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Pascal Zavaleta, Mickael Coutin, Thomas Gelain, Jocelyne Lacoue, Philippe March, et al.. Presentation and first outcomes of the FIGARO research program that aims at assessing the airborne release fraction of plutonium dioxide during glove box fires in nuclear fuel fabrication and reprocessing plants. Mechanical Engineering Journal, 2024, 11 (2), pp.23-00463. 10.1299/mej.23-00463 . hal-04665171

HAL Id: hal-04665171

<https://hal.science/hal-04665171v1>

Submitted on 31 Jul 2024

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Presentation and first outcomes of the FIGARO research program that aims at assessing the airborne release fraction of plutonium dioxide during glove box fires in nuclear fuel fabrication and reprocessing plants

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Received: 19 October 2023; Revised: 14 December 2023; Accepted: 17 January 2024

Abstract

Fires in nuclear fuel fabrication and reprocessing plants can cause the rupture of the glove box (GB) containment with a risk of dispersion of plutonium dioxide (PuO_2) within the facility. Fire safety analyses need to assess the resulting radiological consequences to strengthen the appropriate prevention and protection measures in these plants. To this end, the French Institute for Radiation Protection and Nuclear Safety (IRSN), in partnership with the Japanese Nuclear Regulation Authority (NRA), has carried out since 2019 a research project that aims at assessing the airborne release fraction (ARF) of PuO_2 involved in GB fires. This project, named FIGARO (Fires Involving Glove boxes with Aerosol Release Occurrences) follows a progressive approach, that has started with small and medium-scale analytical tests to study separately the various mechanisms involved in the GB fires and the PuO_2 airborne release and will end with large-scale GB fire tests to transpose and validate the analytical works for realistic assessments of the ARF of PuO_2 both in open and confined conditions. This project also includes the development of a GB fire model in the IRSN SYLVIA and CALIF³S-ISIS softwares and its validation. In addition to presenting the FIGARO program, this paper provides its first outcomes.

Keywords: Airborne release fraction, Electrostatic precipitator, Fire, Glove box, Kyowaglas surrogate, Plutonium dioxide surrogate

Nomenclature

ARF	Airborne release fraction
CFD	Computational fluid dynamic
ESP	Electrostatic precipitator
FIGARO	Fires Involving Glove boxes with Aerosol Release Occurrences
GB	Glove box
HEPA filter	High-efficiency particulate air filter
HRR	Heat release rate (kW)
ICP-MS	Inductively coupled plasma mass spectrometry
IRSN	Institute for Radiation Protection and Nuclear Safety
MDH	Magnesium hydroxide
MO _x	Mixed Oxides

NRA	Nuclear Regulation Authority
PC	Polycarbonate
PMMA	Poly(methyl methacrylate)
PuO ₂	Plutonium dioxide
UO ₂	Uranium dioxide
t ₀	Duration of the incubation stage
wt	Weight

1. Introduction

During the production and reprocessing of nuclear fuel rods, mixed oxide (MOx) powders composed of plutonium dioxide (PuO₂) and uranium dioxide (UO₂) are handled within static containment devices such as glove boxes (GBs) (Dorrian and Bailey, 1995; Massiot et al., 1998). Accidents in nuclear fuel fabrication and reprocessing plants such as a spill, explosion, or fire event, can lead to radiological consequences due to the airborne release of contaminants that these accidents can generate (DOE Handbook, 1994). The current work only addresses the study of the effect of a GB fire on the airborne release of contaminants. Throughout the life cycles of the GBs, the handling and processing of fuel rods induce the release of contaminants, in the form of micron-sized particles, which can be transported and deposited on the GB walls potentially made of polymers such as polycarbonate (PC) and poly(methyl methacrylate) (PMMA). A fire that can occur inside the GB (through pyrophoric reactions/solvents ignition, for example) or outside the GB from a neighboring fire source (electrical cabinet fire...), can cause the rupture of the GB containment (Ohno et al., 2021) with a risk of dispersion of the radioactive elements within the facility and potentially into the environment (Fernandez and Burghoffer, 1995; Nuclear Energy Agency, 2009). The Factory Mutual Research Corporation listed 24 fires and 19 explosions between 1956 and 1965 on laboratory GBs operated by the United States Atomic Energy Commission (Factory Mutual Research Corporation, 1967). The most significant contamination accident resulting from a GB fire occurred at the Rocky Flats plant in the USA in 1957 (U.S. Department of Energy, 1995). Plutonium shavings in a GB located in the Plutonium Recovery and Fabrication Facility building spontaneously ignited. The fire spread to the flammable GB materials. The fire rapidly spread through the interconnected GBs and ignited the large bank of high-efficiency particulate air (HEPA) filters located in a plenum downstream. Within minutes the first filters had burned out, allowing plutonium particles to escape from the building exhaust stacks. The building exhaust fans stopped operating due to fire damage, which ended the majority of the plutonium release. Another major fire occurred in the Rocky Flats plant on May 11, 1969, in the Plutonium Processing Facility building (Flack and Meyer, 2007). As for the fire of 1957, the spontaneous combustion of plutonium shavings in the GB was the cause of this disaster. While the fire showed marked similarities to the 1957 fire, the level of contamination was less severe because the HEPA filters in the exhaust system did not burn (after the 1957 fire, the filter material was changed from cellulose to non-flammable fiberglass). A third GB fire occurred again at Rocky Flats Environmental Technology site on May 6, 2003, in a GB during decommissioning (Massie, 2003). For the French nuclear fuel fabrication and reprocessing plants, 21 incidents related to a fire start on a GB or a confined enclosure were recorded over the period 1988-2008; one third of these incidents led to a loss of their containment.

The total activity corresponding to the release of nuclear materials during a fire, also called "fire source term", is an input to the calculation of the radiological consequences that are required by the fire safety analyses to strengthen the appropriate prevention, protection and fire-fighting measures in the related nuclear plants. A major point of discussion when considering the radiological consequences of fires in nuclear facilities is the airborne release fraction (ARF) values of contaminants to be considered. In general, this ARF is defined as the ratio between the mass of particles released in the airborne phase (in the form of an aerosol) and the mass of particles initially deposited on the surface under consideration (Ouf et al., 2013). This last study proposed a review of values reported in the literature for realistic materials and significant discrepancies were discovered for polymeric materials such as PMMA or PC that can be the main component of GB walls. These high disparities can have significant consequences in terms of decision making during a crisis, such as the geographical perimeter to be secured. Thus, the Institute for Radiation Protection and Nuclear Safety (IRSN), in partnership with the Japanese Nuclear Regulation Authority (NRA), has been running since 2019 a research project that aims at reducing the uncertainties related to the assessment of the ARF of PuO₂ involved in GB fires. This project, named FIGARO (Fires Involving Glove boxes with Aerosol Release Occurrences), first includes

two complementary tasks for separately studying the various mechanisms involved in the GB fires (task 1) and the airborne release of PuO₂ (task 2). These works are especially dedicated to analytical approaches based on small and medium-scale experimental activities for a thorough characterization of the above phenomena. The first task of the FIGARO program also includes modelling activities that regard the development of a GB fire model and its validation, for the two-zone model SYLVIA software (Plumecocq et al., 2017) and the computational fluid dynamic (CFD) CALIF³S-ISIS software (Bascou et al., 2019), both developed by IRSN. Finally, the third task of the FIGARO program aims at transposing and validating the analytical works for realistic assessments of the ARF of PuO₂ during large-scale GB fire tests.

This document first indicates baseline information on the FIGARO program. The second part then presents in detail this program, before providing in the third part its first outcomes.

2. Baseline information on the FIGARO program

This part aims at specifying first the retained representative scenario (regarding the GB configuration, the wall contamination and the GB ignition) and next, the progressive approach which has been adopted to conduct this research program.

2.1 Representative scenario considered in the FIGARO program

A wide range of GBs can be found in French nuclear fuel fabrication and reprocessing plants. These GBs can change by the type of the confinement panels made of either polymeric materials (PC, PMMA, CORIAN¹) or stainless steel, their volume from one to several tens of cubic meters, the presence and nature of biological protection panels, the air gap thickness between the confinement and protection panels and the presence of equipment on the GB panels (type, number and position). The biological protection panels, that usually equip the working sides of the GBs in the nuclear industry, can be made of either Kyowaglas (70 %wt of PMMA and 30 %wt of lead) or leaded glass. The visits of French nuclear plants allowed to select representative values of the parameters related to the GB configurations, particle contaminations in GBs and possible modes of GB ignition. Thus, as part of the FIGARO program, the following items were adopted to specify the studied representative scenario:

- the vertical confinement panels of the GB are made of either PC or stainless steel,
- the horizontal confinement panels of the GB are made of stainless steel,
- the 50 mm thick biological protection panels are made of PMMA or Kyowaglas,
- the air gap between the confinement and protection panels is 30 mm thick,
- the working sides of the GB are equipped with glove holes,
- the GB is not equipped with a filtration system,
- the contamination is spread homogeneously over all the polymeric internal surfaces of the GB,
- the ignition source is outside of the GB².

Regarding the key issue of the specification of the contaminated particle deposition characteristics on the internal walls of the GB, a synthesis of works carried out at IRSN (Ouf et al., 2020) highlighted that PuO₂ deposits on the latter walls tend to be homogeneous, relatively mono-layered and with contamination levels ranging from a few µg/cm² to a few mg/cm². These deposition characteristics are considered in the FIGARO program.

2.2 Progressive approach adopted to conduct the FIGARO program

The FIGARO program has first addressed two complementary tasks to study separately the several mechanisms involved in the GB fires (task 1) and the airborne release of PuO₂ (task 2). These analytical works have especially

¹This material is composed of one third of PMMA and two third of trihydrate d'aluminium.

²External ignition of the GB seems the most probable given that nearly all GBs are filled with an inert gas such as nitrogen, Helium or Argon, nowadays (Ohno, at al., 2021).

concerned small and intermediate scale experimental works that used specific reduced-scale test devices. These apparatuses indeed allow to conduct a significant number of experiments under controlled conditions to thoroughly investigate one of the above phenomena. These experiments also target to support the specification of the large-scale GB fire tests and the development of a database for the validation of the modelling activities. Thus, the controlled-atmosphere CADUCEE medium-scale calorimeter (Coutin et al., 2016), the reduced-scale glove box fire simulator SIMBAG and the ARTEMIS experimental test bench (Ouf et al., 2013), have been used successively to deeply characterize the combustion of GB materials in a controlled and under-oxygenated atmosphere, the GB combustion in open and confined conditions and the airborne release of PuO₂. The second task, related to the analytical study of the airborne release, also includes the development and validation of the aerosol metrology that will be used to assess the ARF of PuO₂ at real-scale during the GB fire tests of task 3. Another major objective of tasks 1 and 2 is the identification of a surrogate for Kyowaglas materials (possible presence of lead in the smoke produced during the GB fire tests) and one for PuO₂ particles (radiotoxic), respectively. Their related safety issues are indeed not suitable with their use in the fire test facilities of IRSN. Thus, it is required to determine for the Kyowaglas materials one surrogate material that has a similar fire behaviour, and for PuO₂ particles, surrogate particles that provide a close behaviour regarding their airborne release during a fire.

The third and last task of the FIGARO program aims at transposing at real-scale the analytical works of tasks 1 and 2 to first validate the fire behaviour of a GB equipped with Kyowaglas surrogate panels and next, to assess the ARF of the PuO₂ surrogate particles involved in the GB fire tests. The latter will be first conducted in open atmosphere, in the well-ventilated and large facility SATURNE that allows an oxygen limitless availability for a full fire development. The GB fire tests will be next conducted in the PLUTON facility that involves a confined and mechanically-ventilated enclosure of 400 m³ in volume. For such conditions, the more reduced oxygen availability than that obtained in open atmosphere conditions, can limit the fire development, as it was observed for another type of fuel³ (Zavaleta et al., 2019).

3. Presentation of the FIGARO program

This section first describes the small and medium-scale fire characterizations of GB materials (PMMA...) of task 1, before presenting the works of task 2 dedicated to the in-depth characterization of the PuO₂ airborne release and to the development of a suitable aerosol metrology adapted to large-scale tests. The last and third task addresses the large-scale GB fire tests devoted to the realistic assessments of the ARF of PuO₂

3.1 Analytical GB fire characterizations (task 1)

3.1.1 Preliminary tests

IRSN carried out between 2013 and 2017 a preliminary series of seven tests to characterize the fire behaviour of a glove box (GB) in open atmosphere conditions and to assess the thermal, chemical and aerodynamic stresses that the radioactive particles could undergo during the fire (Coutin and Audouin, 2017). The tests used a GB of about 1 m³ in volume that was equipped with containment panels made of either PC with glove holes for the handling of radioactive material, or stainless steel (Fig. 1-a). This GB also used PMMA panels (with glove holes) placed over the PC panels, both representative of the GBs working sides. The PMMA panels simulated biological protection panels made of Kyowaglas. This test series provided significant insights related to the combustion regimes in the GB, as obtained in open atmosphere conditions. The combustion regime in the GB turned out to be quasi-complete (i.e., with a combustion efficiency close to unity), promoted by the GB configuration being similar to a “well-stirred reactor” (Fig. 1-b), with swirling flames being observed inside the GB fire source. The mean temperatures measured between 1000 and 1200 °C inside the GBs for all the tests, notably higher than those of typical turbulent diffusion flames (about 800-900 °C), also corroborated the combustion conditions operating inside a “well-stirred reactor”. Furthermore, these outcomes also raised questions related to the effect on the GB combustion regimes, of the air inflow rate in the GB, the GB

³A fuel, such as PC or PMMA, is specified as a material that releases energy when it burns while a nuclear fuel, such as PuO₂, is a material that produces energy when it undergoes nuclear fission in a nuclear power plant.

configuration (number of fuel panels, the panel type) and under-oxygenated conditions typical of fires in confined rooms of nuclear installations. To tackle these matters and to supply data supporting the development and validation of GB fire models, the first part of the FIGARO program therefore addresses analytical combustion studies involving either GB material samples (PMMA, Kyowaglas...) or a medium-scale glove box simulator.

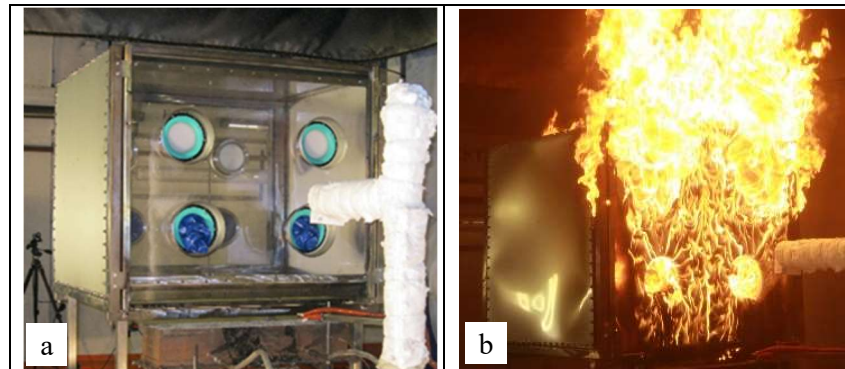


Fig. 1 Glove box used as part of the preliminary test series (Coutin and Audouin, 2017), before fire (a), during fire (b).

3.1.2 Analytical study of solid panel combustion

One goal of this work is to investigate the effect of under-oxygenated conditions on the combustion of vertically-oriented PMMA slabs. The collected data aim at proposing analytical correlations that could be implemented in fire softwares for improving their predictive abilities in the above conditions. This analytical investigation is conducted using the controlled-atmosphere calorimeter, named CADUCEE (Coutin et al., 2016), as seen in Fig. 2. This experimental device was first used by (Alibert et al., 2017) who especially investigated the impact of under-oxygenated conditions on the combustion of horizontally-oriented PMMA slabs and proposed correlations e.g., for the mass loss rate according to the oxygen concentration.

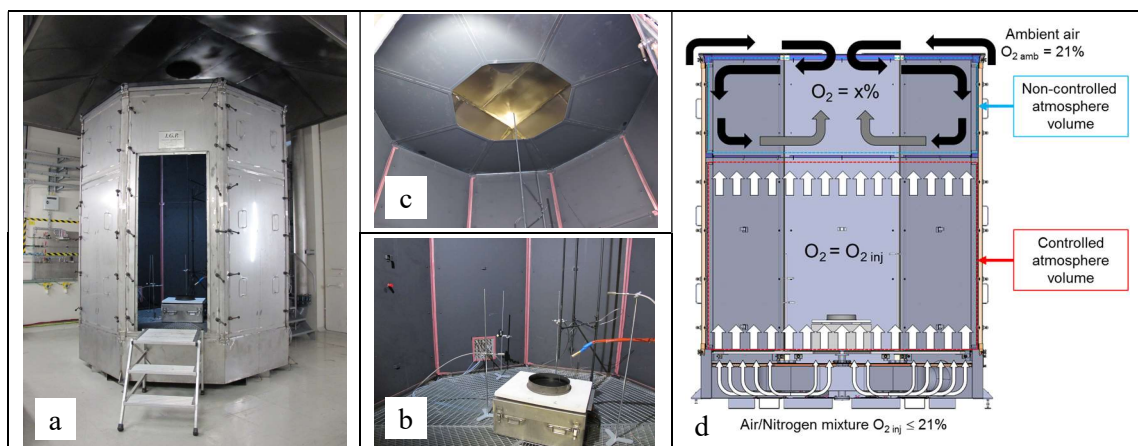


Fig. 2 CADUCEE experimental device (a), bottom inside view (b), top inside view (c), operating principle (d). The combustion chamber is a 304L stainless steel octagonal enclosure (a). This enclosure is 3.1 m high with an inscribed circle diameter of 2.9 m and a circumscribed circle diameter of 3.14 m. The enclosure floor (b), of 7 m² area, corresponds to the top grating floor of the oxidant diffuser. An assembly composed by steel trapezoidal panels (c) reduces the free section to 1.2 m² at the top of the combustion chamber. This open roof prevents fresh air, entering the enclosure through the top of CADUCEE, to disturb the controlled-atmosphere zone in the lower part (d).

Furthermore, despite the widespread use of biological protection panels made of Kyowaglas in the nuclear industry, few data related to the fire behaviour of this material are available. This is mainly explained by the high toxicity of its lead compounds (El-Khatib et al., 2019). Accordingly, as part of the FIGARO program, the Kyowaglas

fire behaviour investigations are limited to small-scale characterizations using the cone calorimeter and following a challenging safety protocol including numerous health measures. Given that such a safety protocol would be incompatible with the conduct of large-scale GB fire tests in the IRSN test facilities, a non-toxic surrogate material with fire behaviour close to that of Kyowaglas must be identified. To this end, the preliminary small-scale fire characterizations of the Kyowaglas material are also crucial to figure out the appropriate surrogate material exhibiting similar fire behaviour.

3.1.3 Medium-scale fire tests using a glove box simulator

To carefully investigate the complex combustion regimes observed during the preliminary GB fire tests, this part deals with three analytical test campaigns using the medium-scale glove box test simulator, named SIMBAG. The objectives of the first two test campaigns are to study the impact of thermal and aerodynamic conditions on the combustion regimes in the medium-scale glove box, for open and confined atmospheres. The last test campaign aims at studying in open atmosphere conditions the impact of the surrogate of Kyowaglas materials on the combustion regimes in the SIMBAG device.

3.1.4 Model development and validation

In parallel to the above multi-scale GB fire characterizations and the related experimental database development, one main goal of the FIGARO program is to develop the abilities of the IRSN softwares to predict GB fires and their consequences in confined and mechanically-ventilated compartments. This section presents the modelling and validation works planned for the two-zone model SYLVIA software and the CFD CALIF³S-ISIS software. The simulation of a glove box fire is complex due to various phenomena occurring in the glove box, its loss of geometry during the fire, large vortex structures, the strong turbulent mixing triggered by hydrodynamic instabilities developing near the lower fresh gas intakes and the heat transfer dominated by radiative effects. No theory to date has been put forward on how to model all aspects of the problem. A global modelling approach is therefore proposed, based on the description of the successive main events that occur during the development of a GB fire (Coutin and Audouin, 2017): the flame spread over the PMMA/Kyowaglas protection panel (where the fire is initiated), the melting and relocation of the PC containment panels at the GB bottom and finally the rapid combustion phase that involves the two combustible protection panels and the melted PC. The IRSN GB fire database, supplied by the ongoing multi-scale fire characterizations, aims at validating the global GB fire model. The latter will be implemented first in the SYLVIA software and afterwards leveraged in the CALIF³S-ISIS software.

3.2 Analytical study of the particle airborne release of PuO₂ (task 2)

This section first describes the analytical investigations related to the PuO₂ airborne release at small-scale and next those at medium-scale using the glove box simulator SIMBAG. The third part describes the efforts devoted to the development of a suitable aerosol metrology adapted to large-scale tests.

3.2.1 Analytical study of PuO₂ surrogates

Due to its high radiotoxicity, PuO₂ powder cannot be used for conducting experimental studies regarding its resuspension potential under the complex thermal stresses representative of fires. Nevertheless, few data regarding the criteria associated with the choice of surrogates for PuO₂ particles have been presented so far. The first goal here is therefore to identify the major properties of particles impacting their airborne release fraction during thermal degradation of the PC material. This material is indeed the component of the containment panels of the GBs where the radioactive particles can be deposited. Based on this work, the next step targets to identify a surrogate of PuO₂ powder with the adequate properties regarding the airborne release. These efforts also include the methodology for controlling the size dispersion and morphology to get the appropriate PuO₂ surrogate powder. Afterwards, experiments with the PuO₂ surrogate particles are planned to be conducted in the ARTEMIS test bench (Ouf et al., 2013) at small-scale to

identify the experimental conditions corresponding to the highest ARF. Finally, as part of the last phase of the FIGARO program, large-scale GB tests with wall deposits of the PuO₂ surrogate will be carried out for assessing its ARF under real fire conditions.

3.2.2 Preliminary study of the airborne release of PuO₂ using the medium scale glove box SIMBAG

Preliminary tests at medium-scale using the SIMBAG device have been designed to first validate metrology points and next to measure the ARF of the PuO₂ surrogate particles. The latter purpose is especially intended to support the specification of the large-scale GB tests (task 3) which will assess the ARF of the PuO₂ surrogate particles for real GB fire conditions. For all tests of this test campaign and in compliance with the representative scenario specified in section 2.1, the PuO₂ surrogate particles will be deposited on the PC walls of the SIMBAG device according to the deposition characteristics that were also indicated in section 2.1.

3.2.3 Development and validation of the aerosol metrology for assessing the ARF at large-scale

This task consists in designing, developing and validating the aerosol sampling lines that will allow the ARF assessment of the PuO₂ surrogate during the large-scale GB fire experiments.

3.3 GB fire tests (task 3)

The third and last task of the FIGARO program aims at transposing the previous analytical works of tasks 1 and 2 to a large scale to validate first the fire behaviour of the GB equipped with the Kyowaglas surrogate panels and next, to assess the ARF of the PuO₂ surrogate during realistic GB fires.

3.3.1 GB fire tests involving the Kyowaglas surrogate

Tests are planned to validate the fire behaviour of the GB equipped with the Kyowaglas surrogate panels. To this end, IRSN previously requested SP Fire Research of Sweden to conduct a fire test with the same 1 m³ volume GB as the one used for the preliminary tests (Coutin and Audouin, 2017) but equipped with Kyowaglas protection panels. This test was carried out under a calorimeter hood where lead-contained releases can be handled. The validation of the Kyowaglas surrogate panels therefore relies on the expected fire behaviour similarity between the GB tests conducted as part of this task and that performed at SP Fire Research.

3.3.2 GB fire tests involving both the Kyowaglas and PuO₂ surrogates

This part deals with the tests that will involve both the Kyowaglas and PuO₂ surrogates to provide an assessment of the ARF of the PuO₂ surrogate for realistic GB fires. These tests are planned in open atmosphere conditions and in confined and mechanically-ventilated conditions to investigate the impact of the test conditions on the ARF assessment. The collected data will support the validation of the abilities of the IRSN SYLVIA and CALIF³S-ISIS softwares, both using the earlier developed GB fire model, to simulate large-scale GB fire tests and their consequences in confined compartments. In addition, the data related to the ARF of the PuO₂ surrogate will be capitalized in the IRSN BADIMIS database which aims at collecting the available resuspension factors and those obtained in the present experiments. These efforts will contribute to support the safety analyses regarding the potential release of PuO₂ particles during accidental GB fires in nuclear fuel fabrication and reprocessing plants. To this end, the BADIMIS database could provide input data for simulations by the SYLVIA software of the transport and deposit of the PuO₂ particles in the rooms and ventilation networks of nuclear facilities.

4. First outcomes of the FIGARO program

This part provides the first outcomes of the FIGARO program obtained so far regarding the analytical GB fire characterizations (task 1), the analytical study of the particle airborne release of PuO₂ (task 2) and the GB fire tests

(task 3).

4.1 Analytical GB fire characterizations (task 1)

This section discusses the analytical combustion studies of PMMA and Kyowaglas materials, before presenting the main results of the first two test campaigns that used the medium-scale glove box SIMBAG simulator. The last part presents the first phase of the GB fire model development in the SYLVIA software.

4.1.1 Analytical study of solid panel combustion

The first step of the combustion study of vertically-oriented PMMA samples (Fig. 3) was performed in the controlled-atmosphere calorimeter CADUCEE device (Fig. 2) by varying the oxygen (O_2) concentration from 21 to 17 vol%. The latter value indeed corresponds to the extinction limit measured in the test conditions of this first step, that is similar to that obtained for horizontally-oriented PMMA samples (Alibert et al., 2017). The size of the PMMA sample was either 200×200 , 400×400 or 600×600 mm². This first step highlights, for all the considered PMMA sample sizes, that one of the key combustion parameters (the fuel mass loss rate) exhibits a linear behaviour versus the oxygen concentration in the 17-21 vol% range.

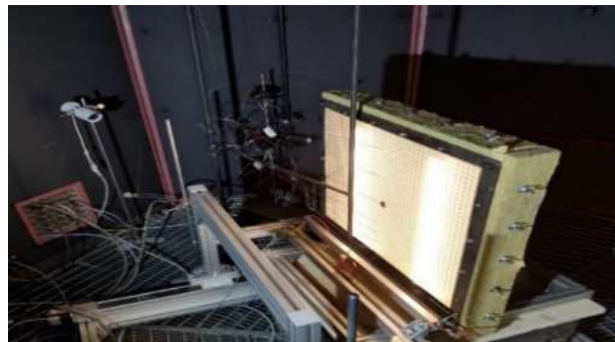


Fig. 3 Vertical test setup implemented in the CADUCEE controlled-atmosphere calorimeter.

The second step targets to obtain more representative confined fire conditions for which the radiation by the smoke accumulated in the room and the hot walls can impact the fuel pyrolysis. To this purpose, an additional heater was recently implemented in the CADUCEE device and will induce a decrease of the O_2 concentration limit, leading to fire extinction.

Concerning the analytical study of Kyowaglas material combustion, tests were first conducted with the cone calorimeter to increase the knowledge of its fire behaviour (Mastori et al., 2021). This investigation showed that, as the Kyowaglas burns (Fig. 4-a), metallic lead is formed and accumulates at the top surface slowing down the heat transfer to the condensed phase. This confers a substantial thermal stability on the Kyowaglas. In addition, at the end of the cone calorimeter test, lead oxide beads formed on the surface of the residue, as seen on Fig. 4-b, due to the oxidation of metallic lead. These insights contributed to support the identification of a surrogate of Kyowaglas. Thus, a composite made of PMMA and magnesium hydroxide (MDH, $Mg(OH)_2$) was selected among several candidates. MDH is indeed an efficient flame retardant that significantly reduces the heat release rate (HRR) (Tang Hao et al., 2013). In addition, the cone calorimeter tests conducted for the Kyowaglas material and PMMA-MDH composite samples of variable composition showed that the closest HRRs between these two materials were obtained for a MDH mass fraction in the 12-15 % range. These outcomes are illustrated in Fig. 5 that gives the HRRs for the Kyowaglas material and e.g., for the composite sample composed of 12 %wt of MDH. This figure also provides the HRR for PMMA, confirming its strong reduction by both lead (Kyowaglas) and MDH (Kyowaglas surrogate) materials.

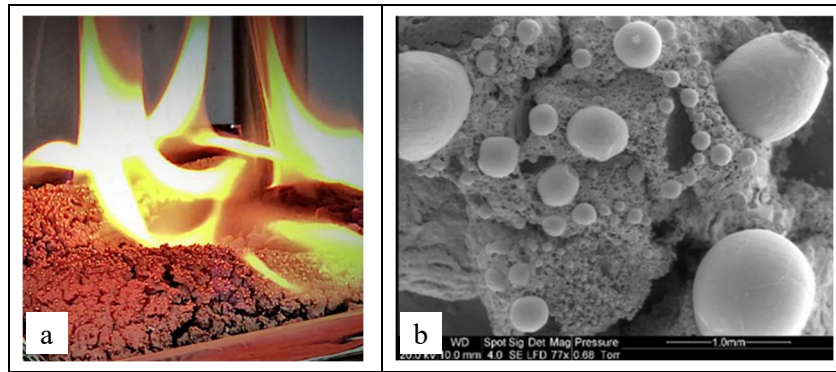


Fig. 4 Kyowaglas combustion tests with the cone calorimeter (a), post-combustion Scanning Electron Microscopy observations of lead beads formed on the surface of the Kyowaglas samples (b).

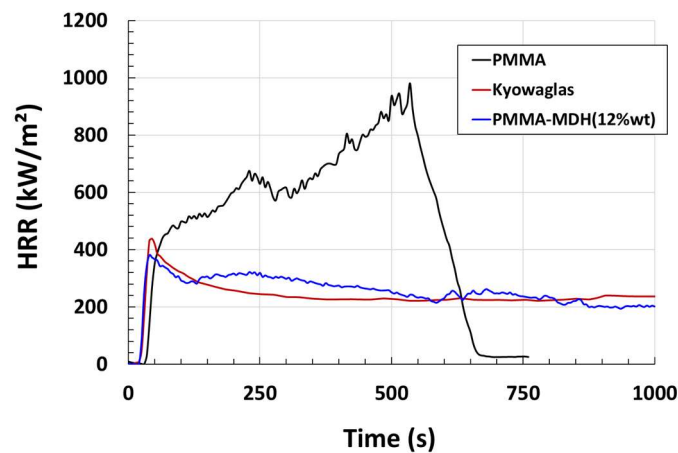


Fig. 5 Heat release rate of PMMA, Kyowaglas and MDH (12 %wt) - PMMA (88 %wt) composite samples under an irradiance of 50 kW/m² in the cone calorimeter. MDH, magnesium hydroxide; PMMA Poly(methyl methacrylate).

4.1.2 Medium-scale fire tests using a glove box simulator

This part deals with the first two medium-scale test campaigns that used the SIMBAG glove box test simulator, for improving the knowledge and modelling of the complex phenomena involved in GB fires.

The goal of the first campaign was to study, in open atmosphere conditions, the impact of the change of thermal and aeraulic conditions on the combustion regimes in the SIMBAG GB simulator. To this end, fourteen medium-scale tests using the SIMBAG device were conducted under the large-scale calorimeter (Fig. 6-a) located in the wide (2000 m³ in volume) and well-ventilated SATURNE facility. The SIMBAG device is composed of 3 parts:

- the analytical GB (Fig. 6-b) with its containment panels,
- the air supply system (Fig. 6-c) for controlling a part of the air inflow rate in the analytical GB,
- the vertical gas burner (Fig. 6-d) that simulates the combustion of the GB protection panel.

The change of thermal and aeraulic conditions in the SIMBAG device was managed by varying the vertical gas burner heat release rate (100 and 300 kW), the reduced-scale GB air inflow rate (0 and 100 m³.h⁻¹), the thickness of the air gap between the SIMBAG containment panel and the gas burner (14 and 30 mm) and the GB configuration. Four GB configurations were investigated all including the vertical gas burner and the PC panel in the front part of the analytical GB and two steel side walls. Otherwise, the rear face of the GB was composed of either a steel containment panel (A config.), a steel containment panel with holes (B config.), a PC containment panel with holes (C config.), as seen in Fig. 6-b, or one PC containment panel and one steel protection panel, both with holes (D config.).

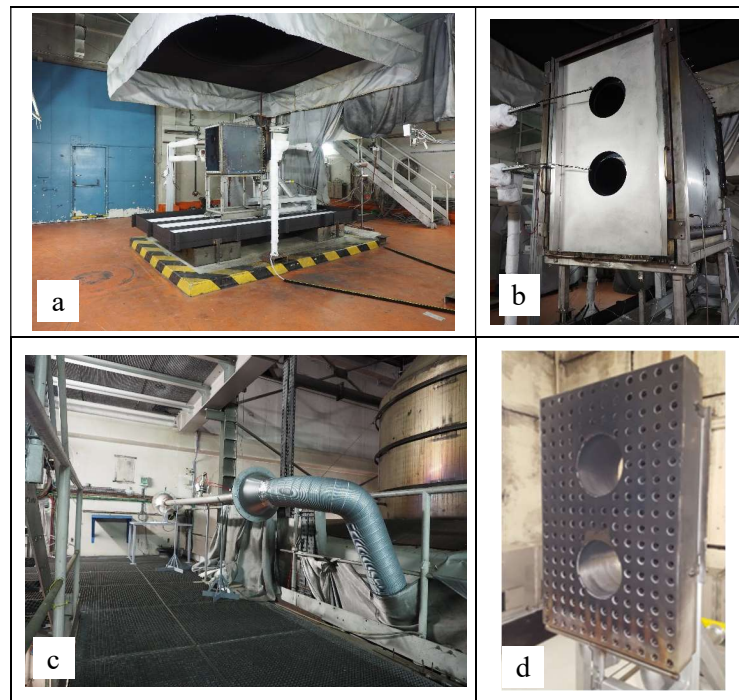


Fig. 6 SIMBAG test device, overall view of the SATURNE facility (a), view from the rear side of the SIMBAG enclosure (b), air supply system (c) and gas burner (d).

The fire tests conducted during this first test campaign, exhibited a fire scenario similar to that observed during the previous GB fires (Coutin and Audouin, 2017). This scenario includes the following steps: incubation stage, growth stage, full-developed fire stage and fire decay stage up to its extinction. Fig. 7-a shows the SIMBAG test device before the fire. After the flame stabilization on the gas burner (Fig. 7-b), the PC front panel starts to degrade. Flames next partially enter in the GB through the glove holes. The collapse of the PC front panel (Fig. 7-c) corresponds to the end of the incubation stage. Accordingly, the gas burner flames enter more widely in the GB, which leads to increase the pyrolysis of melted PC that accumulates on the GB floor. At this stage of the fire development, an increase of the fire power is observed and the heat fluxes in the GB are enough significant to start the degradation of the internal face of the rear PC panel (Fig. 7-d). The latter starts to melt (Fig. 7-e) and finally collapse (Fig. 7-f), which leads to the full development of the fire. The collapse of the PC panels and the consecutive accumulation of melted PC at the bottom of the GB cause a reduction of the exchange surface between fuel and fire. This reduction and that of the available fuel load, caused by its consumption, bring to a decrease of the fire intensity (Fig. 7-g). The burner is stopped when the mass loss rate becomes neglectable (Fig. 7-h) and next the fire stops (Fig. 7-i). This campaign first highlighted that the D configuration was the most favorable for the fire development in the GB. This test campaign also showed that the three other studied parameters (burner heat release rate, GB air inflow rate and air gap thickness) influenced the fire development and sometimes with coupled effects.

The objective of the second test campaign was to study the air vitiation effect on the combustion regimes in the SIMBAG device. Four fire tests were carried out in the confined and mechanically-ventilated room 3 of the DIVA facility, as shown in Fig. 8. Four under-oxygenated conditions were implemented in the vicinity of the SIMBAG device by changing both the inlet and outlet ventilation flow rates in room 3 (2400, 1500, 600 and 300 $\text{m}^3 \cdot \text{h}^{-1}$). The SIMBAG configuration used for this campaign was identical to the glove box configuration used for the preliminary large-scale fire tests (Coutin and Audouin, 2017) and to that of an exploratory SIMBAG test conducted in open atmosphere (SATURNE facility). This configuration included front and rear sides equipped both with protection PMMA and containment PC panels. All the other sides were steel panels. This campaign first showed that the GB fire duration increases when the ventilation flow rate decreases. The tests also revealed that the peaks of the main combustion parameters (fuel mass loss rate and heat release rate) and the maximum average temperature inside SIMBAG, are reduced in confined conditions (DIVA facility) compared to those obtained in open atmosphere conditions (SATURNE facility). However, it is worth noting that the time evolutions of all these parameters are similar for the two highest ventilated tests (2400 and 1500 $\text{m}^3 \cdot \text{h}^{-1}$), suggesting a slight impact of such a ventilation flow rate range on the confined

SIMBAG combustion regime. In addition, the test conducted with the intermediate ventilation flow rate of $600 \text{ m}^3 \cdot \text{h}^{-1}$ exhibited late peaks for all the above parameters that were slightly lower than those observed for the two higher ventilated tests. Otherwise, for the lowest ventilated test ($300 \text{ m}^3 \cdot \text{h}^{-1}$), such a low ventilation flow rate strongly reduced the main combustion properties and temperature in the SIMBAG device.

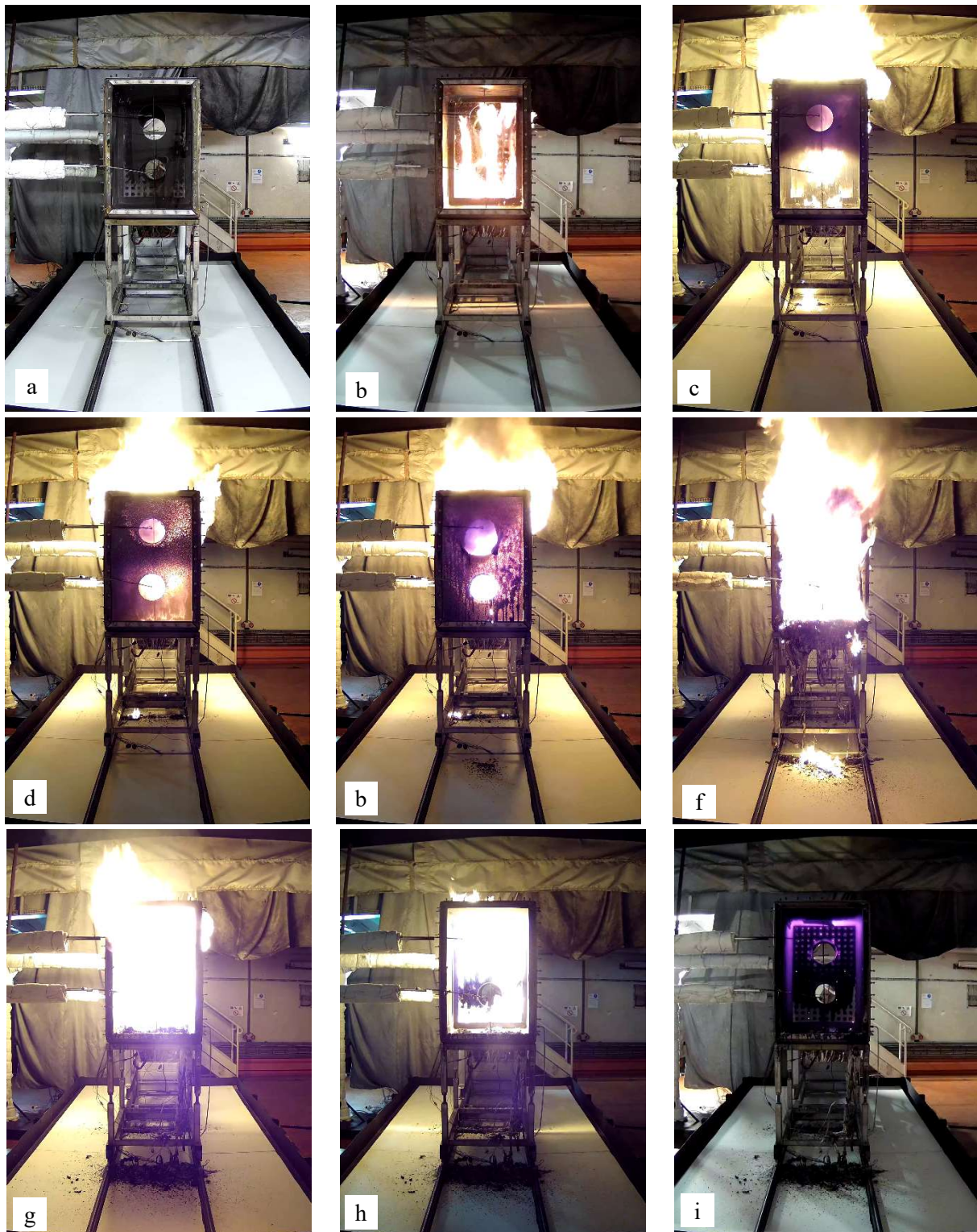


Fig. 7 Characteristic fire scenario observed during the first test campaign using the SIMBAG device, before ignition (a), $t = 40 \text{ s}$ (b), $t = 4 \text{ min}$ (c), $t = 6 \text{ min}$ (d), $t = 8 \text{ min}$ (e), $t = 10 \text{ min}$ (f), $t = 30 \text{ min}$ (g), $t = 60 \text{ min } 40 \text{ s}$ (h) and $t = 62 \text{ min}$ (i, time after the fire extinction).

The last test campaign that aims at studying the impact of the surrogate of Kyowaglas on the SIMBAG combustion regimes, will use the surrogate (PMMA-MDH composite) panels for which the fire behaviour was validated at large-scale, as detailed in section 4.3.

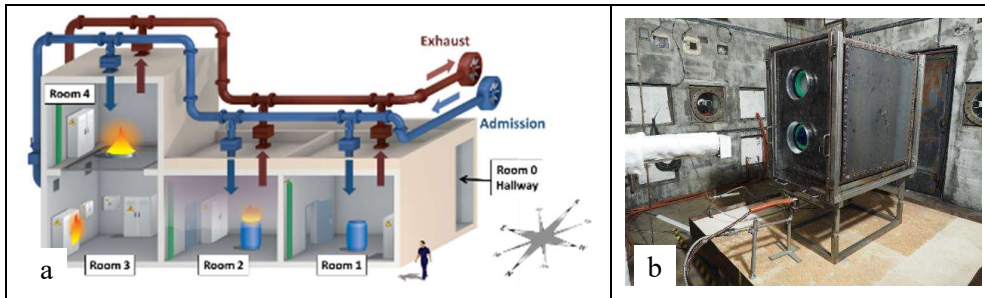


Fig. 8 The multi-rooms and mechanically-ventilated DIVA facility (a) that hosted the SIMBAG glove box fire simulator in room 3 during the second test campaign that investigated the air vitiation effect on the combustion regimes in the GB mock-up (b). Note that the configuration of the rooms shown in Fig. 8-a is only illustrative and does not correspond to that implemented as part of the discussed tests.

4.1.3 Model development and validation

The first step in the development of a mechanistic and global GB fire model in the IRSN SYLVIA software addressed the implementation of horizontal and vertical-wall fire models in this software and their validation. These models are inspired by the cable tray fire model that was previously implemented in the SYLVIA software (Plumecocq et al., 2019). Walls are assumed to be made of a homogeneous combustible material and complex phenomena such as the formation of a bubble layer on the surface of the solid material, the regression of the material thickness under the effect of its pyrolysis or the swelling of the material under the effect of the temperature are not considered. In addition, the aerologic effect on flame spread is not regarded (limitation of zone models) and the soot concentration inside the flame is assumed to be homogeneous over the height of the flame (no local effect of the flame radiation on the fuel surface). Finally, uniform ignition along the width of the wall (1D flame propagation) is assumed.

Based on the Quintiere formulation of the flame spread velocity, V_f (Quintiere, 2002), in open atmosphere, that supposes a thermally-thick behaviour of the material, we considered a time dependence on the ambient gas temperature for confined fires, $T_g(t)$, and a correction factor, $\chi(O_2)$. The latter takes into account the impact of the compartment oxygen depletion on the decrease of the flame radiative heat flux incident to the fuel surface and obtained in well ventilated conditions, \dot{q}_r'' . These considerations give the following equation for V_f :

$$V_f(t) = \frac{4(\dot{q}_r''\chi(O_2))^2 \delta_c}{\pi(k_s \rho_s c_{ps})(T_{ig} - T_g(t))^2} \quad (1)$$

where δ_c is the heated fuel distance (m), $k_s \rho_s c_{ps}$, the thermal inertia of the wall ($W^2.s.m^{-4}.K^{-2}$) and T_{ig} , the ignition temperature of the wall (K). The validation of these models was performed in particular on IRSN bench-scale fire tests involving PMMA slabs (Pizzo et al., 2009).

The next steps of the GB fire model development will focus on the modelling of the PC containment wall relocation and its impact on the radiative heat transfers towards the existing fuel walls, their preheating by the hot gas layer in the GB and finally the modeling of the relocated PC combustion. The efforts devoted to the development of this GB fire model in the SYLVIA software will be next capitalized in the CALIF³S-ISIS software.

4.2 Analytical study of the particle airborne release of PuO₂ (task 2)

This section first discusses the works of task 2 leading to the identification of an appropriate surrogate for PuO₂ particles. The second part addresses the development of a methodology to contaminate the walls of the SIMBAG simulator and those of a GB. The last part describes the specification, implementation and first qualification of the

aerosol metrology that will be used to assess the ARF of the PuO₂ surrogate involved in the GB fire tests carried out in open atmosphere and confined conditions.

4.2.1 Analytical study of PuO₂ surrogates

The major particle properties impacting their ARF during thermal degradation of PC samples were determined by a careful analytical investigation (Ouf et al., 2020). Thirteen powder samples were considered as contaminants and about sixty tests were carried out using the ARTEMIS experimental test bench (Fig. 9).

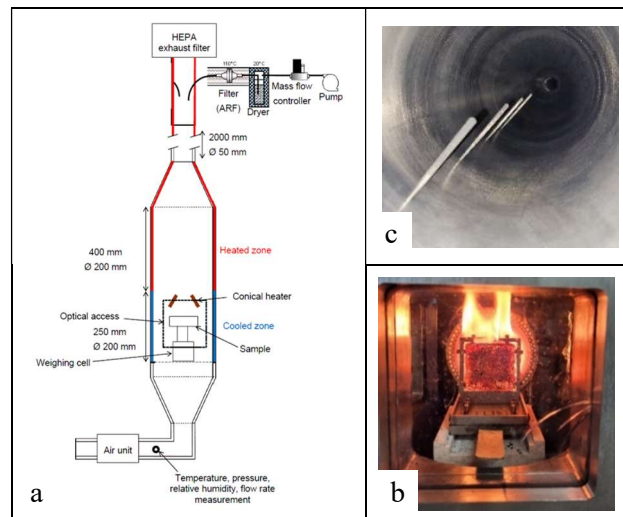


Fig. 9 The ARTEMIS experimental test bench, Overview (a), thermal degradation zone (b), heated zone (c).

A ventilation fan coupled to a critical orifice allows the injection of air at a fixed flow rate (from 40 to 60 m³/h) inside the degradation chamber composed of a radiant conical heater (Fig. 9-b). Particles are then extracted from this chamber inside an exhaust duct heated at 150°C (Fig. 9-c), before being sampled under well-mixed conditions at 2 m from the inlet. The aerosol sampling filters are next analyzed by inductively coupled plasma mass spectrometry (ICP-MS) after each test to determine the mass of released contaminant particles on the sampling membrane. To assess the ARF properly, such mass measurements need to consider the loss of particles within the test bench, as detailed in (Ouf et al., 2020). The latter analytical study highlighted the main significant influence on the ARF of the median geometric diameter⁴ and density of particles. For illustration, Fig. 10 provides e.g., the ARF dependance according to the median geometric diameter for all the studied contaminants powders. (Ouf et al., 2020) suggests that the phenomenology of suspension of particles at the surface of molten PC, can be described according to the balance between attractive forces (capillary force, gravity force...) and repulsive forces (buoyant, lift and drag forces) that apply to particles and that maximal suspension is reached for a particle geometric diameter around 6-8 μm, as shown in Fig. 10. Next, several experimental developments were carried out to identify and characterize the best surrogate of PuO₂ powders according to identified influencing factors (density and particle size). The thermal stability of surrogate powders was also considered since PuO₂ is known to be thermally stable up to 2400 °C. Thus, commercially available

⁴The median geometric diameter D_{geo} can be determined from the mass median aerodynamic diameter (MMAD) and particle density, considering spherical particles (Ouf et al., 2020):

$$D_{geo} = MMAD \cdot \sqrt{\frac{\rho_o}{\rho_p}}$$

where ρ_o and ρ_p are the reference density of 1000 kg/m³ and the particle density, respectively. In addition, the aerodynamic diameter represents the diameter of a sphere with the same settling velocity as the particles considered and a density of 1000 kg/m³ (Ouf et al., 2013). The aerodynamic diameter is useful for characterizing the size distribution of an aerosol but needs to be converted to a geometric diameter to describe the behaviour of a particle deposited on a stable surface and to compute the forces applied to this particle (adhesion, drag, lift, etc.) (Ouf et al., 2013). The particle geometric diameter (or equivalent volume diameter) can indeed directly influence the interactions between the particles and the PC surface during its thermal degradation, as detailed in (Ouf et al., 2020) and summarized in the current work.

hafnium dioxide (HfO_2) powder was selected among several potential surrogate candidates, since its density (9680 kg/m^3) is close to that of PuO_2 ($11,460 \text{ kg/m}^3$), and its thermal stability was proven up to $1200 \text{ }^\circ\text{C}$ which is the maximum temperature measured in the GB during the near-complete combustion regime (Coutin and Audouin, 2017). The last effort in HfO_2 powder characterization was to control the appropriate size dispersion and morphology. To this end, a process was specified for conditioning the particles to reach aerodynamic diameters which, on the one hand, must be compatible with the geometric diameters leading to significant ARFs (Fig. 10), on the other hand, must not too strongly limit their penetration from the GB up to the sampling devices located at the exhaust of the large-scale test facilities.

Experiments will be conducted with the conditioned HfO_2 powder using the ARTEMIS test bench to confirm the experimental conditions corresponding to significant ARFs. These insights will support the specification of the final GB tests that will be performed for assessing the ARF of the HfO_2 particles under real fire conditions.

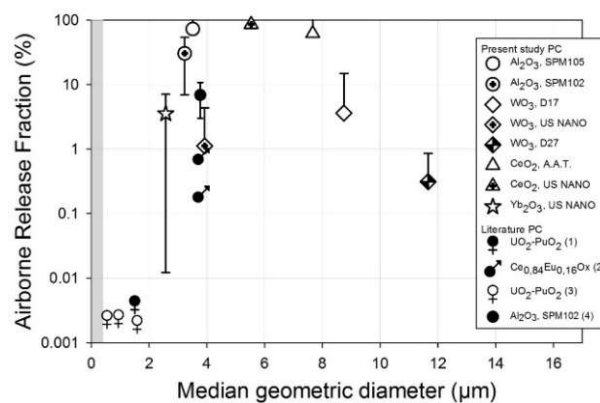


Fig. 10 Airborne release fraction as a function of the median geometric diameter (Ouf et al., 2020) ((1) (Buijs et al., 1989); (2) (Buijs et al., 1992); (3) (Pickering, 1989); (4) (Ouf et al., 2013)).

4.2.2 Preliminary study of the airborne release of PuO_2 using the medium scale glove box SIMBAG

Before conducting the experimental program, as described in section 3.2.2, an important preliminary stage was performed with the development of a methodology to control the contamination of the panels with suitably conditioned HfO_2 powder, using a specific powder disperser device, called MORDORS. This methodology will be used first for the SIMBAG tests planned in this part and next for all the subsequent tests that will use contaminated GBs.

4.2.3 Development and validation of the aerosol metrology for assessing the ARF at large-scale

The GB fire tests that will involve the PuO_2 surrogate particles will be first conducted in open atmosphere conditions, in the SATURNE test facility (Fig. 11-a). A sampling line, called SALSA_S (SAmpling Line for Surrogates Aerosol for Saturne) was developed and connected at the exhaust ventilation duct of the SATURNE facility (Fig. 11-b). The SALSA_S device is equipped with electrostatic precipitators (ESPs) (Fig. 11-c) that aim at especially collecting the PuO_2 surrogate particles while avoiding the risks of clogging and rupture caused by the large soot release that can be expected in fire experiments (Tasaka et al., 2019). The specifications of the SALSA_S sampling device was supported by using the aerosol transport and deposition model (Nerisson et al., 2011) with the ANSYS CFX computer code (Gélain et al., 2018). In addition, an experimental methodology was also developed to extract the HfO_2 particles collected on the metallic grids of the ESPs, by using an ultrasonic bath in which the grids will be immersed after the GB fire tests. Finally, the satisfactory collection efficiency of ESPs with respect to hafnium oxide was experimentally validated under the operating conditions of the SALSA_S sampling line (without the presence of soot). The following step consisted in verifying this good performance in the presence of soot produced by hydrocarbon fires with a controlled dispersion of HfO_2 particles. Note that these tests, like all the subsequent GB fire tests, also implemented a controlled injection of aluminium oxide powder (Al_2O_3) used as a particulate tracer of HfO_2 powder (the two powders must have the same aerodynamic diameters). This is crucial, as previously mentioned, to assess the mass of released

particles from that determined on the ESPs and corrected for losses of particles between their release location and their collection on ESPs.

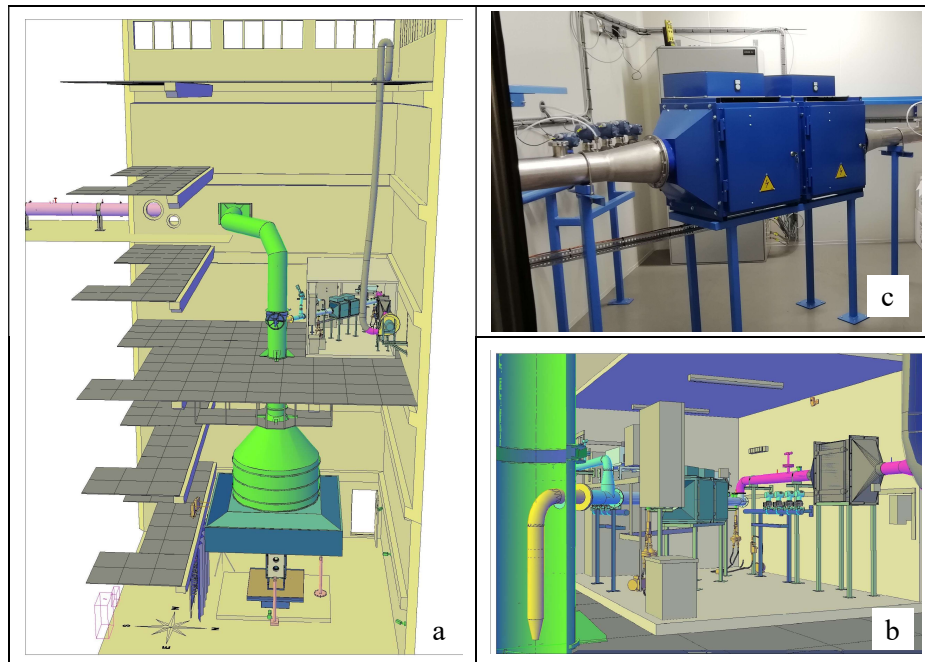


Fig. 11 SATURNE facility with the large-scale hood connected to the exhaust ventilation duct (a), SALSAS sampling line connected to the exhaust ventilation duct (b), enlarged view of the electrostatic precipitators of the SALSAS line (c).

The GB fire tests that will involve the PuO_2 surrogate particles will be next conducted in confined and mechanically-ventilated conditions, in the PLUTON test facility (Fig. 12). Accordingly, a second sampling line, called SALSAS_P (SAmpling Line for Surrogates Aerosol for Pluton), identical to the first SALSAS_S sampling device, was connected to the exhaust ventilation network of the PLUTON facility.



Fig. 12 The 400 m³ volume of the confined and mechanically-ventilated PLUTON facility.

4.3 GB fire tests involving the Kyowaglas surrogate (task 3)

This section discusses the GB fire tests that targeted to validate the fire behaviour of the GB equipped with the Kyowaglas surrogate panels. Significant preliminary efforts focused on the manufacturing of the Kyowaglas surrogate panels for the GBs. Given the challenging fabrication of homogeneous PMMA-MDH composite panels about 1 m side and 5 cm thick, two manufacturing processes by extrusion and casting were considered. However, only the PMMA-MDH panels made by casting provide full satisfaction in terms of the MDH mass content and its dispersion (13.5 ± 1.5 %wt) and the panel geometry (uniform width of 5 cm and an excellent surface condition, as seen on Fig. 13-a). The first GB fire test with these panels showed that the HRR was slightly lower than that measured for the

test conducted at SP Fire Research, which used the same GB but equipped with Kyowaglas protection panels. Accordingly, additional composite panels were supplied by casting with a lower MDH content of 11 %wt and a last test was conducted with such panels (Fig. 13-b). This test and that carried out with the Kyowaglas panels give identical HRR peaks, as seen on Fig. 14. This figure also highlights that the incubation stages⁵ of the tests using the panels of kyowaglas ($t_{01} \sim 9$ min) and of its surrogate ($t_{02} \sim 19$ min) are shifted of about 10 min and that the two HRR time evolutions around the fire peak are very close. Thermal degradation of the PC containment panels, that could promote the airborne release of the deposited⁶ particles (Ouf et al., 2020), is supposed starting after the incubation stage, during the fire growth stage and to continue beyond the fire peak (Coutin and Audouin, 2017). Accordingly, the validation of the fire behaviour of a GB fitted with Kyowaglas surrogate panels, based on similar HRR time evolutions around the fire peak rather than equivalent incubation stage durations, appears more relevant to obtain representative ARF values of those that would be obtained during a fire of a contaminated GB equipped with Kyowaglas panels.

Fig. 14 also gives the HRR for the GB fire test that involved the PMMA panels (Coutin and Audouin, 2017). The whole outcomes shown in this figure for HRR confirm its significant reduction at large-scale by the presence of lead (Kyowaglas) and MDH (Kyowaglas surrogate). These results therefore show that the PMMA-MDH(11 %wt) composite panel made by casting is the most appropriate Kyowaglas surrogate panel that will equip the GBs for the last fire tests which aim at providing realistic assessments of the ARF of the PuO₂ surrogate (section 3.3.2).



Fig. 13 Glove box fire tests with the kyowaglas surrogate panels. View from the front side of the glove box before the fire (a). View from the rear side of the glove box during the fire (b).

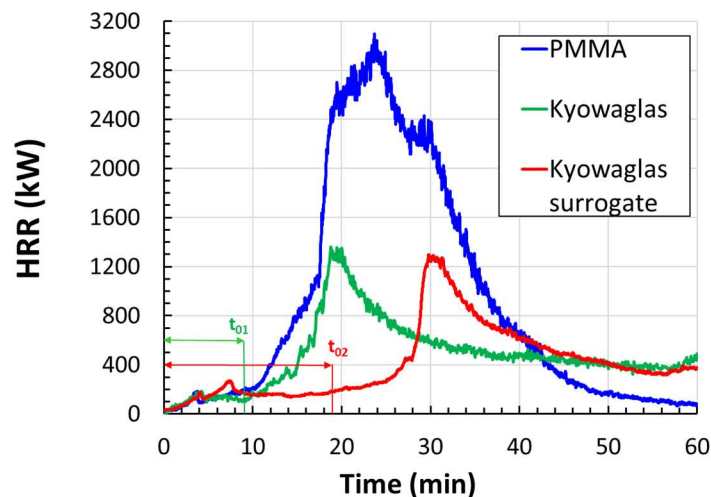


Fig. 14 Heat release rate (HRR) obtained during the GB fire tests using the panels of either PMMA, kyowaglas or kyowaglas surrogate. t_{01} (~ 9 min) and t_{02} (~ 19 min) are the incubation stage durations of the GB fire tests using the panels of kyowaglas and of its surrogate, respectively.

⁵The incubation phase corresponds to the first fire stage for which the fire power is quite low and steady, before it increases continuously up to the fire peak during the growth stage (Zavaleta et al., 2019).

⁶The PC containment panels will be contaminated with the PuO₂ surrogate particles (i.e., HfO₂ particles) as part of the tests that will aim at assessing the ARF of these particles (see sections 3.2.2 and 3.3.2).

5. Conclusion

The first two complementary tasks of the FIGARO program that have studied separately the various mechanisms involved in the GB fires (task 1) and the PuO₂ airborne release (task 2) are completed. First, the surrogates of Kyowaglas and plutonium dioxide were identified based on in-depth analytical investigations to obtain similar fire behaviour between Kyowaglas and its surrogate and similar properties in terms of ARF between plutonium dioxide and its substitute. In addition, the analytical studies conducted with the medium-scale SIMBAG simulator provided valuable insights for improving knowledge of the complex and unexpected combustion regimes that were observed during the preliminary GB fire tests. The outcomes of these tests, complemented by those of the study dealing with the effects of under-oxygenated conditions on the combustion of GB materials (PMMA...) in the CADUCEE controlled-atmosphere device, allow to support the development and validation of models such as the glove box fire model developed in this project. The transposition and validation of the analytical works, as desired in the third and last task of this project, was achieved for the Kyowaglas surrogate, which exhibited fire behaviour very similar to Kyowaglas at both small and large-scale. The last step will regard the large-scale ARF assessments of the PuO₂ surrogate for realistic GB fire conditions. To this end, the suitable aerosol metrology was implemented in the large-scale test facilities, and the qualification tests of this metrology showed satisfactory outcomes.

Acknowledgments

The authors are very grateful to the Nuclear Regulation Authority (NRA) of Japan for its partnership in the FIGARO project. The authors also express their gratitude to P. Wiszniowski from the IRSN aerosol physics and metrology laboratory, S. Pons, G. Basso, V. Cozar and the other members of the technical team of the IRSN fire experimentation laboratory, for their assistance in the development and implementation of the aerosol metrology and the achievement of the experiments.

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