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# Fabricating Fukushima Daiichi in-vessel and ex-vessel fuel debris simulants for the development and qualification of laser cutting technique

Christophe Journeau<sup>a</sup>, José Monerris<sup>a</sup>, Brigitte Tormos<sup>a</sup>, Laurent Brissonneau<sup>a</sup>,  
Emmanuel Excoffier<sup>b</sup>, Véronique Testud<sup>b</sup>, Christophe Chagnot<sup>c</sup>, Damien Roulet<sup>d</sup>

<sup>a</sup> *Commissariat à l'Energie Atomique et aux Energies Alternatives (CEA), Cadarache,  
13108 Saint-Paul-lez-Durance, France*

<sup>b</sup> *Commissariat à l'Energie Atomique et aux Energies Alternatives (CEA), Marcoule, 30207 Bagnols/Cèze, France*

<sup>c</sup> *Commissariat à l'Energie Atomique et aux Energies Alternatives (CEA), Saclay, 91191 Gif sur Yvette, France*

<sup>d</sup> *ONET Technologies, 26701 Pierrelatte, France*

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## Abstract

In their roadmap towards the decommissioning of Fukushima Daiichi reactors, the Japanese authorities plan to start removing fuel debris by the end of 2021. To reach this objective, several R&D projects have been launched and subsidized by the Japanese government. In this framework, a French consortium (COMEX Nucléaire/ONET Technologies, CEA and IRSN) has been selected to implement R&D related to the laser cutting of Fukushima Daiichi fuel debris and related dust collection technology.

Some of the key aspects of this project are to fabricate representative corium debris simulants, to test laser cutting technique on these simulants and to measure the aerosols and dusts that are released during the debris thermal cutting. This paper concentrates on the first issue.

Two representative corium compositions have been selected: in-vessel debris having the average of Fukushima Daiichi Unit 2 (1F2) lower head debris composition best estimates from the OECD Benchmark Study of the Accident at the Fukushima Daiichi Nuclear Power Station (BSAF); ex-vessel debris composition chosen from DOE calculations on Fukushima Daiichi Unit 1 (1F1) molten core concrete interaction in order to maximize the concrete content and bound the possible composition space.

Fission Products expected 10 years after plant shutdown have also been taken into account. As it was not possible within the current project timeframe to perform debris cutting tests with (depleted) uranium oxide, it has been necessary to simulate uranium oxide and hafnium oxide has been considered as the best option.

Based on its 20 year experience in corium experimental R&D, PLINIUS platform at CEA Cadarache has been chosen to fabricate these simulants. Two blocks of 8-10 kg have been induction melted. After melting and cooling, samples have been analyzed (microstructure and chemical composition) and laser cutting of these simulant blocks has been successfully carried out.

*Keywords:* Fukushima Daiichi accident, In-Vessel Corium, Ex-Vessel Corium, Corium Simulants.

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## 1. Introduction

In their roadmap towards the decommissioning of Fukushima Daiichi (1F) reactors [1], the Japanese authorities plan to start removing fuel debris by the end of 2021. To reach this objective, the fuel debris retrieval methods for the first implementing unit shall be selected during Japanese Fiscal year 2018. Cutting fuel debris (corium) is an important issue and several R&D projects have therefore been launched and subsidized by the Japanese government to study potential techniques. In this framework, a French consortium (COMEX Nucléaire / ONET Technologies, CEA and IRSN) has been selected to implement R&D related to the laser cutting of 1F fuel debris and related dust collection technology.

Laser technology dates back to the 60's. It has been used for industry applications for almost as long, and the feasibility of cutting applications was quickly demonstrated. In recent years, with the help of high-power lasers, this technology of laser-cutting has started to be applied to the extreme environments that can be found in nuclear facilities, especially in the case of nuclear decommissioning [2][3], where remote operations are often mandatory.

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\*Corresponding author

Email address: [christophe.journeau@cea.fr](mailto:christophe.journeau@cea.fr) (C. Journeau)

Asides from experimental optimization of the laser-cutting technique on commercially-available samples such as electrofused zirconia, steel and zirconium plates, one specificity of our approach is to have specifically fabricated simulant of Fukushima-Daiichi fuel debris that have in a second part been subjected to laser cutting in our facility while aerosol production has been analyzed.

This paper concentrates on the fabrication of representative samples. In a first section, the selection of two representative debris compositions, one for in-vessel corium, one for ex-vessel corium will be presented. Then the fabrication of the two simulant blocks will be described followed by material analyses, with a focus on the ex-vessel fuel debris simulant block. Finally, some information will be provided on their laser cutting.

## 2. Definition of debris simulant compositions

The objective of the debris simulant is to reproduce as far as possible typical conditions that are expected in the dismantling of Fukushima Daiichi debris with respect to laser cutting and aerosol production. Therefore, it is necessary to be as close as possible to typical compositions that are expected for 1F molten fuel debris: oxide component, metallic phase, non-volatilized fission products.

As appropriate room for the laser cutting and aerosol sampling of radioactive prototypic corium samples with depleted uranium was not available during the current phase of the project, it has been decided to utilize a simulant material. Hafnium has been considered as surrogate for uranium as the thermal properties of hafnium and uranium oxides are quite close [4]: melting points at respectively 3063 and 3033 K; thermal conductivities in solid state at close to  $2 \text{ W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$  in the solid state and the heat of fusions are of the order of  $100 \pm 30 \text{ kJ/kg}$  for both oxides; specific densities at room temperatures of 9 680 and  $10\,500 \text{ kg/m}^3$ . Nevertheless, uranium has 4 oxidation states (+3, +4, +5, +6) leading to the formation of 4 gaseous species in the U-O system (U, UO, UO<sub>2</sub>, UO<sub>3</sub>) two of which are quite volatile, while hafnium presents only the +4 oxidation state.

No known simulant can represent this complex chemistry [5]. Uranium isotopes contribute only to 7 millionths of the debris total radioactivity, as estimated for 10 years after the accident [6], so it will contribute to a very small fraction of the aerosol radioactivity, but it will nevertheless be a major part of the aerosol mass. Therefore the volatilization of uranium compound cannot be represented by our simulant whereas the thermal cutting can be satisfactorily appreciated.

### 2.1. Fission products

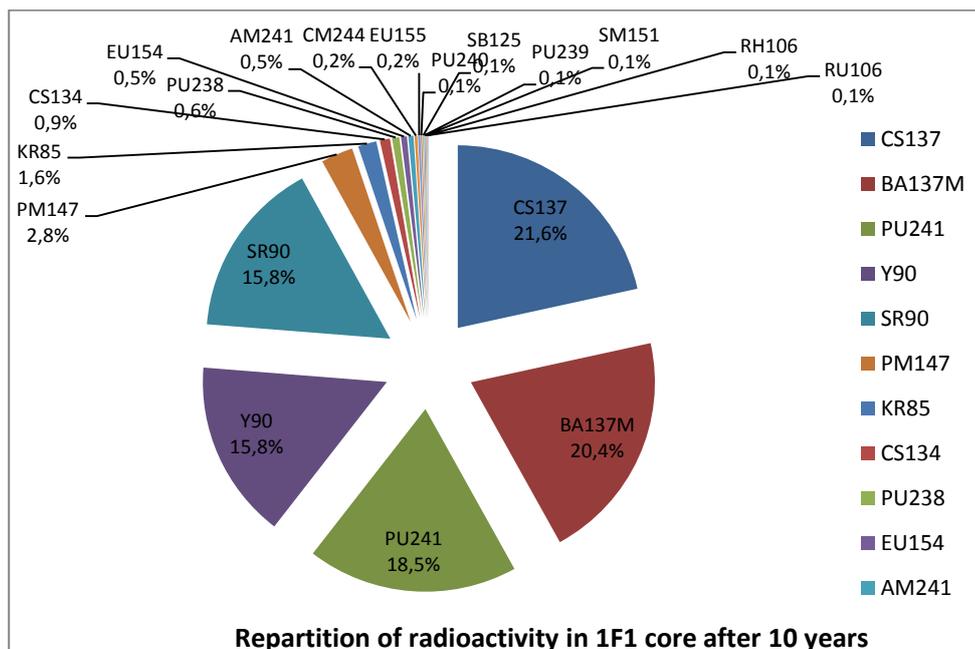


Figure 1 Repartition of 1F1 core radioactivity, 10 years after accident (from [6] data)

From JAEA published data on the isotopic composition of 1F reactor fuels [6], the core inventory 10 years after the accident is such that the isotopes with the largest radioactivity are (Figure 1): Caesium 137 (which as a volatile fission product must be largely out of the fuel debris), Barium 137 (the daughter of caesium-137, which must have similarly been largely released), Plutonium 241 (a low volatile with only 1 to a few percent release; ), Yttrium 90 (the daughter of strontium-90, another low volatile) and Strontium 90 (a low volatile). The repartition of masses is somewhat different due to the different half-lives and atomic masses. No major differences have been obtained between the 3 damaged plants in terms of isotopic composition 10 years after accident.

## 2.2. In-vessel fuel debris

Pellegrini et al. [7] have reported the debris compositions obtained by various teams on the benchmark on Fukushima Daiichi accident calculations organized by OECD/NEA (BSAF project). For the unit 2 lower head debris (Figure 4), the average between the lower head debris bed calculated by CIEMAT, CRIEPI, IAE and IBRAE is approximately:

37 wt% UO<sub>2</sub>, 37 % Zr, 16% ZrO<sub>2</sub>, 6% Stainless Steel, 3% Steel oxides, 1% B<sub>4</sub>C.

This correspond to an oxidation ratio of zirconium of C24<sup>1</sup>. This composition has been processed to include the largest minor elements taking into account the knowledge of 1F2 inventory (fission products, transuranians, etc.) as given by the compilation of Nishiara et al. [6]. The Stainless Steel has been considered as 304L steel.

Uranium has been replaced mole by mole by hafnium in the chosen simulants. Cerium has been chosen to simulate plutonium. For the fission products, natural isotopic composition are used instead of the isotopic composition to be found in reactor. This will allow for the same thermochemistry and aerosol properties while enabling experimentation outside of a hot laboratory, but it is less favorable for detection.

All elements corresponding to more than 0.01 wt% of the calculated debris composition have been considered, except Technetium which has no stable isotope and ruthenium which raises a health issue for workers in oxidic environment (this issue is negligible in presence of radioactive isotope where protective measures are anyhow taken). Altogether, more than 95% of the expected radioactivity after 10 years is simulated in the composition presneted in Table 1.

Table 1 In-vessel simulant designed composition (in g per 10 kg)

HfO <sub>2</sub>	3 019.29	SiO <sub>2</sub>	19.35
Zr	3 789.99	MoO <sub>2</sub>	9.37
ZrO <sub>2</sub>	1 983.70	Cs <sub>2</sub> O	6.64
Fe	619.90	Ba	4.29
Cr	177.19	La <sub>2</sub> O <sub>3</sub>	3.52
B <sub>4</sub> C	109.48	PdO	3.30
Ni	102.16	Pr <sub>2</sub> O <sub>3</sub>	3.30
SnO <sub>2</sub>	99.25	Sr	2.04
CeO <sub>2</sub>	19.17	Sm <sub>2</sub> O <sub>3</sub>	1.61
MnO	11.68	Y <sub>2</sub> O <sub>3</sub>	1.56
Nd <sub>2</sub> O <sub>3</sub>	11.71	TeO <sub>2</sub>	1.50

## 2.3. Ex-vessel fuel debris

In order to represent one typical Molten Core Concrete Interaction (MCCI) debris composition, we have considered the published works of Robb et al. [9] which modelled spreading and MCCI in Fukushima Daiichi unit 1 (1F1). They have considered several cases corresponding to different code calculations of the in-vessel breach. We have selected the MAAP calculation for the low pressure case (actually the high pressure case gives similar values). This has been made in such a way that the ex-vessel sample is considered with the maximum average concrete content from the surveyed configuration while the in-vessel sample has indeed 0% concrete. This correspond to a maximum fraction of 54 wt% concrete, assuming there was no mixing between the sump

<sup>1</sup> Molar ratio of oxidized zirconium to total amount of zirconium: C24 means than 24 mol% of zirconium have been fully oxidized to +4 state.

(where most MCCI is considered to have occurred) and the remainder of the corium spread (for which a lower fraction of concrete has been calculated) and that the densities of corium and concrete are respectively of 8000 and 2100 kg.m<sup>-3</sup>. Considering SURC experimental program [10] published composition for basaltic concrete and the fraction of iron reinforcement bars used in MOCKA experiments [11], and applying the same substitutions as for the in-vessel case, Table 2 presents the selected composition for the ex-vessel fuel debris simulant.

Table 2 Ex-vessel simulant designed composition (in g per 10 kg)

	For 10 000 g	wt%		For 10 000 g	wt%
HfO <sub>2</sub>	2 036.62 g	20.366%	Fe <sub>2</sub> O <sub>3</sub>	286.37 g	2.864%
CeO <sub>2</sub>	18.47 g	0.185%	Nd <sub>2</sub> O <sub>3</sub>	8.93 g	0.089%
ZrO <sub>2</sub>	1 240.32 g	12.403%	MoO <sub>2</sub>	8.46 g	0.085%
SnO	15.80 g	0.158%	Cs <sub>2</sub> O	5.04 g	0.050%
B <sub>4</sub> C	17.36 g	0.174%	Ba	3.33 g	0.033%
Cr <sub>2</sub> O <sub>3</sub>	58.11 g	0.581%	La <sub>2</sub> O <sub>3</sub>	2.66 g	0.027%
Fe	630.09 g	6.301%	PdO	2.55 g	0.026%
FeO	1 177.76 g	11.778%	Pr <sub>2</sub> O <sub>3</sub>	2.45 g	0.024%
Ni	52.04 g	0.520%	Sm <sub>2</sub> O <sub>3</sub>	1.83 g	0.018%
SiO <sub>2</sub>	3 039.51 g	30.395%	Sr	1.47 g	0.015%
Al <sub>2</sub> O <sub>3</sub>	758.62 g	7.586%	Y <sub>2</sub> O <sub>3</sub>	1.15 g	0.012%

### 3. Debris simulant blocks fabrication and analyses

#### 3.1. Ex-vessel fuel debris simulant

#### 3.2.

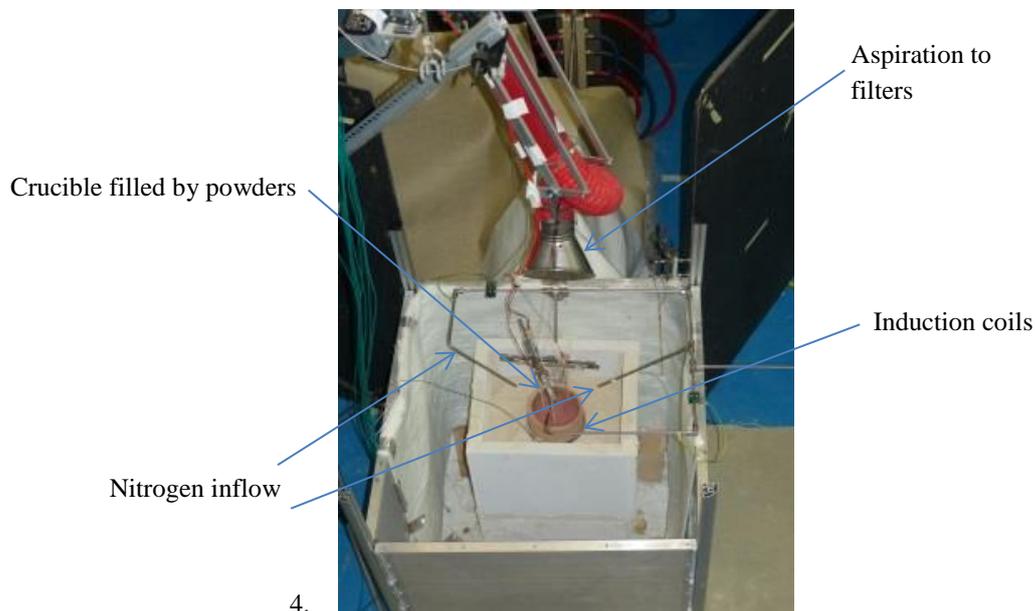


Figure 2 Experimental induction melting set-up

An Ex-Vessel (Molten Core Concrete Interaction) fuel debris simulant block has been fabricated at CEA Cadarache PLINIUS experimental platform on April 19th, 2016. About 10 kg of the load described in Table 2 have been inserted inside a Ø 180 mm x 290 mm-high (inner dimensions) magnesium-stabilized-zirconia (Zirconia 3001) crucible. The crucible is surrounded by a copper induction coil protected by oxidic powder (**Erreur ! Source du renvoi introuvable.**). Power has been initially induced to the steel balls using PLINIUS platform new induction generator. The load temperature progressively increased, leading to progressive melting of the load (Figure 3 left) and induction coupling to the oxide melt pool. When high temperatures (around 2000°C) have been reached, partial volatilization of some compounds occurred (Figure 3 center) as during a severe accident. Due to the important reduction of porosity between powder and melt, a cavity was formed above the

melt and below a partly sintered upper crust (Figure 3 right). Material from this crust progressively sank inside the melt. Then, the heating has been terminated and the fuel debris cooled. After removal of the remaining powder and of projections, about 8 kg of fuel debris simulant have been fabricated.

The high efficiency filter collecting aerosols has been weighed before and after the test. Less than 0.5 g of aerosols has been collected. The VF-03 filter has been transferred to CEA Cadarache LIPC material analysis laboratory and the collected aerosols have been examined with a ZEISS EVO HD15MA Scanning Electron Microscope (LaB<sub>6</sub> gun) equipped with an OXFORD SDD X-Max 50 Energy Dispersive Spectroscopy detector for chemical analysis.



Figure 3: Three steps of Ex-vessel 1F1 Fuel debris fabrication  
Left: start of melting phase – Center: Melting and aerosol release – Right: Cooling phase

The micrographs presented in this report are made with backscattered electrons (BSE). The grey levels mainly indicate composition differences: the higher is the density in heavier elements (high Z: Zr, Hf...) in a zone, the lighter it appears on the micrograph. The INCA software (Oxford Instrument) is used to process and analyze EDS spectra. An uncertainty of  $\pm 1/10^{\text{th}}$  of the measured concentration must be considered for each element.

Deposits on the filter fibers and some powder composed of small particles (less than 0.2  $\mu\text{m}$ ) or small droplets (0.5 to 5  $\mu\text{m}$  diameter, Figure 4) can be observed.

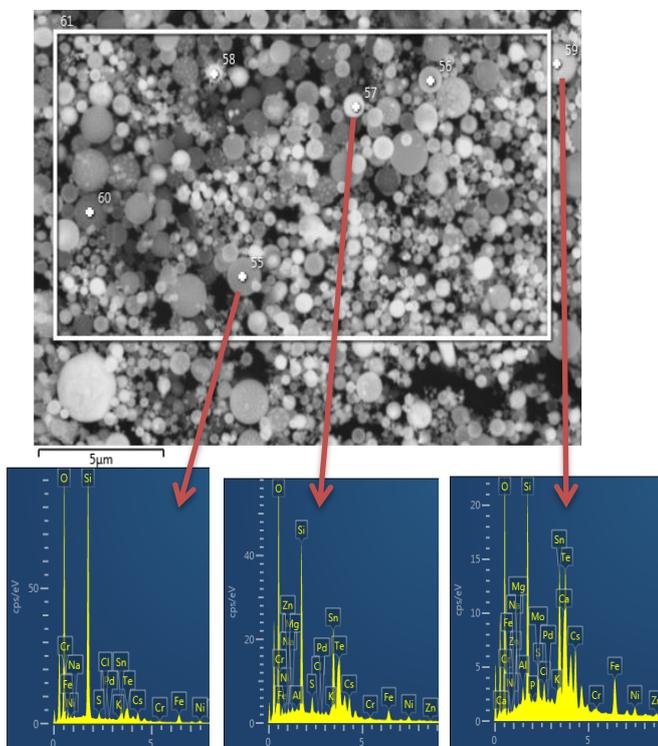


Figure 4: Small droplets in the powder. Note that very small droplets appear at the surface of bigger ones (the lighter-colored is a droplet, the more it contains the heavy elements Sn, Te, Cs).

The composition of the powder or of the droplets is very similar to the one of the deposits: the elements which are mainly and simultaneously detected are Si, Sn, Te, Cs, Cr, Fe, Ni and O (see **Erreur ! Source du renvoi introuvable.**), sometimes with C, Al, Pd and Mo.

Table 3 Volatilization of Ex-vessel simulant fuel debris during fabrication stage

Element	Load wt. %	Volatilization class[12]	Volatilization during melting
Hf	25.5%	Non volatile	Present in few droplets. Limited evaporation
Fe	23.1%	Semi volatile [13]	One of the main elements of aerosols.
Si	21.0%	Semi volatile	One of the main elements of aerosols.
Zr	13.6%	Non volatile	Present in few droplets.
Ca	6.5%	Semi volatile [13]	Present in few droplets.
Al	5.9%	Low volatile [13]	Detected in some aerosols
Cr	2.03%	Semi volatile [13]	One of the main elements of aerosols.
Ni	1.07%	Semi volatile [13]	One of the main elements of aerosols.
Mo <sup>2</sup>	0.30%	Semi-volatile	Detected in some aerosols. About 80-90% of the initial molybdenum has been released
Ce	0.22%	Low volatile	Not detected
B	0.20%	Semi volatile [13]	Not detectable
Sn	0.18%	<i>Not described</i>	One of the main elements of aerosols (as e.g. in Phébus FPT3 [14])
Nd	0.11%	Non volatile	Not detected
Cs	0.07%	Volatile	One of the main elements of aerosols. About 50% depletion from melt
Ba	0.05%	Semi-volatile	Not detected
La	0.034%	Low volatile	Not detected
Pr	0.033%	Non volatile	Not detected
Pd	0.031%	Semi-volatile	Detected in some aerosols
Sm	0.024%	<i>Not described</i>	Not detected
Sr	0.023%	Low volatile	Not detected
Te	0.014%	Volatile	One of the main elements of aerosols. Around 90% of the original tellurium has been released
Y	0.013%	Low volatile	Not detected

Some droplets of slightly larger size might have different compositions: Si-Fe-Cr-O. A few larger droplets (25-50 µm diameters) were observed which are likely to have escaped in liquid state from the molten pool. They contain: Si, Zr, Hf, Ca, Fe, Cr, Ni, O. Small peaks of Te, Cs and Sn are detected on these droplets. Large plates (>100µm) contains Si, K, Mg, Na, Al, Ti, O, Fe and a few rare blocks of ZrO<sub>2</sub> (>50 µm) and HfO<sub>2</sub> (>20 µm) can also be found.

Due to the collected mass of aerosols (<0.5 g in filter), it is unlikely that any element totally volatilized from the debris. From the analysis of the filter samples, the following evaporation behavior of simulant debris elements has been observed during the heating phase (somewhat simulating the volatilization during severe accident) and listed in **Erreur ! Source du renvoi introuvable.**. It corresponds well to the volatilization classes established for severe accident conditions in [12].

In order to keep a large block for laser cutting, samples have been taken from the periphery of the fuel debris simulant block in a first stage. One oxidic sample (from splashes above the free surface) and one metallic sample (from below) have been analyzed. A second series of analyses from the block center, has been carried out after it has been laser cut.

The samples have been transferred to CEA Marcoule LMAC laboratory, separated in several subsamples and then crushed in an inert mortar. Three dissolution techniques have been used: solubilization in HNO<sub>3</sub>-HCl-HF mixtures, acid solubilization under pressure in microwave furnace, solubilization using an alkaline flux. 98 to 100% dissolution has been obtained depending on the technique.

<sup>2</sup> Molybdenum was introduced both as fission product simulant and as part of stainless steel

Table 4 tentative composition (wt.%) of the two phases after fabrication process

Element	Oxide (periphery)	Oxide (core)	Metallic part
O	24 %	Not determined	Not determined
Hf	21%	13%	
Si	17%	11%	
Fe	14%	9%	81%
Zr	11%	20%	
Ca	5%	3.2%	
Al	4%	3.3%	
Cr	2%	1.0%	0.8%
Ce	0.2%	0.15%	
B	0.2%	0.01%	
Nd	0.9%	0.06%	
Sn	0.4%	0.06%	0.2%
Ni	0.03%	0.25%	14%
Cs	0.03%	0.03%	
La	0.02%	0.02%	
Pr	0.02%	0.02%	0.3-0.7%
Sm	0.02%	0.014%	
Sr	0.02%	0.015%	
Y	0.01%	0.025%	
Mo	0.01%	0.031%	0.4%
Pd	0.01%	0.005%	
Te	0.002%	<0.002%	0.015%

A tentative composition (in wt.%) for the debris can be proposed for the oxidic part (main phase of the debris) as well as for the metallic part, taking into account the initial load inventory, the measured sample compositions and the releases. It is listed in Table 4.

It occurs that the core composition is somewhat different with that of the periphery (which may not have completely been mixed and which has been less subject to volatilization, being rapidly solidified). Considering the inner core composition (which is the average of 7 subsample analyses) and the aerosol collection during the melting phase, release fractions during fabrication have been assumed in order to fit the observations and analyses for each element (except for palladium) and are presented in Table 5.

Table 5 Assumed released fractions during fabrication process

Element	Hf	Fe	Si	Zr	Ca	Al	Cr	Ni	Mo	Ce	B
	0.01%	0.2%	0.2%	0.01%	0.01%	0.1%	2%	2%	10%	0.01%	20%
Element	Sn	Nd	Cs	Ba	La	Pr	Pd	Sm	Sr	Te	Y
	30%	0%	40%	1%	20%	35%	?	0	0	90%	0

#### 4.1. In-vessel fuel debris simulant

Similarly, more than 10 kg In-Vessel fuel debris simulant has been fabricated at CEA Cadarache PLINIUS experimental platform during test VF-04 on April 28<sup>th</sup>, 2016 using a similar procedure as for the ex-sample. The major difference was that argon was blown above the test section instead of nitrogen. Figure 5 left presents the fabricated block.

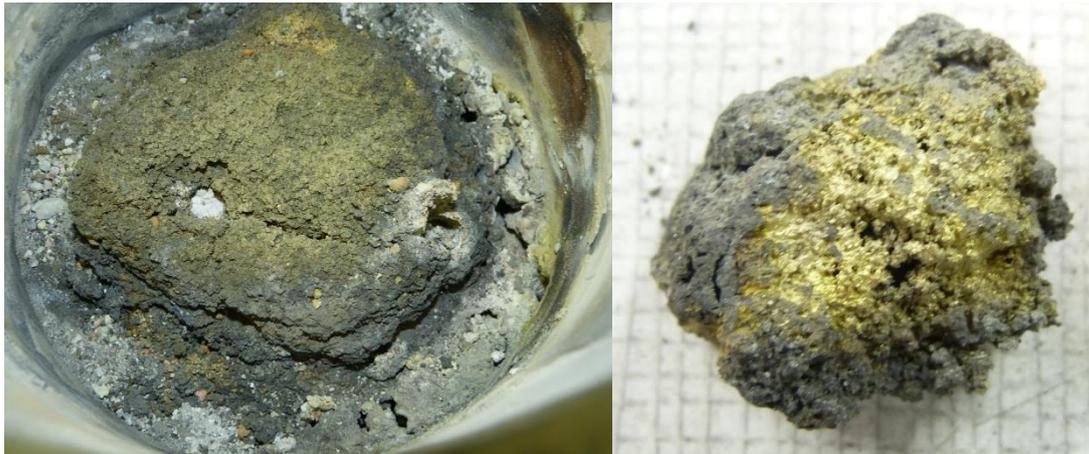


Figure 5: Left: Top-view of the in-vessel fuel debris simulant Right: sample for analysis

A sample has been taken from the fuel debris simulant block periphery (Figure 5 right). The same analysis procedure has been applied at LMAC laboratory in CEA Marcoule than for the ex-vessel oxidic samples. The averaged results of the analyses of 4 sub-samples dissolved with microwave furnace solubilization are reported in Table 6. Solubilization using an alkaline flux has also been used for confirmation (but is not reported here as some elements cannot be analyzed since they are also present in the alkaline flux). The uncertainty is estimated at 10% of the reported values. Similarly, a sample from the core of the block has been analyzed after laser cutting (Table 7).

The analysis of the debris sample shows that some of the major elements fractions are quite different from the initial composition. This is likely to be a consequence of the separation of VF04 melt in two liquids, an oxidic melt and a metallic melt, while only the oxidic melt has been found at the surface. No stratified melt has been found when the block has been cut, but it seems that different phases must have been present since the composition varied a lot from a sub-sample to the other. The fact that the highest standard deviations correspond to the metallic phase elements (Fe, Cr, Ni, Sn, Mo, Te) support this hypothesis. Further works are needed to better characterize this heterogeneous in-vessel block.

Table 6: Averaged measured composition of in-vessel oxidic sample (periphery)

Elements	Zr	Hf	Sn	B	Fe	Ce	Nd	Mo	Cr	Pd	La
wt. %	44	31	1.3	1.2	0.40	0.30	0.11	0.11	0.046	0.042	0.031
Elements	Al	Ni	Pr	Sr	Y	Sm	Te	Si	Ca	Ba	Cs
Wt. %	0.030	0.027	0.018	0.016	0.017	0.015	0.0049	<0.06	<0.06	<0.002	<0.002

Table 7: Averaged measured composition of in-vessel oxidic sample (core of block)

Elements	Zr	Hf	Sn	B	Fe	Ce	Nd	Mo	Cr	Pd	La
wt. %	46	17	0.2	0.1	9.1	0.14	0.1	0.02	1.6	0.004	0.026
Elements	Al	Ni	Pr	Sr	Y	Sm	Te	Mg	Ca	Ba	Cs
Wt. %	0.14	0.3	0.018	0.014	0.017	0.011	0.003	0.05	0.055	0.003	<0.002

## 5. Debris simulant blocks laser cutting

Laser-cutting has been performed in air at CEA CELENA laser shot unit of the ALTEA platform. A Trumpf 8002-type 1030-nm laser source with continuous power of 8 kW is connected by optical fibers to CEA laser head positioned on a PRIME 3-axis robot. Two compressed air jet flows are provided, one around the laser head and one at 45° from the corium surface (Figure 6).

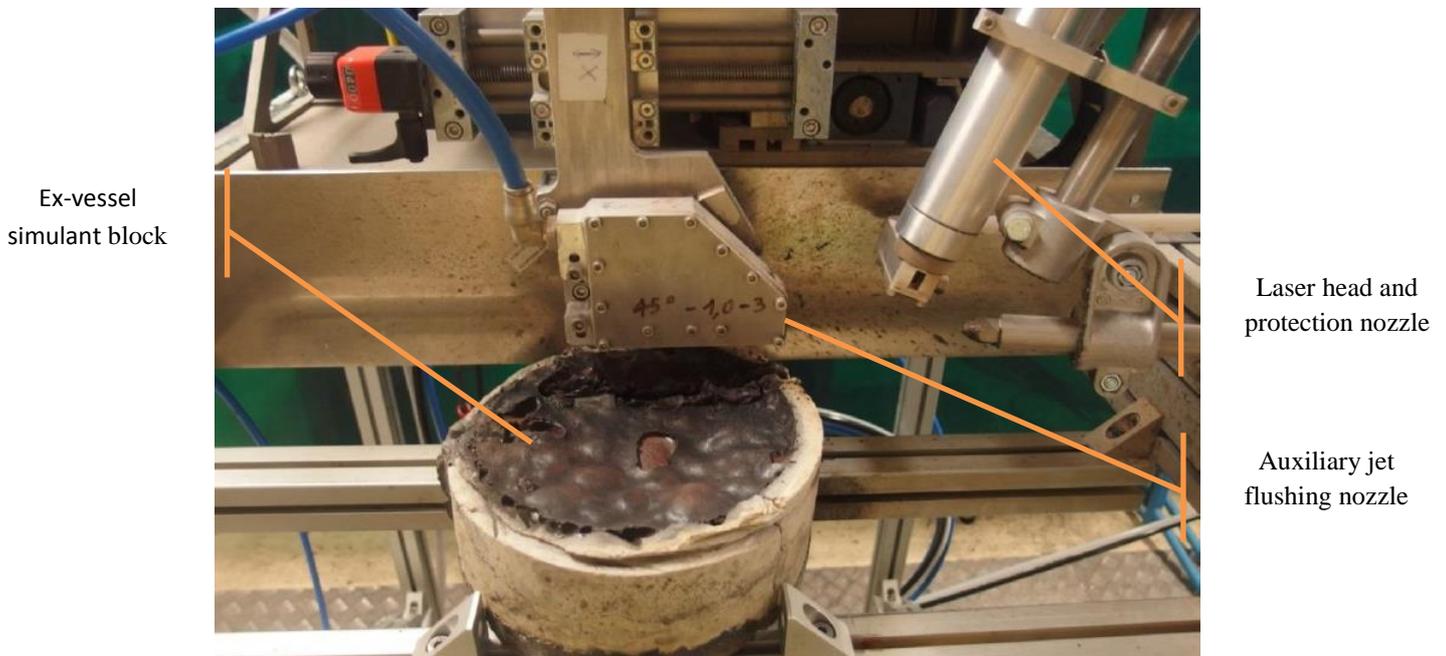


Figure 6: Ex-vessel corium simulant in the CELENA laser shot unit

The laser head has been moved at a speed of 15 cm/min and the two corium simulant blocks have been both cut to a depth of approximately 40 mm (Figure 7). The grooves are clear and clean and little adhesive scoria accumulates at their sides, so that the resulting corium-head distance is relatively unaffected. From the collection of aerosols and dross, it is estimated that about 10 g are ejected far of the block when 1 meter of corium is being laser cut.



Figure 7: Cut In-vessel (left) and Ex-vessel (right) corium simulant blocks.

## 6. Conclusions and perspectives

Two representative corium compositions have been selected: in-vessel debris having the average of Fukushima Daiichi Unit 2 (1F2) lower head debris composition best estimates from the OECD Benchmark Study of the Accident at the Fukushima Daiichi Nuclear Power Station (BSAF); ex-vessel debris composition chosen from DOE calculations on Fukushima Daiichi Unit 1 (1F1) molten core concrete interaction in order to maximize the concrete content and bound the possible composition space. Fission Products expected 10 years after plant shutdown have also been taken into account. As it was not possible within the current project timeframe to perform debris cutting tests with (depleted) uranium oxide, it has been necessary to simulate uranium oxide and hafnium oxide has been considered as the best option.

Two blocks of 8-10 kg have been induction melted. After melting and cooling, samples have been analyzed (microstructure and chemical composition) and laser cutting of these simulant blocks has been successfully carried out.

At the time of writing, these corium simulant blocks are being laser cut in air and underwater while released aerosols are being measured and sampled. Further material analyses are also ongoing. Nevertheless current analyses have indicated that the found collected aerosols correspond well to the volatilization classes established for severe accident conditions.

In a further stage, it is proposed to fabricate prototypic fuel debris including depleted uranium and ruthenium and to laser cut them in a specifically adapted space at CEA, in order to improve the representativity of the laser cutting tests with respect to the release of volatiles.

This experimental R&D is aiming at industrial application on site if requested by Fukushima Daiichi fuel debris retrieval strategy that will soon be decided by the Japanese authorities.

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