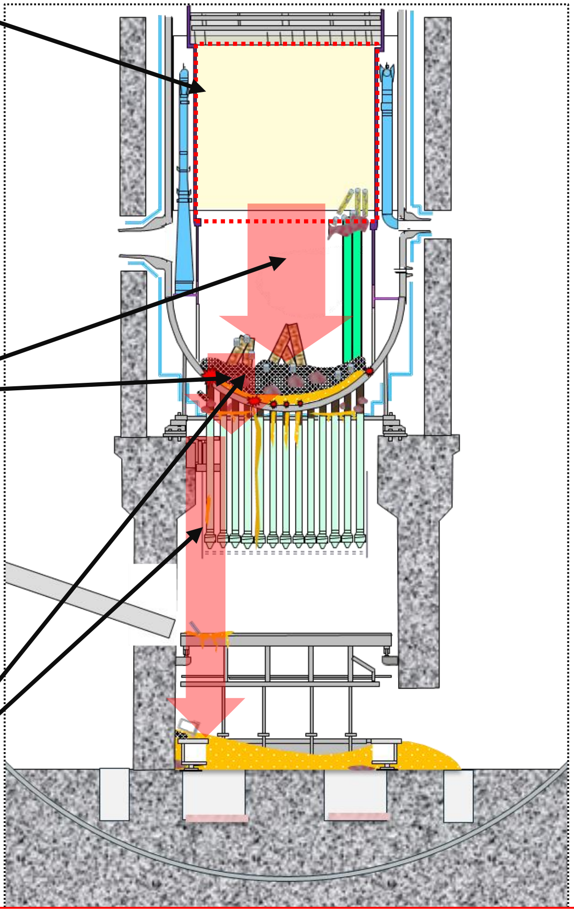
② Structures at the bottom of the core and the lower plenum got caught up in the molten material as it migrated and the structural material elements oxidized

- (Entrainment of structures)Lot of structural material elements (Fe+Cr+Ni). **【p.8,32】** Al detected **【p.36】**
  → Suggests entrainment of the CRD housing (stainless steel), welding material, stub tube (Ni alloy), and insulating layer (Al alloy), etc.
- (Oxidization of structural material elements)A Fe-Ni metal phase exists, and Fe oxides and Fe-Cr oxides exist in the vicinity of the Fe-Ni metal phase **【p.11】**
  → It is possible that structural material elements, such as stainless steel etc., oxidized from the water vapor generated and that Cr and Fe, which oxidize relatively easily, oxidized in succession.

③ Solid-liquid mixed state（More than approx. 1,350℃）

- (Solid-liquid mixed state)Regions with high concentrations of Fe exist around cubic crystals （Zr-U-O phase） **【p.10】**
  → This suggests the possibility that U-Zr oxide particles dispersed in the liquid phase, which is high in concentration of Fe and Cr.
- (Micro-mixed phase formation)Cubic crystals (Zr,U)O_2, cubic crystals (U,Zr)O_2, FeNi metal, Fe_3O_4 and $FeCr_2O_4$ are finely mixed together **【p.11】**
  → It is assumed that precipitation began at a temperature lower than the (A)Zr-U-O phase, and that the solidification of Fe oxides ended at approximately 1,350℃.

Diagram of the assumed migration path of debris from which the first sample was taken



④ Sample surface layer formation

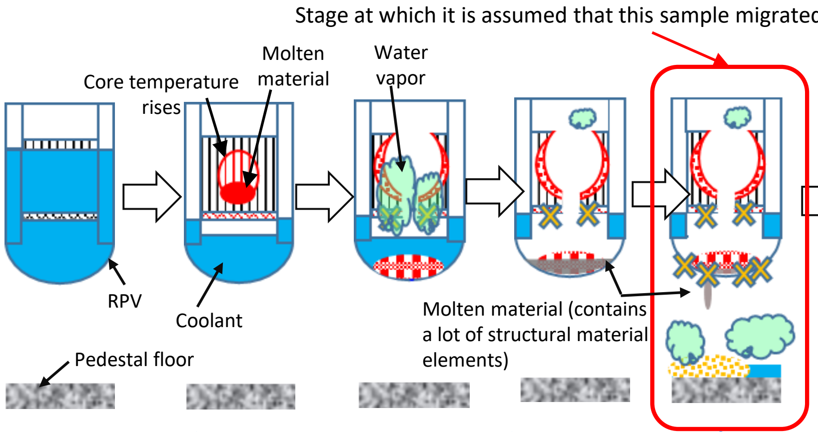
- A similar layered structure was observed on the surface of Fe-Ni metal phase. A similar structure was not seen inside the sample. **【p.13】**
  → It is assumed that the surface layered structure was created after the majority of the sample was formed through melting/solidification.

Utilization of analysis results to ascertain conditions inside the reactor

By continuing to estimate how the accident unfolded in conjunction with other deliberations to date, we are striving to ascertain conditions inside the reactor, such as the distribution of fuel debris, etc., and utilize that information to deliberate fuel debris retrieval and internal investigations.

【The positioning of sample analysis results】

It is assumed that this fuel debris(first sample) was created through the oxidization of structural material elements, etc. by the steam vapor generated in the reactor pressure vessel as the fuel migrated into the pedestal.

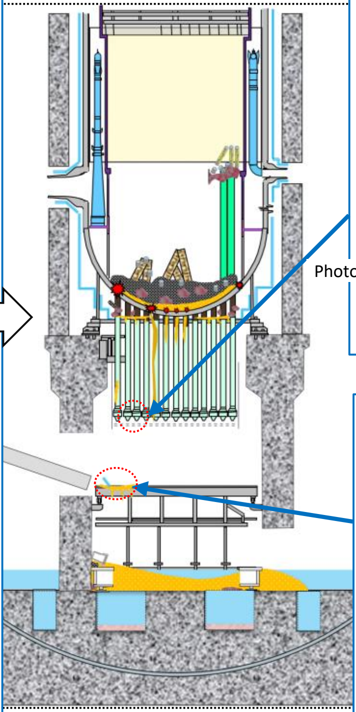


Core temperature rises with a decrease in water level

High temperature fuel migrates into the lower plenum → Water vapor generated when it comes in contact with coolant

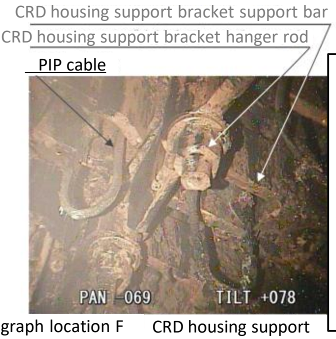
Molten material continues to migrate into the pedestal and water vapor is continuously generated

Estimated accident progression at Unit 2 [1]



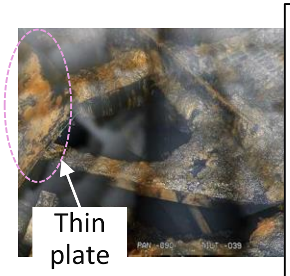
Estimated conditions inside of the Unit 2 reactor [2]

【Knowledge gained from internal investigations】



No significant damage was found to the CRD housing supports
⇒ It is assumed that when the fuel debris migrated to the pedestal, temperatures had not largely exceeded the melting point of stainless steel（Approx. 1,450℃）

External appearance of CRD housing support [3]
（Investigation period: January 2018）



A thin plate was found on top of the platform. A comparison with design schematics suggests the possibility that the insulation layer directly underneath the RPV (Aluminum alloy; melting point: Approx. 660℃) fell off[5].
⇒ It is assumed that this insulating layer got caught up in the fuel debris as it migrated into the pedestal.

External appearance of platform near the CRD rails [4]
（Investigation period: February 2017）

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[1]Diagram source: Edited excerpt from Yamashita et al. Annals of Nuclear Energy, 173 (2022) 109129.

[2]JAEA, FY2024 final report (October 2025) on “Decommissioning and contaminated water project expense subsidies (analysis for ascertaining fuel debris attributes and developing treatment technology (Development of technology for hypothesizing RPV damage conditions and the behavior of fuel debris as it migrated inside the PCV))” which began in FY2023

[3]TEPCO HD, IRID, Unit 2 Primary Containment Vessel Internal Investigation Implementation Results. 50th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting, Document 3-3

[4] TEPCO HD, IRID, Unit 2 Primary Containment Vessel Internal Investigation Implementation Results. 39th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting, Document 3-3

[5] JAEA, FY2022 final report (September 2023) on “Decommissioning and contaminated water project expense subsidies (analysis for ascertaining fuel debris attributes and developing treatment technology (Development of technology for hypothesizing RPV damage conditions and the behavior of fuel debris as it migrated inside the PCV))” which began in FY2022

Conclusions and future plans

1. Grasping the condition of the sampled area (grasping the condition of the fuel debris samples)【Already reported [1] [2]】

Required information	Assessment results
Basic information for deliberating retrieval (dose rates, density distribution)	The fuel debris samples are heterogeneous overall with pores distributed throughout.
Basic information for deliberating storage methods and safety measures during retrieval, such as criticality assessments (element content, isotope ratio)	Fuel elements (U) account for most of the sample mass. However, a comparison with core composition found that the mass of structural material elements (Zr, Fe, Cr, Ni) was larger than fuel elements (U), so it is assumed that the fuel debris was created as various material got entrained in the molten fuel as it migrated into the PCV.
	The concentration of U is approx. 1.9wt% (^{235}U/U ratio), the samples are not skewed, and the value is close to the core average.
Information for deliberating the development of non-destructive measurement technologies during fuel debris retrieval (radioactivity concentration)	In terms of discharged γ-ray nuclides, the concentration of Cs is low, and the concentrations of Eu and Co are high. There is a good possibility that radioactive Cs volatilized in the high temperatures during fuel debris generation. And the association between ^{154}Eu and ^{244}Cm, and U was confirmed.
Basic information for deliberating retrieval methods/equipment (element, compound distribution)	Primarily comprised of Zr-U-O phases, Fe-Ni metal phases, micro-mixed phases and pores. Assumed to be relatively easy to crush.

[1] 133rd Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting, Document 3-3

[2] 140th Decommissioning, Contaminated Water, and Treated Water Countermeasures Team Meeting , Document 3-3

2. Estimation of the fuel debris formation process

Required information	Assessment results
Information on how fuel debris was created in order to elaborate on the diagram estimating conditions inside the reactor (crystalline structure, composition of phases that contain U)	It is assumed that after molten material was formed inside the RPV (more than approx. 1,900℃) and lower structures got caught up in the molten material, that material migrated into the pedestal and solidified after which a micro-mixed phase was formed at around approx. 1,350℃, which is the temperature at which FeO precipitates

【Future plans】

- Analysis originally planned for the first sample have been completed. Assessments will be revised as necessary based on the results of additional analyses being planned (XRD, radioactivity analysis, etc.).
- Detailed analysis is under way for the second sample, and the results will be compared with those from the first sample.

Utilization of analysis results for decommissioning (1/2)

◆ Valuable data has been obtained from the analysis of a small fuel debris sample, and this knowledge will be leveraged for future fuel debris retrieval.

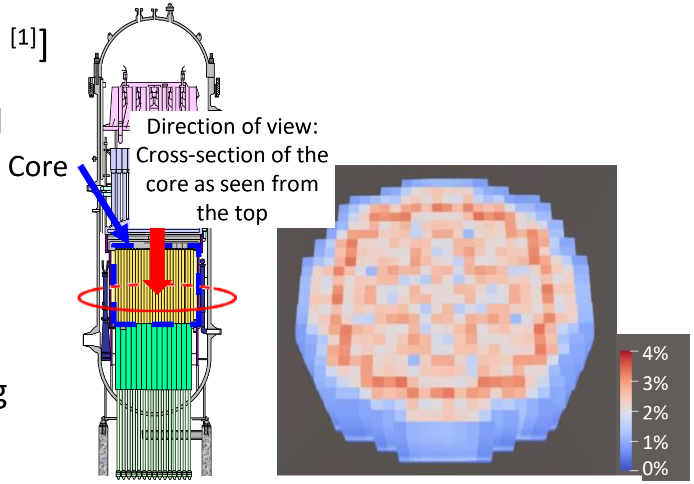
◆ This assessment is based on the analysis results of a small sample, and revisions will be continually made based on knowledge obtained through future analysis.

1． Grasping the condition of the sampled area [Already reported [1]]

■ Deliberating safety measures and storage methods during retrieval （Scope of uranium enrichment）

- The level of uranium enrichment was close to core means. Since the level of uranium enrichment in the fuel debris is close to the enrichment distribution in the Unit 2 core prior to the accident (approximately less than 1%~4%), it is possible that the level of enrichment equalized as the fuel mixed during melting and solidification.

➢ The degree of enrichment is a parameter used to assess criticality when deliberating safety measures and storage methods during retrieval, so if the anticipated degree of enrichment can be reduced based on actual analysis results, then it will be possible to design the retrieval method more logically.



Example of enrichment level distribution in the BWR core
(red shows the degree of high enrichment)

（Radiation sources that should be considered during dose assessments）

- It appears that Cs volatized in conjunction with the high heat/melting of fuel during the accident, and that the concentration of ^{137}Cs was approximately 1/1,000 that of the core mean (spent fuel).

➢ However, since there is little ^{137}Cs, the impact of ^{154}Eu and ^{60}Co was relatively large, so this knowledge will be leverages for exposure countermeasures.

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[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment

Utilization of analysis results for decommissioning (2/2)

1． Grasping the condition of the sampled area (follow-up) [Already reported [1]]

- Information used to deliberate the development of non-destructive measurement technologies used during fuel debris retrieval
  - ^{154}Eu and ^{244}Cm, which are difficult to volatize, are associated with U. By analyzing the amount of ^{154}Eu, ^{244}Cm and U present in actual fuel debris, we have quantitatively confirmed that the association is good.
  - ➢ Data pertaining to association can be leveraged to deliberate non-destructive analysis methods such as those that use ^{154}Eu and ^{244}Cm, which emit gamma rays and neutron rays, in lieu of, and to indicate, U, which is relatively difficult to measure, such methods already being developed.
- Basic information for deliberating retrieval methods/tools
  - Observation of cut faces revealed many pores and the fractionated samples could be crushed by hand at analysis facilities.
  - ➢ It is assumed that fuel debris with the same structure can be crushed so this information will be used to deliberate processing jigs.

2． Estimation of formation process of fuel debris [Update]

- Refining hypotheses about conditions inside the reactors will help to deliberate retrieval methods and internal investigations

【Updates】

  - It is assumed that in the core, fuel and cladding tubes melted and mixed over a fairly wide area, after which other materials along the path of migration to the floor of the pedestal were entrained.
  - After assessing the mechanism by which fuel debris formed based on existing knowledge and the analysis results of the crystalline structures, etc., it is possible to explain melting and solidification at temperatures between approx. 1900~2400℃, meaning that the temperature did not necessarily have to reach the melting point of $(U, Zr)O_2$ (approx. 2500℃)
  - It is assumed that the fuel debris formed after a suspension comprising the liquid phase high in Fe and Cr concentration and the U-Zr-O solid phase was created. Since the suspension is fluid it is possible that similar fuel debris samples exist near the sampling location.
  - ➢ By mixing this deliberation with information obtained to date, we will continue to estimate how the accident unfolded, and strive to ascertain conditions inside the reactor, such as fuel debris distribution, etc., with the intention of utilization this information to deliberate fuel debris retrieval methods and internal investigations.

---

[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment

[Reference] Fuel element composition

Reference) U、Pu、Nd、Gd isotope composition（mass spectrometry）

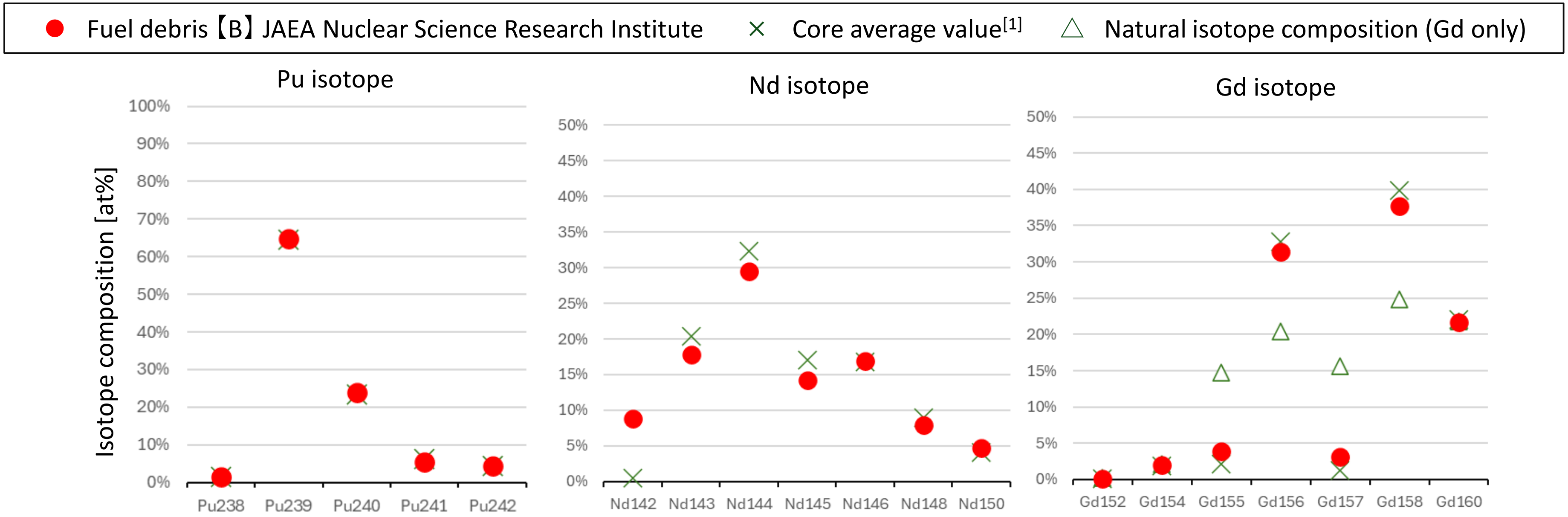
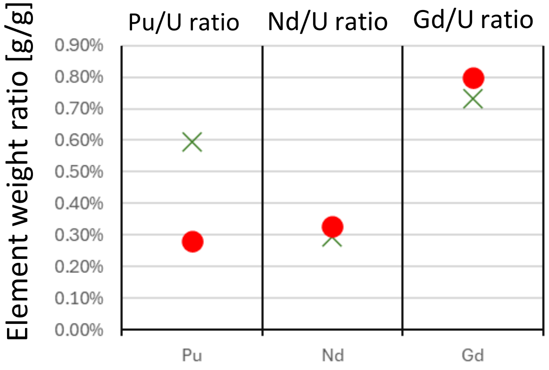


Figure 1　TIMS isotope composition analysis results (U has been omitted)

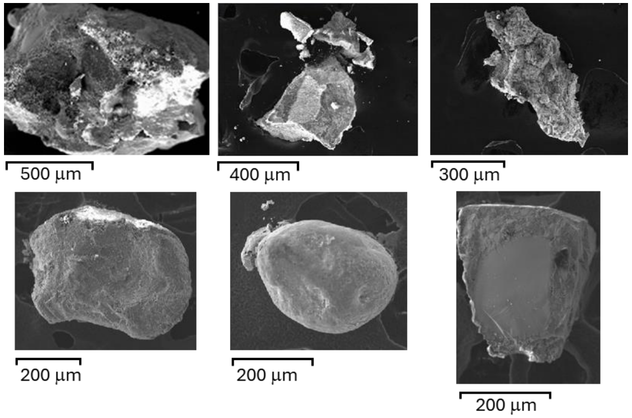


IDMS element mass analysis results
(compared to the ratio of U)

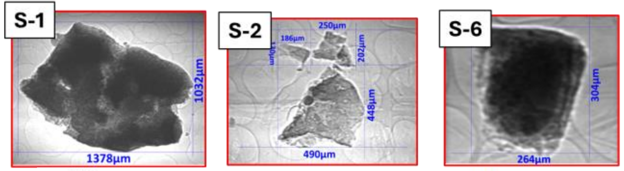
- The isotope composition is consistent with the average values for U, Pu, Nd, and Gd.
- Measurement of the element mass ratio to U shows that Gd/U and Nd/U are approximately the same as the average values, but the Pd/U ratio is approximately half of the average value.
- The chemical behavior of Pu depends greatly on the oxidation-reduction environment, so this provides a clue about the environment in which the fuel debris sample was formed.
- IDMS will be used to re-measure the Pu concentration in a standard solution in order to confirm the accuracy of the Pu analysis values.

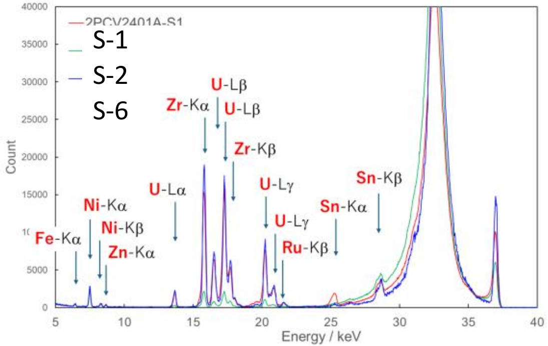
[1] Okumura, et al., Atomic Energy Society of Japan 2021 Spring Conference, 3B01.

[Reference] Synchrotron radiation analysis results(μ-XRF, μ-XRD）

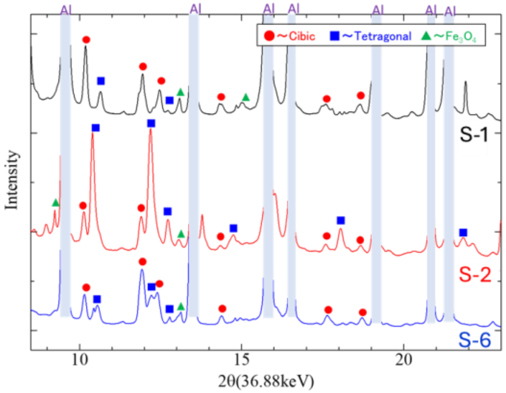


U-rich（S-1, S-2, S-6）



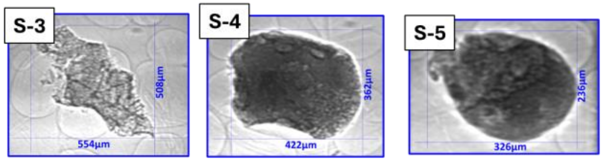


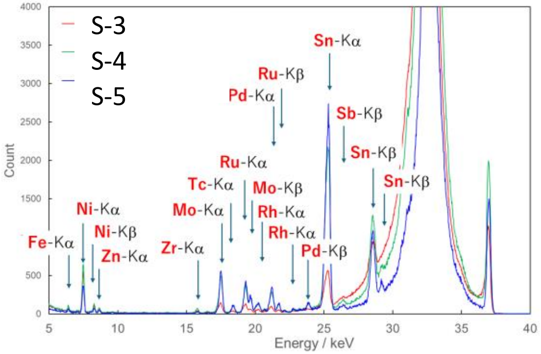
Primarily U, Zr, Fe, Ni. Small amounts of Ru and Sn.



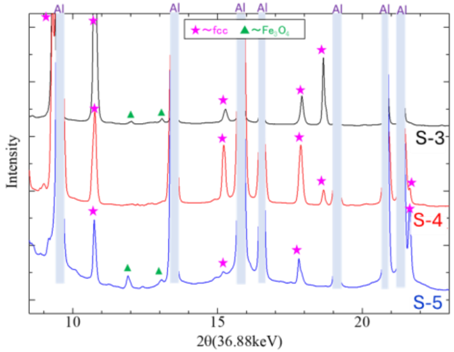
- Cubic & tetragonal primarily exist.
- There is a peak that suggests Fe_3O_4

U-poor（S-3, S-4, S-5）





Primarily Fe, Ni, Mo, Tc, Ru, Rh, Pd, and Sn. Small amount of Zr. Almost no U.

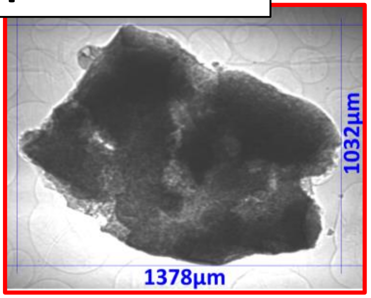


- Fcc（metal phase）primarily exists.
- There is a peak that suggests Fe_3O_4

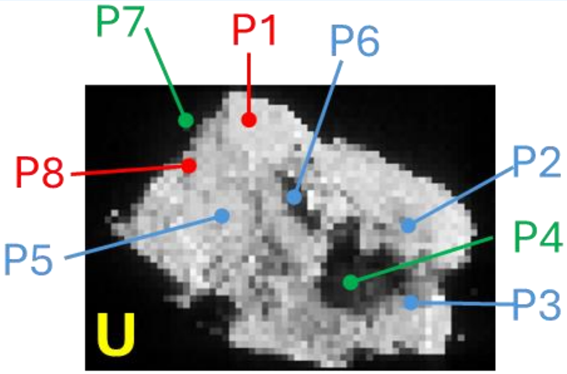
Tetragonal crystals also observed with cubic crystals in the U-rich phase that contains U

[Reference] Synchrotron radiation analysis results (μ-XRF：Pu distribution)

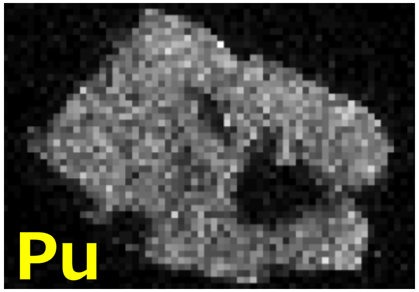
Specimen S-1



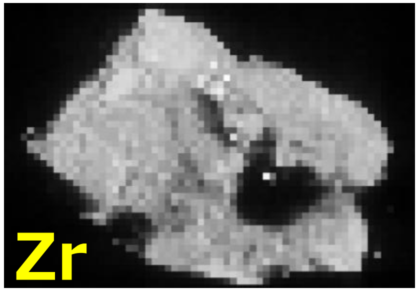
X-ray transmission image



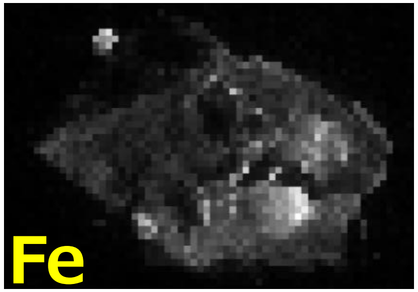
U-Lα Map



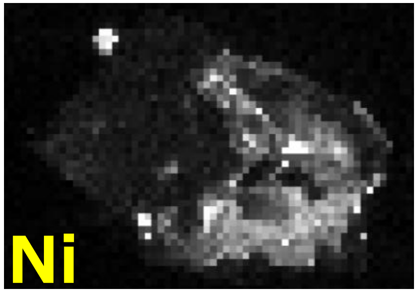
Pu-Lα Map



Zr-Kα Map

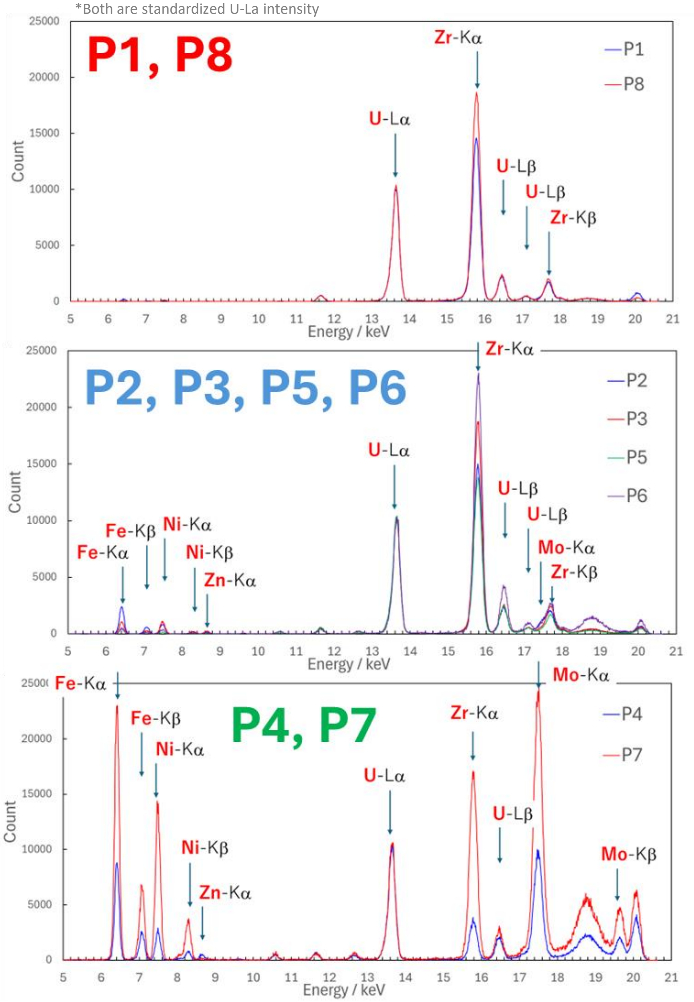


Fe-Kα Map



Ni-Kα Map

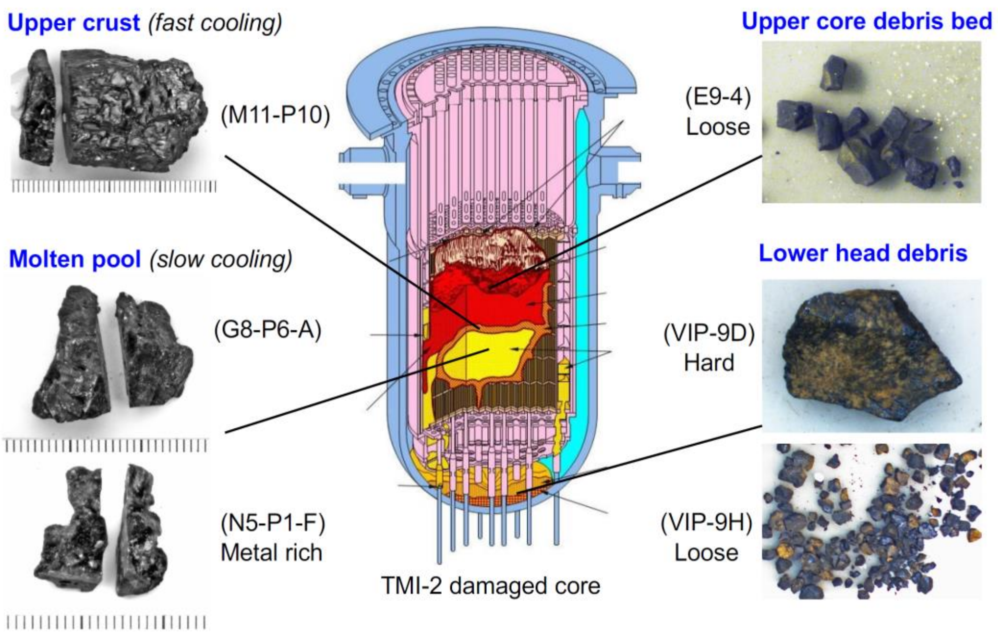
*Both are standardized U-La intensity

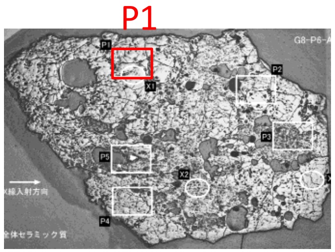
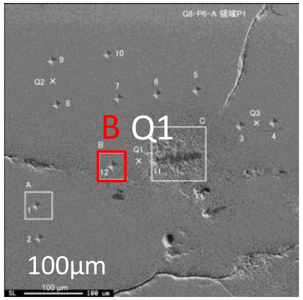
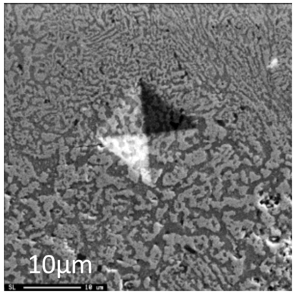


U, Zr and Pu distribution are roughly the same.
There are regions with an uneven distribution of Fe and Ni.

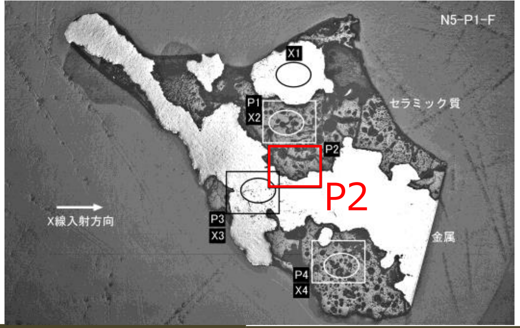
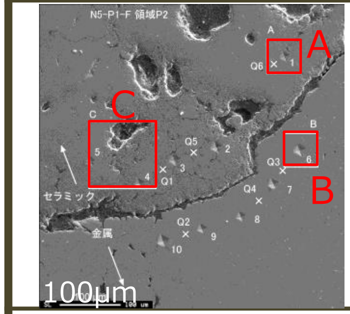
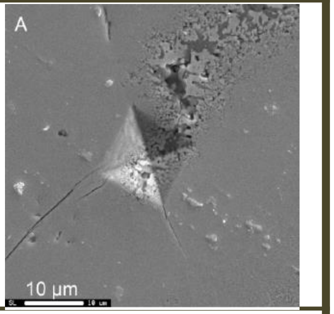
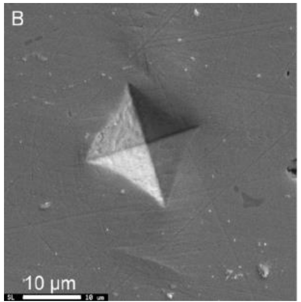
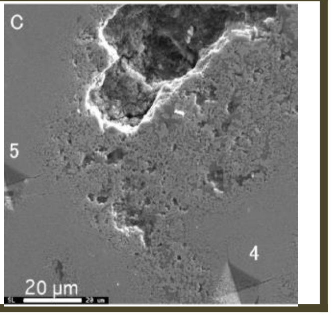
The main elements of P1 and P8 are U and Zr.
Fe, Ni, Mo also found in P2, P3, P5, P6.
P4, P7 contain a lot of Fe, Ni and Mo.

[Reference]TMI-2 fuel debris properties [1]



Molten pool		
G8-P6-A	P1	B
		

Tetragonal crystal $(Zr,U)O_2$ mixes with the cubic crystal $(U,Zr)O_2$ parent phase resulting in a miniscule amount of Fe-Cr-Ni precipitate.

Molten pool	
N5-P1-F	
	
P2	A
	
B（Metal phase）	C
	

These comprised of an oxide phase and a metal phase. The metal phase parent is Ag-In-Sn and the oxide phase parent is a cubic crystal $(U,Zr)O_2$

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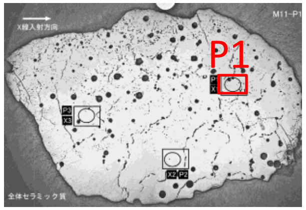
[1] Obtained from FY2013 supplementary budget "Decommissioning and contaminated water project expense subsidies (ascertaining fuel debris attributes and developing treatment technology)" and the FY2014 Supplementary budget "Decommissioning and contaminated water project expense subsidies (ascertaining fuel debris attributes)" (Implemented during FY2014-2015)

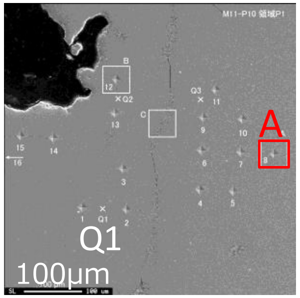
[Reference]TMI-2 fuel debris properties [1]

Upper crust

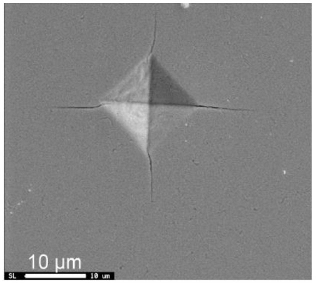
M11-P10



P1



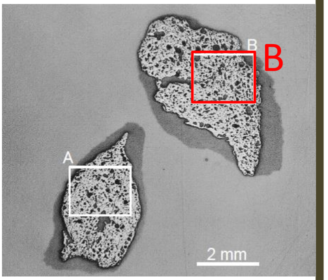
A



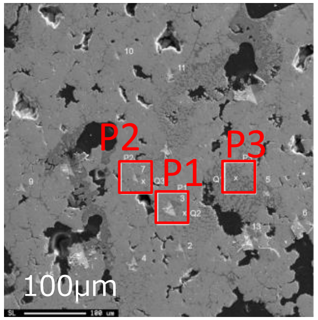
Cubic crystal (U,Zr)O_2
(Single phase)

Lower head loose debris

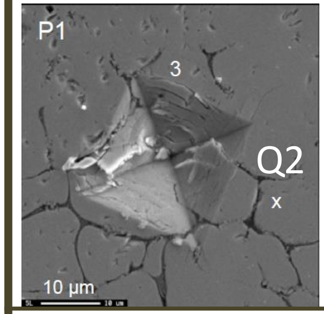
VIP-9H



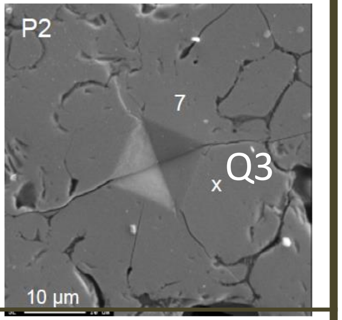
B



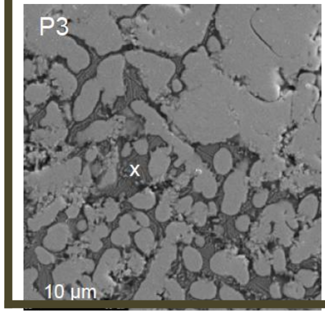
P1



P2



P3



Parent phase cubic crystal (U,Zr)O_2
formation phase Fe-Cr-Ni-Al-O

Vickers hardness of each phase

Specimen	Measurement location	Hardness [GPa]	Notes
VIP-9H	P1	3.4	Includes grain boundary precipitate
	P2	12.8	Cubic crystal (parent phase)
M11-P10	P1 A	13.3	Cubic crystal (parent phase)
G8-P6-A	P1 B	11.3	Cubic crystal (parent phase) ＋ Tetragonal crystal (mixed)
N5-P1-F	P2 A	11.2	Cubic crystal （parent phase） ＋Tetragonal crystal (precipitate)
	P2 B	0.8	Ag-In-Sn (parent phase)

（Reference) Vickers Hardness of Metal and Ceramic Materials [2]
Stainless steel: Approx. 2 GPa （SUS304）
Alumina: Approx. 15 GPa （Al_2O_3）

- In all the samples the cubic crystal (U,Zr)O_2 exists as the parent phase.
- There is little Fe, Cr and Ni contained in the upper crust and molten pool samples.
- There are many precipitates on the grain boundary of the U-Zr-O phase in the lower head loose debris sample. The precipitate is an oxide phase containing much Fe and Cr.
- The hardness values of regions containing precipitate phases are low.

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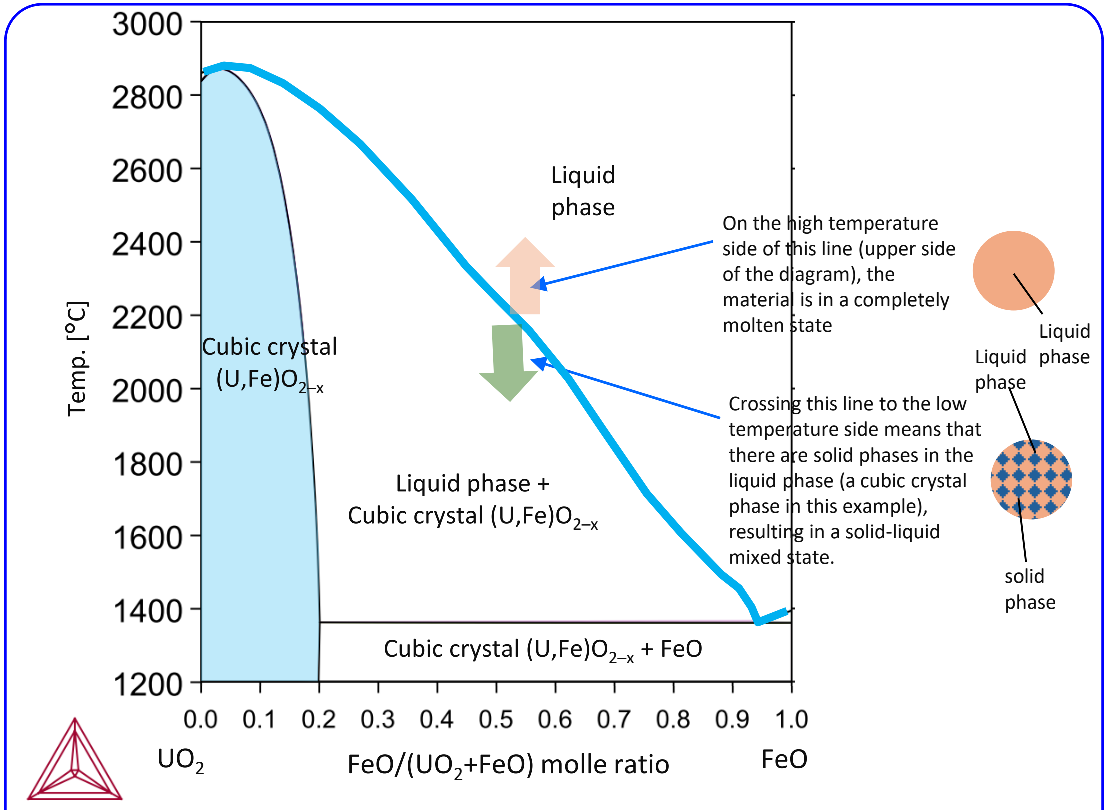
[1] Obtained from FY2013 supplementary budget “Decommissioning and contaminated water project expense subsidies (ascertaining fuel debris attributes and developing treatment technology)” and the FY2014 Supplementary budget “Decommissioning and contaminated water project expense subsidies (ascertaining fuel debris attributes)” (Implemented during FY2014-2015)

[2] IRID, FY2014 Supplementary budget “Decommissioning and contaminated water project expense subsidies (ascertaining fuel debris attributes)” FY2015 Achievement Report, April 2016

[Reference] How to read a phase diagram

What is a phase diagram・・・A graph that shows the stable phases of a material under different temperatures and pressures

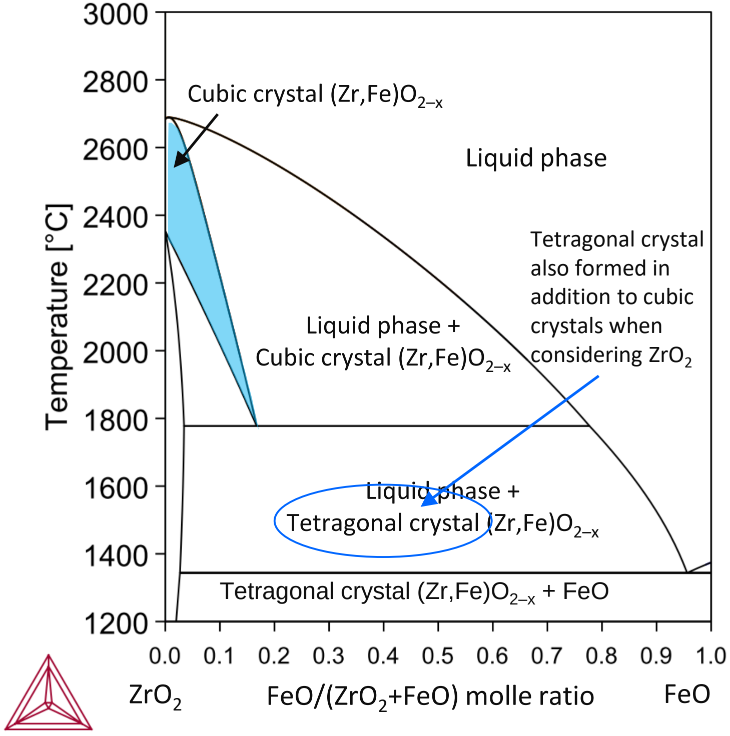
- ➢ There are various databases for creating phase diagrams based on experiments with melting UO_2, Zr, and Fe.
- ➢ There are many simplified※ forms of phase diagrams used to examine only major elements.



On the high temperature side of this line (upper side of the diagram), the material is in a completely molten state

Crossing this line to the low temperature side means that there are solid phases in the liquid phase (a cubic crystal phase in this example), resulting in a solid-liquid mixed state.

UO_2–FeO pseudo-binary phase diagram **【P11】**



Tetragonal crystal also formed in addition to cubic crystals when considering ZrO_2

Reference) ZrO_2–FeO Pseudo-binary system calculated phase diagram

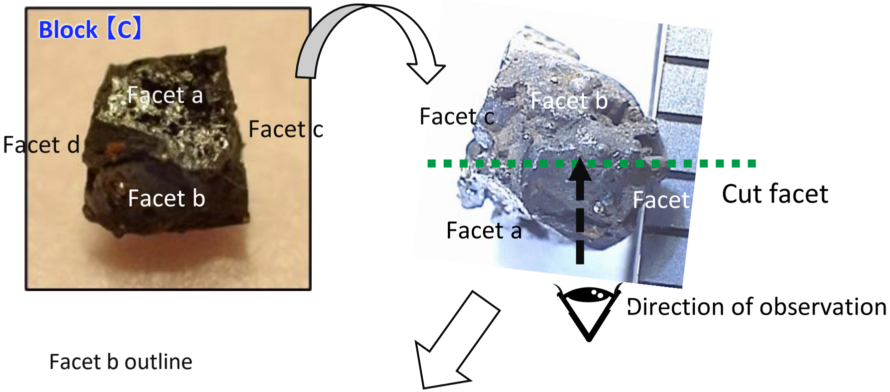
Used database: TAF-ID ver.15.0

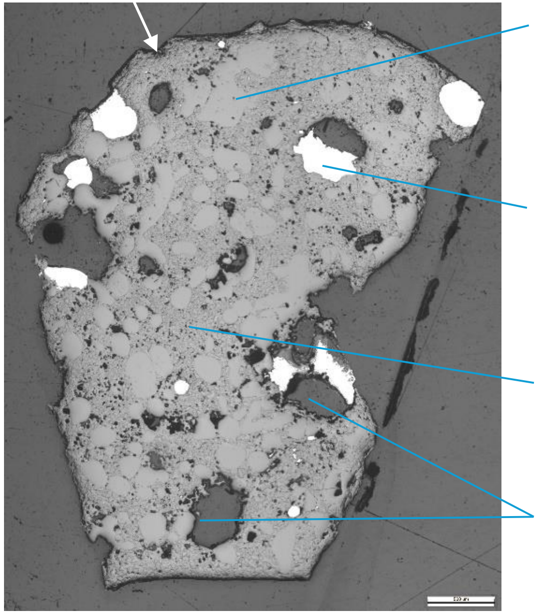
※ Example of simplification: U-Zr-Fe-Cr-Ni-O simplified as ZrO_2-FeO

- ✓ It is assumed that U is replaced by Zr and exists as ZrO_2 # Because Zr>U in each batch, and primarily exists as dioxides.
- ✓ It is assumed that Cr and Ni are replaced by Fe, and exist as FeO. # Because Fe>Cr in each batch, there is Ni, and Fe exists both as a metal phase (Fe-Ni) and an oxide phase (Fe_3O_4、$FeCr_2O_4$) (In the phase diagram above the formation of FeO is deemed to be the formation of metal Fe and Fe_3O_4(including $FeCr_2O_4$))
- ✓ It is assumed that the percentage of FeO is approx. 0.4–0.7 # In light of the possibility that various structural material elements became entrained during the migration of the molten material, a specific value has not been assumed and a range has been given that shows the percentage of Fe+Cr+Ni out of the five primary elements in the sampl (Approx. 40–70 mol%)

[Reference] Solid analysis results from the NFD —Cross-sectional observations/composition assessment [1]—

- Block【C】was cut and an image of the cross-section was obtained (Figure 1) to match with the x-ray CT image after which dose rates and micro-phase structure were assessed.
- Estimates of the porosity and the composition ratios of each region were assessed from analyzing the image of the cross-section. (figure 2)





Optical microscope image

(A)Zr-U-O phase（several tens ~ several hundreds μm）

- The ratio of Zr/U was approx. 2
（Almost consistent regardless of particle)
- Also includes small amounts of Fe, Cr, and Ni

(B)Fe-Ni metal phase（several ~ several hundreds μm）

- Fe/Ni ration was approx. 1～3
（Differs depending on particle)

(C)Micro-mixed phase

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

(D)Pore（several μm ~ several hundreds μm)

- Approx. 20% of the area of the cross-section

Figure 1 observations of the cut facets and cross-section of the fuel debris

Image analysis example: Assessment of void area ratio

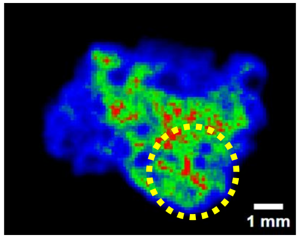
Areas not red are assumed to be voids

(A) Zr-U-O	(B) Fe-Ni	(C) Micro phase	(D) Void
20	4.4	56	19

（A)Zr-U-O phase calculated as 100-(B)-(C)-(D)

Figure 2 Area ratio assessment results for each region [%]

Going forward CT scans of these four regions will be taken in order to examine the ratios of the phases throughout the entire fuel debris sample.



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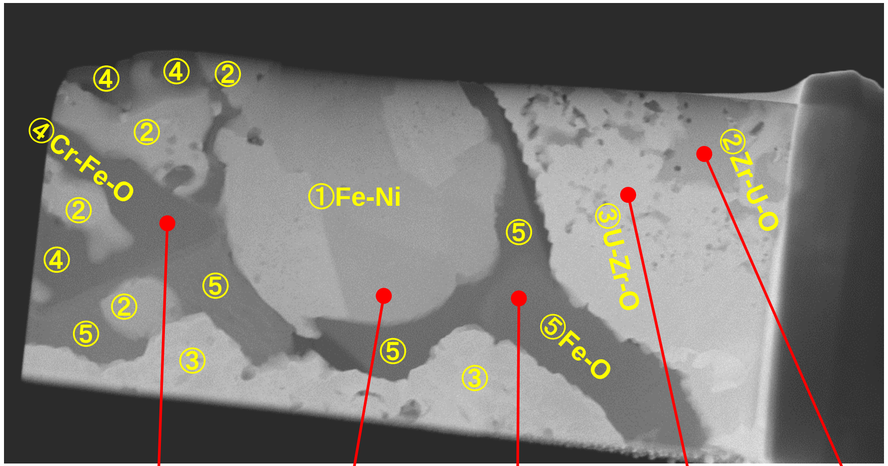
[1] 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment, Document 3-3

[Reference] The attributes and composition of the five phases observed TEM analysis results

(C) Micro-mixed phase



Units: at%

Element	④Cr-Fe-O	①Fe-Ni	⑤Fe-O	③U-Zr-O	②Zr-U-O
O	45	n.d.	39	32	38
Cr	32	n.d.	1	n.d.	L.O.Q.
Fe	22	56	59	n.d.	6
Ni	n.d.	43	n.d.	n.d.	n.d.
Zr	1	n.d.	n.d.	9	32
U	n.d.	n.d.	n.d.	59	23
Misc.	n.d.	Sn	Mn	Si	Mn

Note) With the exception of n.d. and LOQ, the percentage of the elements in the chart are out of 100%

- n.d.: Indicates that no significant amount was detected on the spectrum（”not detected”）
- L.O.Q.: Lower limit of quantification. Although a slight peak was seen in the spectrum, It was below 0.5at% and, therefore, lower than the lower limit of quantification.

STEM-EDX Point analysis results from each analysis location

Upper right region:

②Zr-U-O phase and ③U-Zr-O phase exist with microphases mixed in at the boundaries of each.

Left end:

Small ④Cr-Fe-O, ②Zr-U-O, and ⑤Fe-O phases are mixed together.

Near the center Fe-Ni phase:

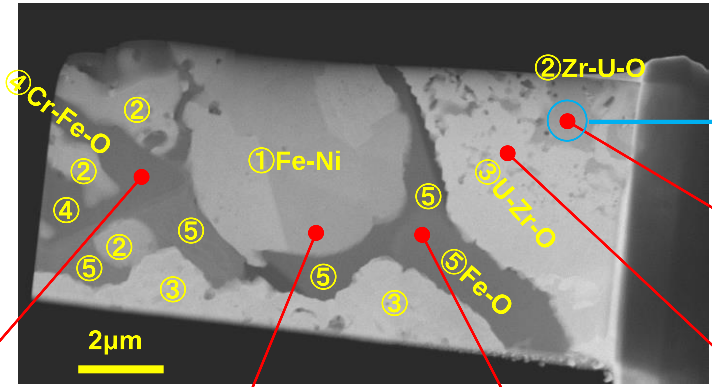
①Fe-Ni is a polycrystal surrounded by ⑤FeO.

[Reference]Estimation of crystalline structure
TEM analysis results and Raman spectroscopy results

- TEM electron diffraction pattern obtained at each point analysis location for the five phases to identify the crystal system. And, in light of the compositional analysis results from each location shown on the previous page the crystalline structure was estimated. （Figure 1）
- Even though there is a high concentration of Zr, location ②c-$(Zr,U)O_2$ showing a cubic crystal phase was subject to Raman spectroscopy to reconfirm the crystal structure, and it was confirmed that it is indeed a cubic crystal phase. (Figure 2)

●…TEM Analysis location

○… Raman spectroscopy location



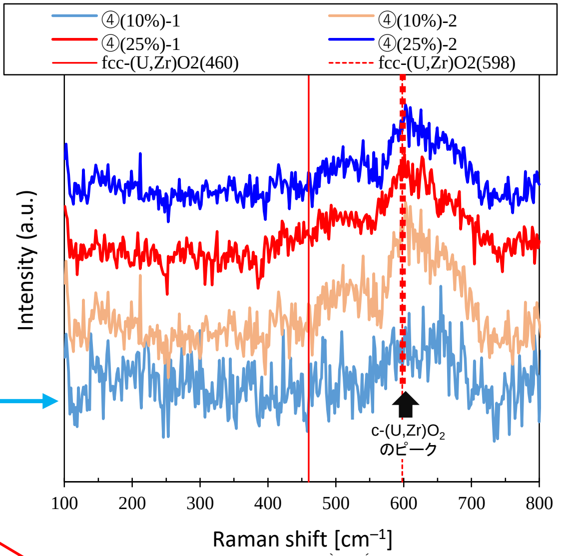


Figure 2 ②Zr-U-O phase Raman spectroscopy results

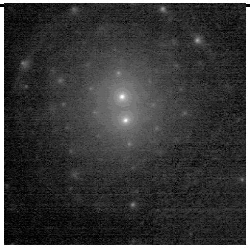
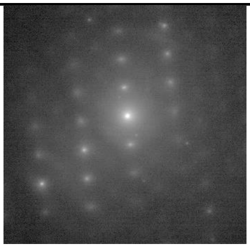
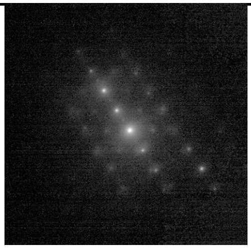
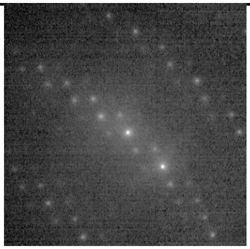
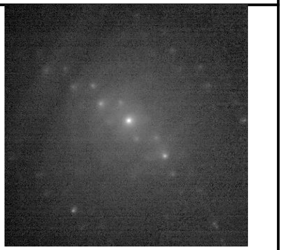
Position	④Cr-Fe-O	①Fe-Ni	⑤Fe-O	③U-Zr-O	②Zr-U-O
TEM Electron diffraction pattern					
The crystal system identification results	Spinel [136]	fcc-FeNi [112]	Spinel [111]	c-UO_2 [101]	c-ZrO_2 [101]
Estimated crystal based on compositional analysis	c-$FeCr_2O_4$	fcc-FeNi	c-Fe_3O_4	c-$(U,Zr)O_2$	c-$(Zr,U)O_2$

Figure 1 Estimated results of crystalline structure at each analysis location

c:cubic（Cubic crystal）

[Reference] Materials inside the PCV

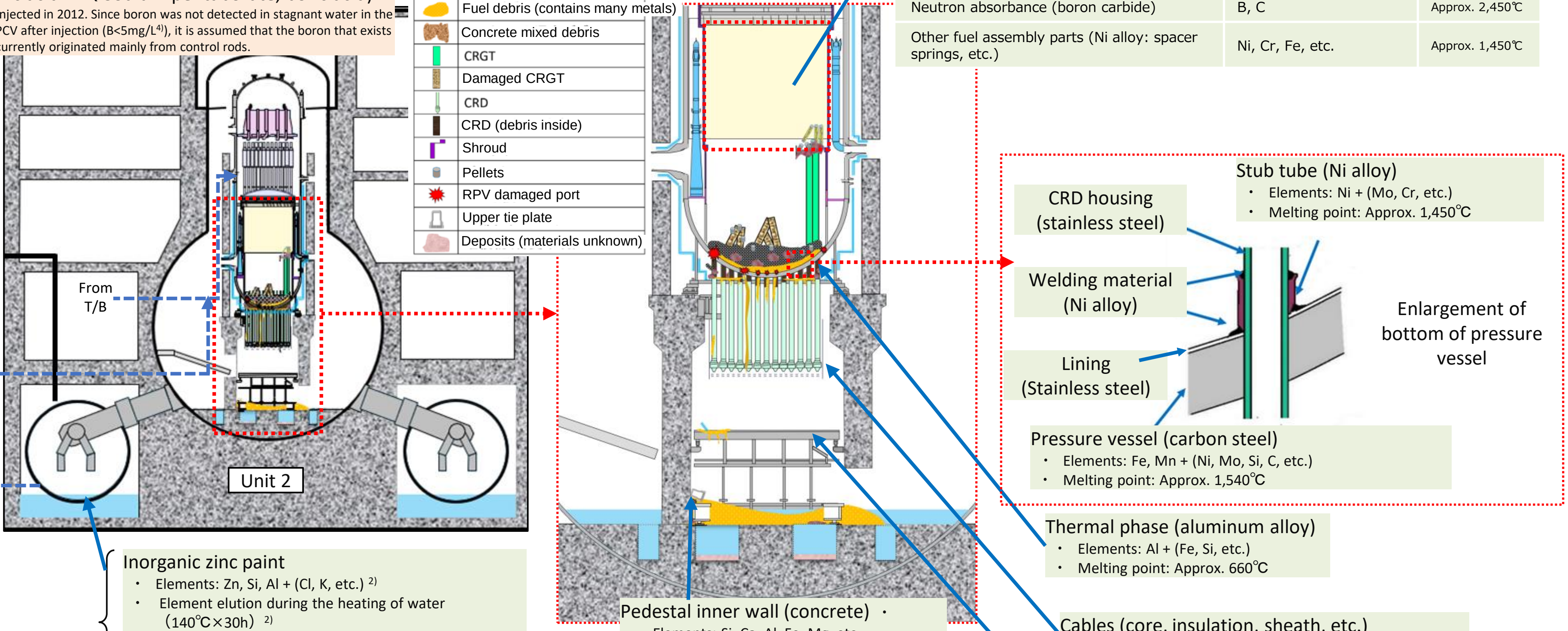
Light green:	Materials that were originally present in the RPV and PCV
Light orange:	Materials introduced following the earthquake

- 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.

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

Seawater
・Elements: Cl, Na, Mg, S, K, Ca, etc. 1)

Boric acid ※ （Sodium pentaborate, boric acid)
※ Injected in 2012. Since boron was not detected in stagnant water in the PCV after injection (B<5mg/L4)), it is assumed that the boron that exists currently originated mainly from control rods.



Stub tube (Ni alloy)
- Elements: Ni + (Mo, Cr, etc.)
- Melting point: Approx. 1,450℃

CRD housing (stainless steel)

Welding material (Ni alloy)

Lining (Stainless steel)

Pressure vessel (carbon steel)
- Elements: Fe, Mn + (Ni, Mo, Si, C, etc.)
- Melting point: Approx. 1,540℃

Thermal phase (aluminum alloy)
- Elements: Al + (Fe, Si, etc.)
- Melting point: Approx. 660℃

Cables (core, insulation, sheath, etc.)
- Elements: Cu + (Si, Sb, Pb, etc.)

Pedestal internal structures (steel + zinc paint)

Pedestal inner wall (concrete) ・
Elements: Si, Ca, Al, Fe, Mg, etc.

Inorganic zinc paint
- Elements: Zn, Si, Al + (Cl, K, etc.) 2)
- Element elution during the heating of water (140℃×30h) 2)

Epoxy sealants
- Elements: Ti, Ba, Si, Al, S, etc. 3)

estimating conditions inside of the unit 2 reactor[1]

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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-.

[Reference] Abbreviation and overview of analysis methods

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 μm order can be obtained by combining Raman spectroscopy with conventional optical microscopes.

[Reference] Abbreviation and overview of analysis methods

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 stimulable luminescence. Dose distribution of the sample can be obtained.

[Reference] Overview and purpose of each item in debris analysis

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

Non-destructive analysis

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

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

[Analysis methods] External appearance, weight, dose rate, IP, X-ray CT, γ-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.

[Analysis methods] SEM-EDX, SEM-WDX, TEM-EDX, SIMS, Raman spectroscopy, μ-XAFS, μ-XRF, μ-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.

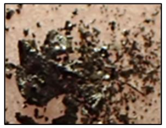
[Analysis methods] ICP-MS, ICP-AES, TIMS, γ-ray spectrometry, α-ray spectrometry

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.

[Reference] Element content [1]

◆ 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.

【D】NDC



【B】NSRI

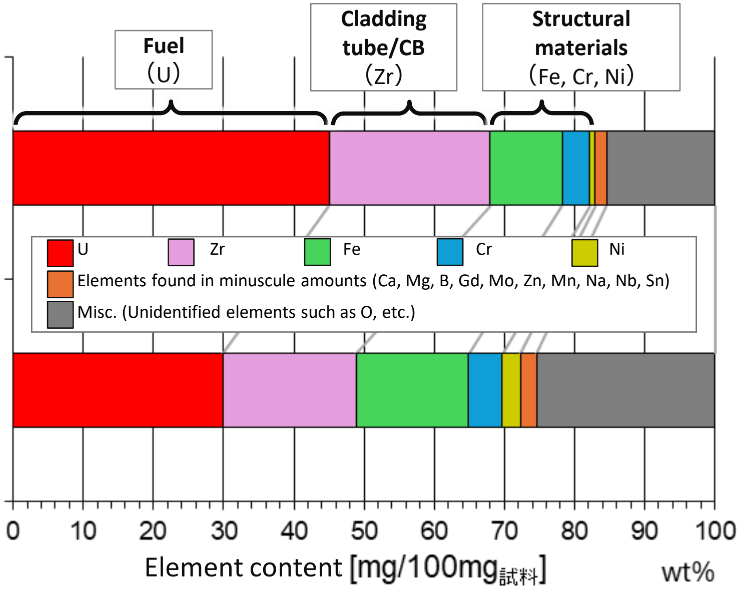


Figure 1 Elemental composition of the sample
(ICP-AES and ICP-MS analysis results)

Converted to the molar ratio of primary elements

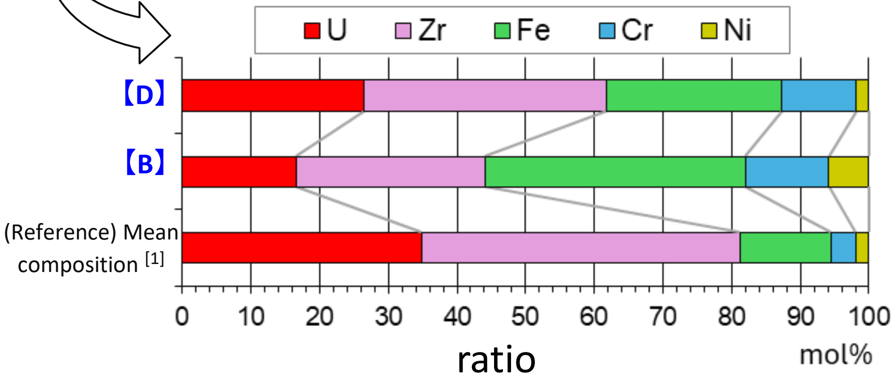


Figure 2 Ratio of major elements (U, Zr, Fe, Cr, Ni)

- 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

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[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment

[2] 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

[Reference] U isotope ratio [1]

◆ In order to ascertain differences, each fractionated sample from block【D】 and particles【A-2】【B】 obtained through crushing were dissolved and TIMS or ICP-MS were used to quantify the uranium isotopes (^{234}U、^{235}U、^{236}U and ^{238}U) in the solution.

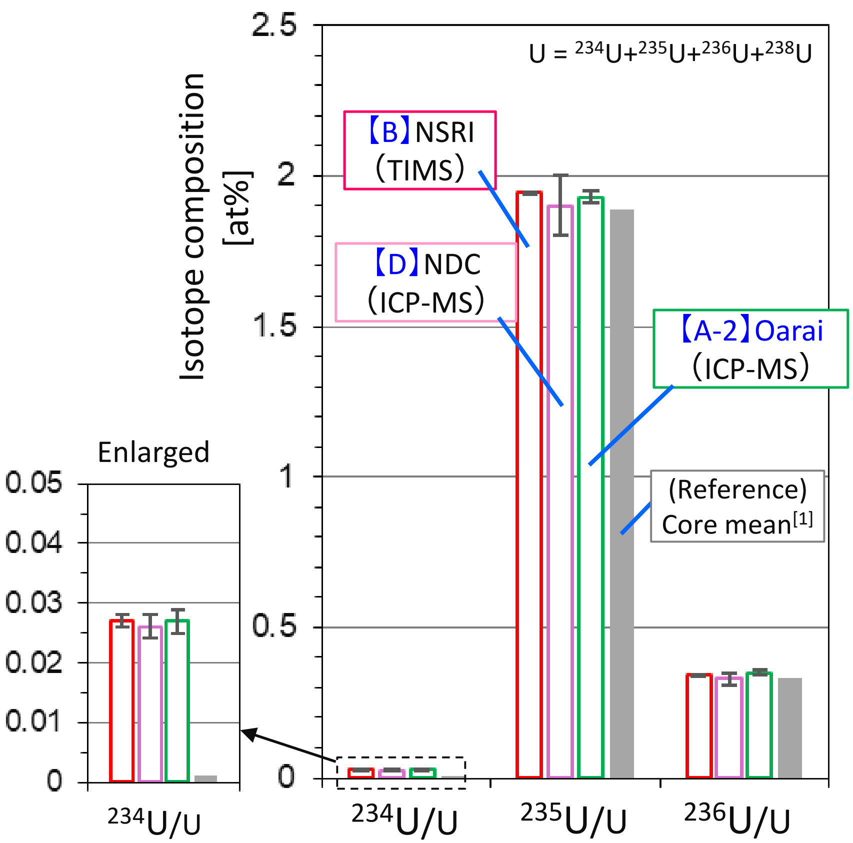

Figure 1 U isotope composition analysis of dissolved solution

○ Level of uranium enrichment （^{235}U/U ratio）

- ^{235}U/U ratio was approx. 1.9at% （Approx. 1.9wt%） for all specimens, and it was confirmed that the difference between fractionated specimens is miniscule. (Figure 1)

➢ ^{235}U/U ratio in the samples was practically the same and close to the core means.

⇒ It is possible that the ^{235}U/U ratio in the fuel debris equalized to that prior to the accident through the melting/mixing process.

U isotope ratio analysis will be continued to estimate the degree of melting/mixing.

※ ^{235}U/U ratio distribution in the core prior to the accident was between less than 1% to approx. 4%

A large range of fuel debris samples will need to be analyzed going forward to determine whether or not the level of enrichment indeed equalized throughout all of the fuel debris.

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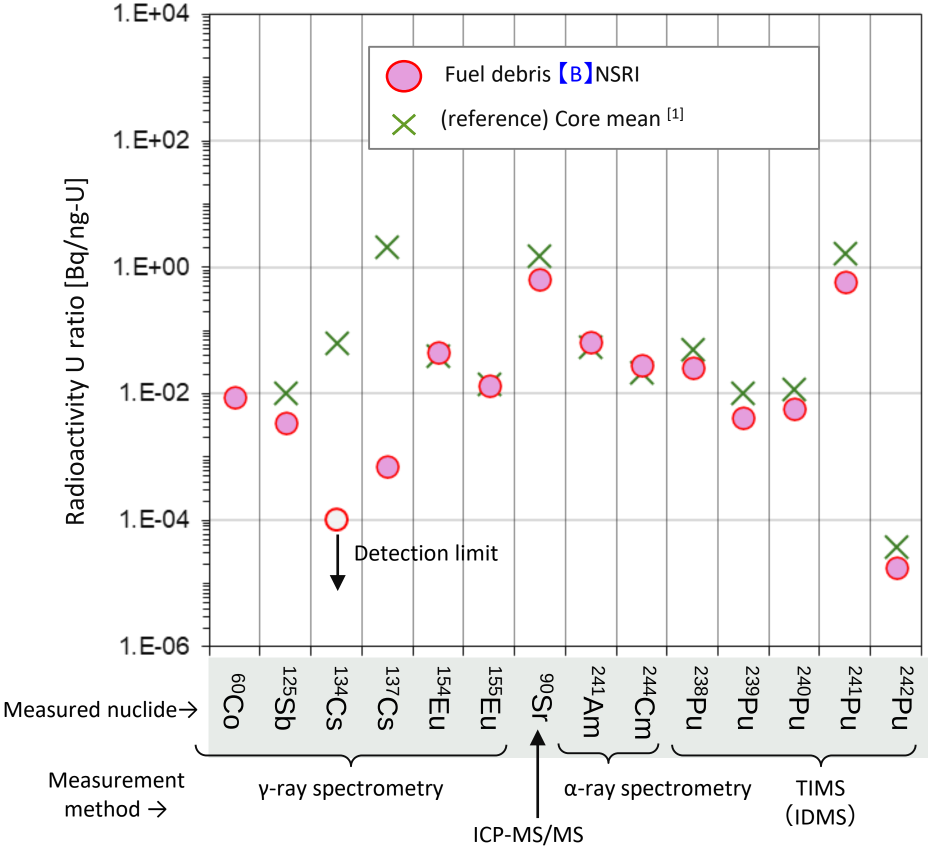
[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment

[2] Okumura, etc. annual meeting of the Atomic Energy Society of Japan spring 2021, 3B01.

[Reference] Radioactivity concentration [1]

◆ 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 γ-ray spectrometry and α-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.



Ratio of each nuclide radioactivity to uranium mass

Note: Radioactivity has been corrected as of May 31, 2025 based on literature [1].
Pu isotope radioactivity was assessed from isotope composition obtained through TIMS (Thermal Ionization Mass Spectrometry) and the amount of Pu measured through IDMS (Isotope Dilution Mass Spectrometry).

○ Major radioactive nuclides

- ^{90}Sr, α nuclides (^{241}Am, ^{244}Cm, and Pu isotopes), ^{154}Eu and ^{60}Co contribute greatly to the radioactivity in fuel debris samples. (Left figure)
- The contribution of ^{134}Cs and ^{137}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 γ dose rates.

○ Accompanied condition of U

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

⇒ ^{154}Eu and ^{244}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.

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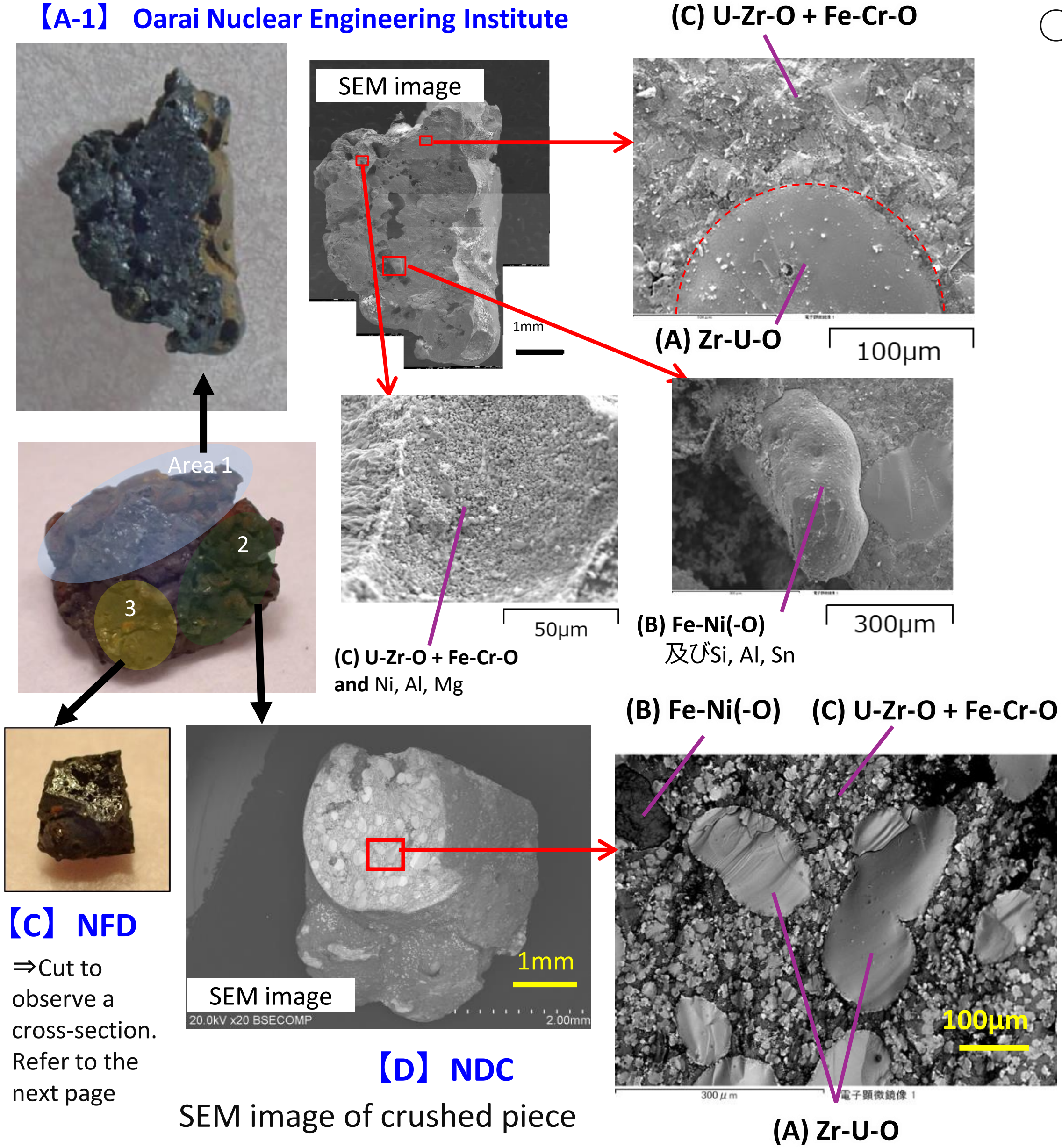
[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment

[2] JAEA-Data/Code 2012-018.

[Reference] Element/compound distribution [1]

【A-1】 Oarai Nuclear Engineering Institute



【C】 NFD

⇒Cut to observe a cross-section. Refer to the next page

【D】 NDC

SEM image of crushed piece

○ **Micro-structure <u>inside</u> the samples: SEM-EDX observation results for <u>the surface of the crushed area.</u>**

- The observed surface is comprised completely of U-Zr-Fe-Cr-Ni-O compounds.
  (A)Zr-U-O coarse particles
  (several tens~Approx. two hundreds μm)
  (B)Fe-Ni(-O) particles (≲several hundreds μm)
  (C)U-Zr-OとFe-Cr-O fine compound region

⇒ It is hypothesized that fuel debris formed when oxidized structural materials, such as zirconium alloys and stainless steel, etc., melted and mixed with the fuel.

- Mg、Al、Si、Sn are present in localized areas

⇒ It is possible that small amounts were caught up in the seawater, thermal layer, and paint, etc.

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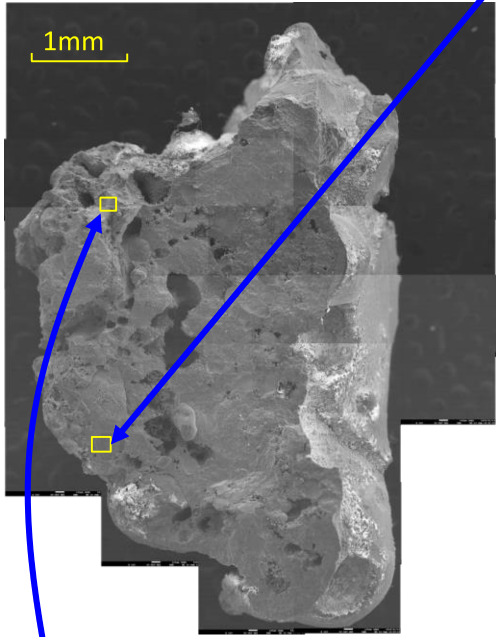
[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment

[Reference] Solid analysis results [1]

― Fractured surface observations/composition assessments of each piece ―

Block【A-1】：JAEA Oarai

Overall SEM image of fragment



Measured with the sample affixed to carbon tape without vapor deposition

Micro-mixed phase (1)

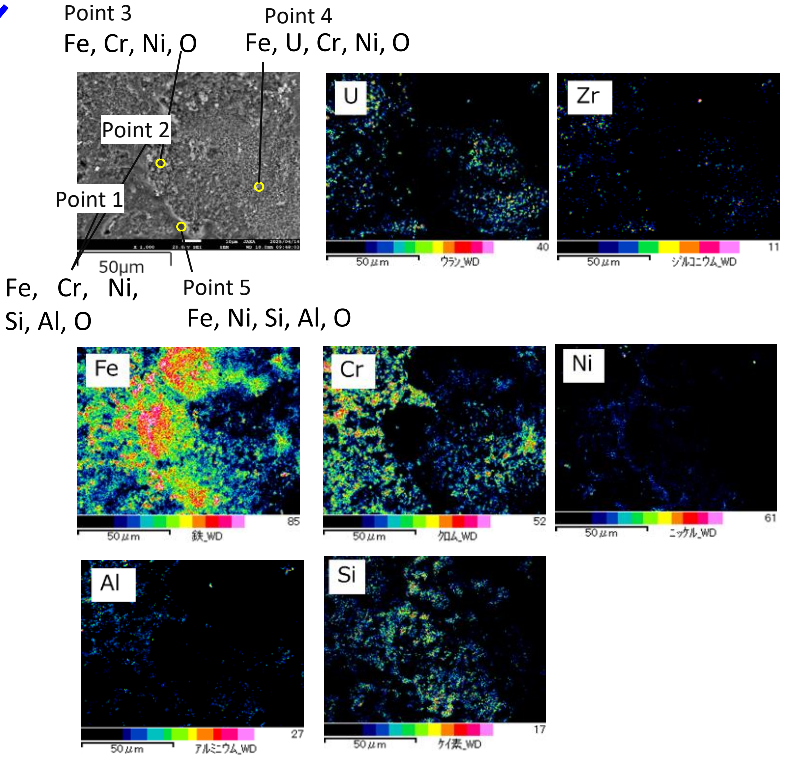


Figure 1 SEM image and element mapping

- Densely packed micro-phases 1~10μm in size exist. They consists primarily of U, Zr, Fe, Cr and Ni. (Figure 1)
- Small amounts of Al and Si were also detected. (Figure 2)

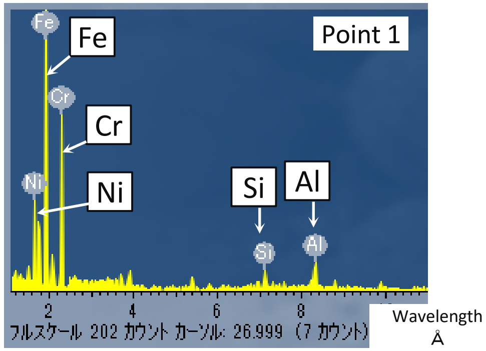
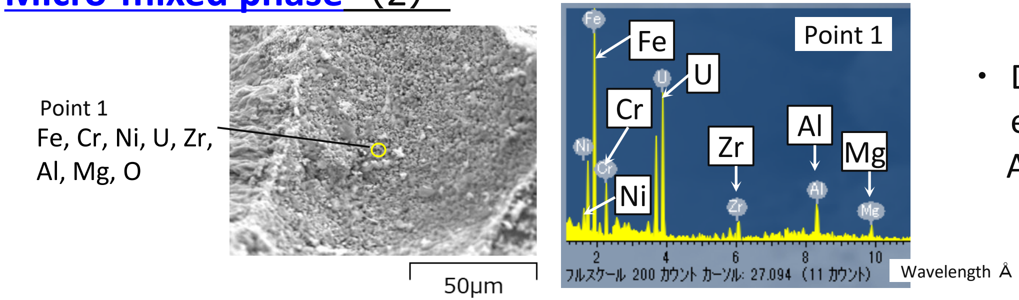


Figure 2 WDX point analysis spectrum of micro-mixed phases

Micro-mixed phase (2)



- Densely packed micro-phases 1~10μm in size exist just like in 1 (Figures 1 and 2).
  A very small amount of Mg exists. (Figure 3)

Figure 3 WDX point analysis spectrum and SEM images of micro-mixed phases

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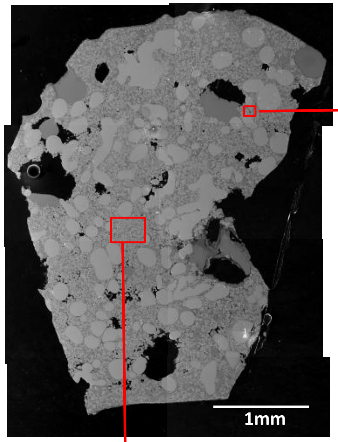
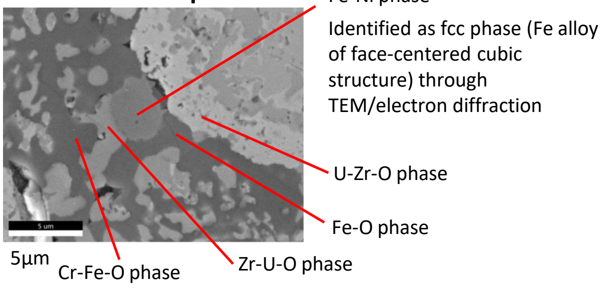
[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment

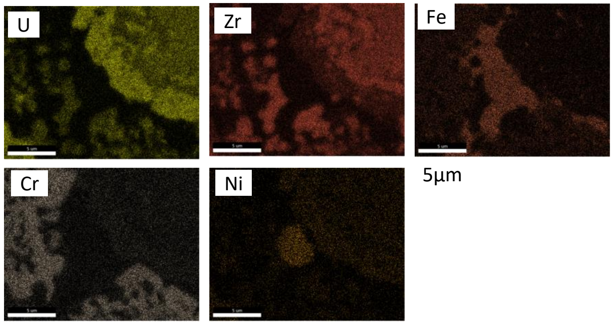
[Reference Solid analysis results[1] – Cross-sectional observation and composition assessment –

Block【C】：NFD (C)Micro-mixed phase

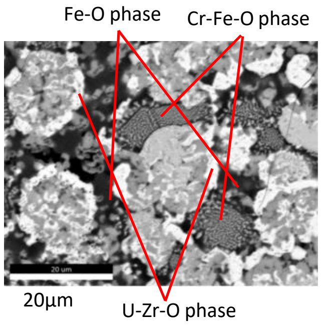


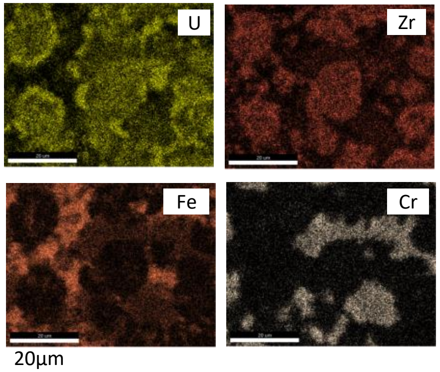
Example including micro Fe-Ni metal phase





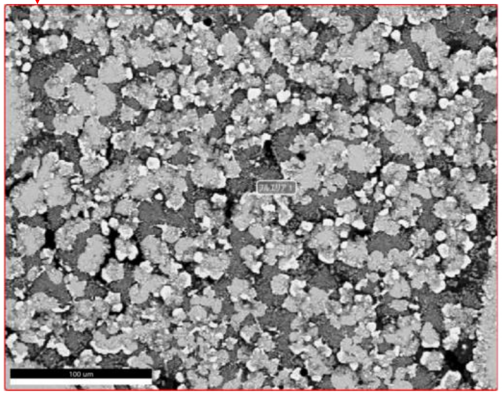
Example of micro oxide phase





- The micro-mixed phase contains a mix U-Zr-O, Cr-Fe-O and Fe-O phases in addition to small (~several µm) Zr-U-O and Fe-Ni phases.

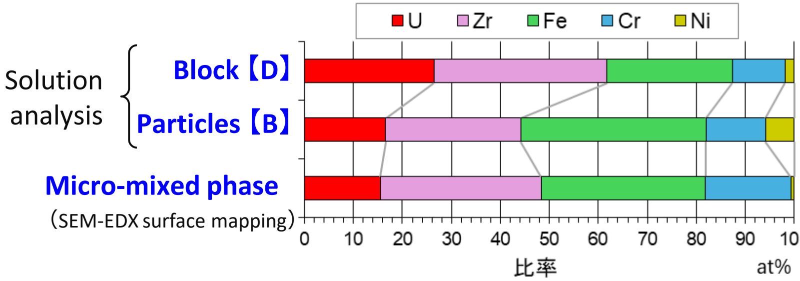
  ⇒ It is assumed that the Zr-U-O phase grew into a molten oxide body with a high concentration of Fe and Cr that separated into each phase as it cooled and solidified.



at%

Major element	Surface analysis
O	48
Cr	10
Fe	18
Ni	1
Zr	15
U	8

Micro-mixed phase SEM-EDX analysis results



Comparison of the ratio of major elements

- The ratio of U:Zr:Fe:Cr:Ni in the micro-mixed phase is close to the composition of particles【B】obtained through pulverization, and may be the reason why it was easy to pulverize.

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[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment

[Reference] Liquid analysis results [1]
— Element content ratio/isotope ratio/radioactivity concentration —

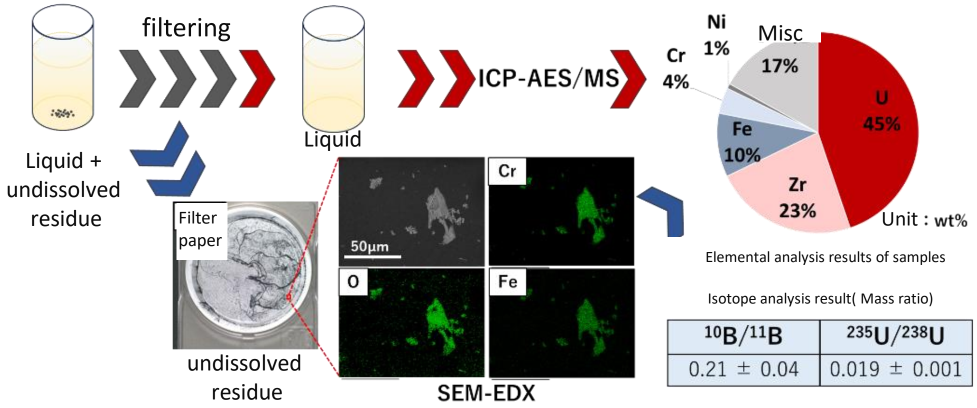
◆ NDC liquid analysis

○ Element content ratio → Chart 1

- Block【D】 was further crushed and approx. 0.1g of the particles obtained was pressure melted in heated acid.
- ICP-AES and ICP-MS were used to measure the quantity of elements in the obtained liquid.
- Approx. 5% of the specimen mass was undissolved residue (primarily Fe-Cr oxide), so element content was assessed using SEM-EDX to estimate the element content of the undissolved residue.
- The ratio of elements contained in the sample were assessed by combining the analysis values for the element content of the liquid with the estimates of the element content of the undissolved residue.

○ Isotope ratio (U) → Chart 2

- The amounts of ^{234}U, ^{235}U, ^{236}U and ^{238}U in the liquid was measured using ICP-MS.



Isotope analysis result(Mass ratio)

^{10}B/^{11}B	^{235}U/^{238}U
0.21 ± 0.04	0.019 ± 0.001

Figure 1 NDC element content ratio assessment flow

Chart 1 Element content ratio assessment results

k=2 Expanded uncertainty

Element	Element content ratio [mg/100mg specimen]
U	45 ± 2
Zr	23 ± 1
Fe	10.2 ± 0.8
Cr	4 ± 1
Ni	0.79 ± 0.03
Si	<0.9
Ca	0.22 ± 0.02
Al	<0.3
Mg	0.098 ± 0.005
B	0.049 ± 0.004
Gd	0.39 ± 0.05
Mo	0.123 ± 0.007
Sb	<0.003
Pb	<0.006
Zn	0.062 ± 0.003
Ti	<0.03
Mn	0.42 ± 0.02
Na	0.087 ± 0.006
Nb	0.090 ± 0.008
Sn	0.033 ± 0.001

Chart 2 Uranium isotope ratio analysis results

k=2 Expanded uncertainty

Nuclide	U isotope ratio※1 [at%]
^{234}U/U	0.026 ± 0.002
^{235}U/U	1.9 ± 0.1
^{236}U/U	0.33 ± 0.02
^{238}U/U	97.7 ± 0.2

※1 Ratio of U = ^{234}U+^{235}U+^{236}U+^{238}U

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[1] Document 3-3 from the 140th Meeting of the Secretariat of the Team for Countermeasures for Decommissioning and Contaminated Water Treatment