

**A FIRST ORIENTING INVESTIGATION OF THE INTERACTION OF
CABLE FIRE PRODUCTS WITH PASSIVE AUTO-CATALYTIC
RECOMBINERS (PARs)**

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ABSTRACT

Passive auto-catalytic recombiners (PARs) have been installed inside LWR containments in many countries in order to remove hydrogen and thus to mitigate the combustion risk during a severe accident (SA). Due to the challenging SA boundary conditions, PARs are exposed to several deactivation risks during operation which may cause a reduction of the hydrogen removal capacity. Such a deactivation may occur through different mechanisms and could in principle affect the start-up behavior up to the full loss of catalytic activity.

In order to assess the interaction of PARs with products of cable fires, a set of PAR catalyst samples has been introduced to the atmosphere of cable fire tests performed at IRSN (France). The subsequent surface analyses performed at JÜLICH (Germany) reveal significant amount of carbon, chlorine (constituent of PVC), zinc, and antimony (flame retardant) on all catalyst samples compared to reference samples. The subsequent performance tests confirm that all catalyst sheets suffer a significant start-up delay between 17 min and 45 min compared to the reference samples. However, after burning off the soot deposition, the catalyst samples reach full conversion capacity and show immediate start-up behaviour in a subsequent test.

The present results clearly demonstrate the adverse effect of cable fire products on the efficiency of hydrogen conversion in a PAR. In order to further understand and quantify the impact of cable fire products and to assess their relevance for severe accident scenarios, further experimental as well as theoretical investigations are required. Especially, the combined influence of cable fire products and humidity which has been intentionally omitted in the present study, should be investigated in the future due to the possible corrosive impact on the catalyst as well as the influence of humidity on the nature of the soot deposition.

KEYWORDS

Hydrogen, passive auto-catalytic recombiner (PAR), cable fire, severe accident

1. INTRODUCTION

During a severe accident in a light water reactor, large amounts of combustible gases such as hydrogen or carbon monoxide are produced due to various chemical processes. While hydrogen is mainly generated in the zircaloy-steam reaction during heating-up of the fuel elements, a major source for carbon monoxide is the molten core – concrete interaction (MCCI) in the late phase of the accident [1]. The explosive mixture of these gases with air inside the containment may threaten the containment integrity and lead to a loss of the last barrier against contamination of the environment.

Passive auto-catalytic recombiners (PARs) are a key element of the mitigation strategy against combustible gas mixtures in many nuclear power plants worldwide [2]. PARs convert hydrogen into steam and carbon monoxide – under favourable conditions [3] – into carbon dioxide by means of a controlled surface reaction on catalyst sheets. After the Fukushima accident in 2011, the interest in backfitting containments with PARs has increased significantly.

Due to the challenging atmospheric boundary conditions during a severe accident (e.g. presence of high humidity and various species of aerosols), PARs are exposed to several deactivation risks during operation which may cause a reduction of the hydrogen removal efficiency. Such a deactivation may affect the start-up behaviour and lead to partial or even full loss of the catalytic activity [4,5]. In the year 2011, the event of a plastic fire in the NPP Ringhals/Sweden [6] revealed a strong interaction of the fire products with the catalyst sheets of PARs installed inside the containment. In addition to a severe

contamination of the catalyst sheets with soot, significant corrosion effects were detected. These observations emphasise the necessity to study the impact of severe accident-relevant fire products on the catalytic activity of PARs.

In order to further justify related research, a set of catalyst samples was introduced to the atmosphere of cable fire tests performed at IRSN/Cadarache. After exposure to the fire products, the samples were transferred to Jülich Research Centre (JÜLICH) in order to investigate:

- the nature of depositions of cable fire products on the surface (‘surface analyses’), and
- the impact of the depositions on the start-up behaviour and on the hydrogen conversion efficiency of the catalyst samples (‘performance tests’).

2. CABLE FIRE TESTS (DIVA FACILITY)

2.1. Facility Description and Sample Preparation

The DIVA facility is a large-scale multi-room facility including four compartments and a corridor [7]. The configuration used for the present investigation consists of two rooms with a volume of 120 m³ each (floor area 5 m x 6 m, height 4 m, see Fig. 1). Both rooms are interconnected via a door opening allowing gas exchange in both directions. The ventilation system of the facility has an inlet opening in Room 1 and an outlet opening in Room 2. Room 1 contains a vertical set-up of 5 cable trays which have been set on fire in two subsequent tests. In the first test (CFS-1), 245 halogenated power cables with a total length of approx. 588 m have been used. The second test (CFS-2) included 220 halogenated control cables with a total length of approx. 528 m.

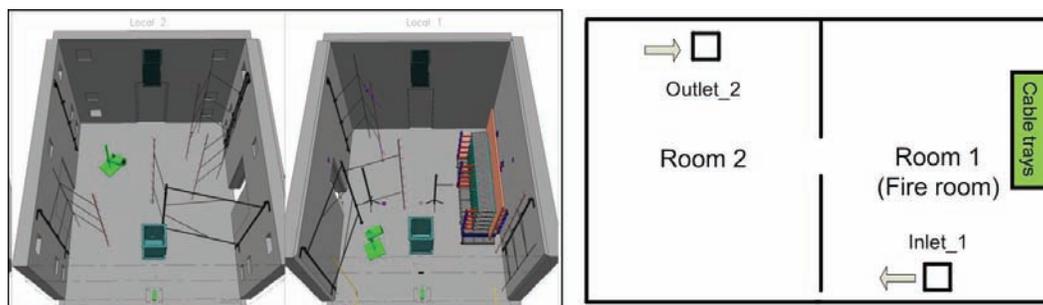


Figure 1. DIVA facility, top view.

A set of catalyst sample sheets with a size of 5 x 5 cm² has been introduced to both DIVA tests. In total, four types of catalysts obtained from different sources including commercial and research catalysts were used. Due to the limited number of catalyst samples of some of these types, only samples of types 2 and 3 are available for both cable fire tests (Fig. 2). For legal reasons, only the results achieved with samples of type 2 can be published in the present paper. The catalyst samples of type 2 are composed of a ceramic washcoat with platinum catalyst on a stainless steel carrier sheet. This catalyst, manufactured by company CCD in Aachen/Germany, is representative of a typical catalyst used in commercial PARs.

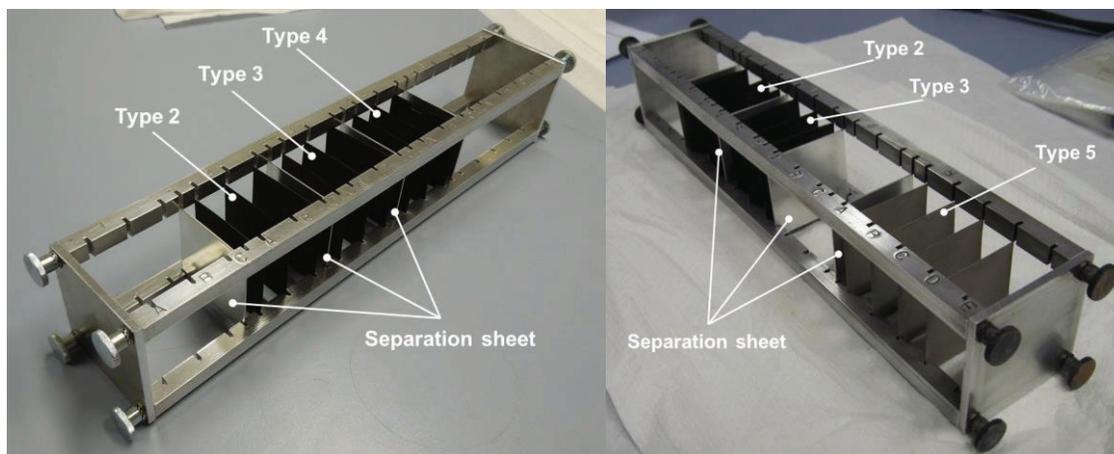


Figure 2. Sample holder equipped with the catalyst sheets (left: CFS-1, right: CFS-2).

The catalyst sheets are arranged inside a sample holder in order to expose all samples in an equal manner to the test atmosphere. In order to avoid interaction of the different catalyst types, samples of the same type are placed in separate sections. The sample holder is installed inside the DIVA facility in the ‘southeast corner’ at an elevation of approximately 3 m (Fig. 3). A single thermocouple is positioned above the holder in order to detect possible temperature increase due to exothermal reactions on the catalyst sheets.

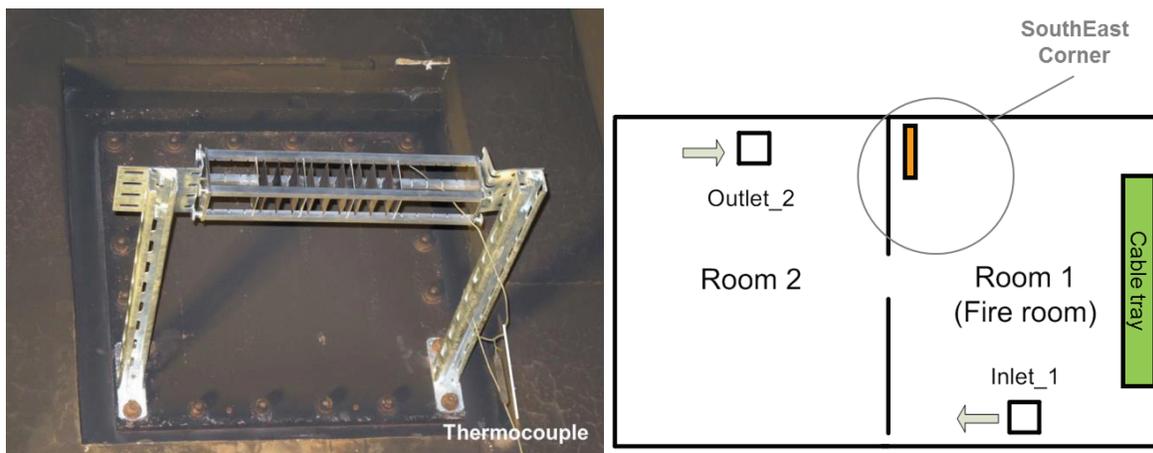


Figure 3. Sample holder inside the DIVA facility (CFS-1).

2.2. Experimental results

The DIVA cable fire tests are initiated by setting the cable trays on fire by means of a propane burner. For the duration of the tests (approx. 1 h), the fire products are distributed inside the atmosphere of both rooms. The resulting temperature loading of the catalyst samples during both tests is given in Fig. 4. The diagrams show the gas temperature evolution measured at different elevations in the ‘southeast corner’. The respective elevations of the measurement positions are given in the legend in the unit of cm. The gas temperature measured above the catalyst sheets (‘TG_Coupons’) is given as the red curve. The uniform progression of this value being slightly higher than the temperature measured at 305 cm elevation reveals no evidence of a significant catalytic reaction during the test.

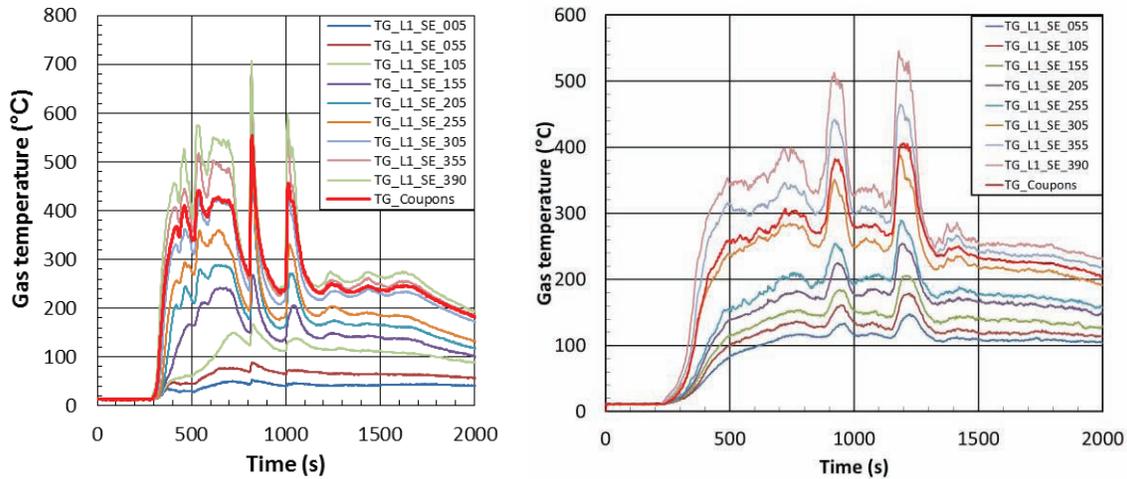


Figure 4. Gas temperature evolution during both cable fire tests (left: CFS-1, right: CFS-2).

The catalyst samples are exposed to a maximum temperature of approx. 400°C for a duration of approx. 500 s in CFS-1 and to approx. 280 °C for a duration of 800 s in CFS-2. In both tests, two reignitions of unburnt gases (e.g. pyrolysis gases accumulated under the ceiling) occurred leading to short-term significantly higher thermal loads.

Fig. 5 (left) shows gas concentrations of carbon monoxide (CO) and carbon dioxide (CO₂) measured in the vicinity of the catalyst samples at an elevation of 3.3 m. The CO concentration reaches values of up to 4 vol.% in CFS-1 and merely 2 vol.% in CFS-2, although here a short peak of 3.3 vol.% is reached. Conventional Pt- and Pd-based catalysts used for hydrogen recombination are known to be very sensitive to CO poisoning [8]. Fig. 5 (right) shows the time evolutions of hydrogen chloride (HCl) concentration obtained by Fourier Transform Infrared Spectroscopy (FTIR) implemented at the outlet of Room 2 in both tests. These measurements reveal maximum concentration values between 0.5 vol.% (CFS-1) and 0.7 vol.% (CFS-2). Maximum HCl concentrations in Room 1 (fire room) can be expected to be significantly higher than in the adjacent room (Room 2). HCl could be the source for corrosive effects on the catalyst sheets.

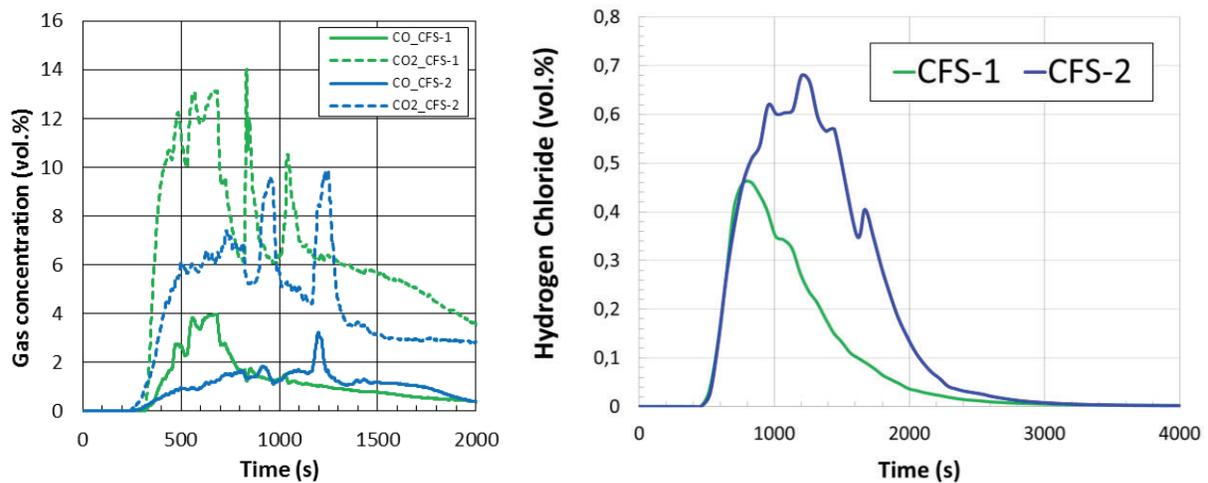


Figure 5. Gas concentration evolution at the catalyst holder during both cable fire tests (left: carbon monoxide and carbon dioxide, right: hydrogen chloride).

After both cable fire tests, the removal of the sample holder from the fire room reveals a significant coverage of both catalyst sheets and sample holder with soot (Fig. 6). Despite the gas combustion occurring during the tests, all catalyst sheets are still in place. No thermal damage of the sample holder made of aluminium is observed.

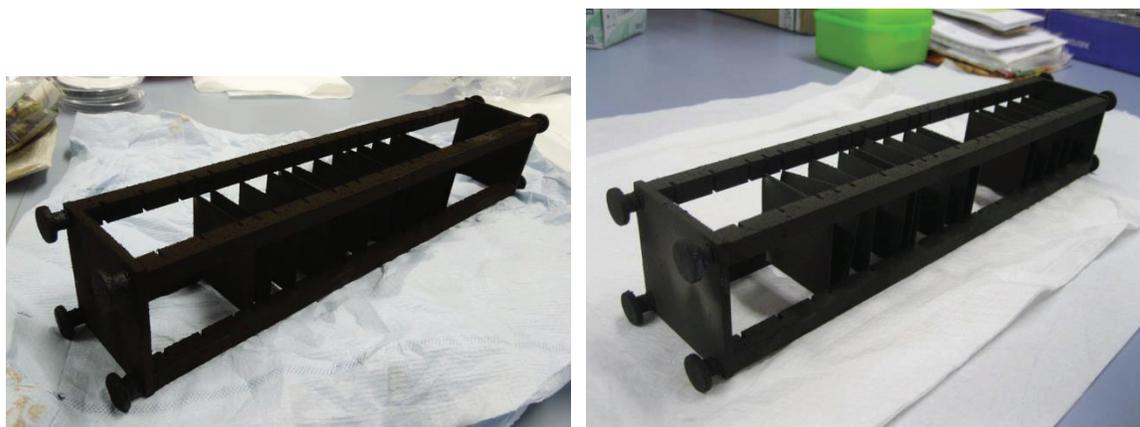


Figure 6. Sample holder covered with soot after tests (left: CFS-1, right: CFS-2).

The catalyst samples are carefully removed from the holder, individually wrapped in parchment paper and stored inside plastic bags for transportation to JÜLICH. For the subsequent investigation, the samples are assigned to surface analyses and functional tests. If available, reference samples of the different catalyst types ('R') are added to the selection.

3. SURFACE ANALYSES

The goal of the surface analyses is to characterise the deposition on the catalyst sheets. For this purpose, the following analyses are performed:

- identification of catalyst damages (e.g. corrosion): SEM*
- semi-quantitative element screening: XRF
- quantification of inorganic deposition: IC , ICP-AES

The SEM microsections which are produced for the catalyst samples used in test CFS-1 reveal no corrosion effect on the steel substrate. Due to the absence of steam in the investigation, this result has been expected. One representative catalyst sample from each cable fire test is used to perform a semi-quantitative element screening by means of X-ray Fluorescence Spectroscopy (XRF) in order to identify those elements on the catalyst surface, whose presence had significantly changed. Catalyst samples from both tests show significant higher amount of chlorine (Cl, constituent of PVC), and zinc (Zn) as well as a new amount of antimony (Sb, flame retardant), see Tab. I. In addition, the catalyst sheet used in test CFS-2 shows a significantly higher amount of lead (Pb, possibly flame retardant). A significant carbon signal is also obtained from both samples used in the cable fire tests.

* SEM: Scanning Electron Microscopy, XRF: X-ray Fluorescence Spectroscopy, IC: Ion Chromatography, ICP-AES: Inductively Coupled Plasma Atomic Emission Spectroscopy

Table I. Results of XRF analyses (proprietary information obliterated with ‘’)**

kilo counts per second peak intensity	CFS-1				CFS-2			
	Reference		Contaminated		Reference		Contaminated	
	front	back	front	back	front	back	front	back
Elements								
*	78,165	77,217	65,908	65,405	56,874	57,139	46,103	53,405
*	1,524	1,782	1,040	1,130	0,043	0,033	0,040	0,041
S	0,144	0,144	0,099	0,088	0,092	0,100	0,096	0,094
Cl	0,016	0,019	1,545	1,584	0,035	0,030	2,273	1,980
K	0,027	0,027	0,026	0,014	0,019	0,007	0,017	0,009
*	0,032	0,039	0,029	0,026	0,025	0,024	0,023	0,027
*	4,537	5,227	4,054	4,165	4,482	4,177	3,604	5,251
*	0,085	0,069	0,064	0,056	0,036	0,025	0,025	0,072
*	9,769	10,856	8,929	9,117	9,418	9,669	8,680	11,794
Zn	0,030	0,029	0,747	1,032	0,024	0,023	0,527	0,443
*					2,254	2,365	2,258	1,510
*	0,511	0,446	0,513	0,449	0,495	0,525	0,478	0,352
*	0,570	0,656	0,526	0,537	0,552	0,576	0,518	0,448
*	0,132	0,133	0,181	0,183	0,112	0,156	0,105	0,115
*	4,869	4,668	5,513	5,381	5,737	5,902	5,675	3,958
Pb							0,283	0,225
Sb			0,097	0,123			0,121	0,157

The elements found on the catalyst surfaces are quantified by means of ion chromatography (IC, for Cl), Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES, for Sb, Zn, Pb), and total carbon assay (for C). The results (Tab. II) reveal a significant amount of carbon (C) between 6.3 and 9.5 wt-%. Here, the reference sample shows already a relatively high contamination of 2.3 wt-%. The amount of antimony (Sb) on the samples from both tests are of similar value (0.51 – 0.59 wt-%). The amount of Zinc (Zn) on the sample from CFS-1 is about twice as high compared to the sample from CFS-2, while lead (Pb) is only present on the CFS-2 sample. The amount of chlorine (Cl) shows values between 1.18 and 1.40 wt-%.

Table II. Results of quantitative surface analyses

wt-%		CFS-1	CFS-2
Element	Ref.	Cont.	Cont.
Cl	0,04	1,18	1,40
Zn	< 0,01	0,96	0,51
Pb			0,35
Sb	< 0,01	0,59	0,51
C	2,27	9,46	6,30

The in-house soot qualification aiming at determining the ratio of organic (OC) and elementary carbon (EC) couldn't be performed because the catalyst material causes adverse side effects (CO oxidation) which interfere with the detection method. Further analyses of the OC and EC composition would be quite useful in future investigations.

4. PERFORMANCE TESTS (REKO-1 FACILITY)

The goal of the performance tests is to identify possible impact of the soot deposition on the start-up behaviour and conversion efficiency of the catalyst samples. In order to avoid overlapping effects of soot and humidity related start-up delay, the tests are performed in dry atmosphere. In order to obtain a maximum effect, challenging conditions (low hydrogen concentration, low temperature, and low flow rate) are selected.

4.1. Facility Description and Sample Preparation

The REKO-1 test facility [9] consists of a vertical flow tube with a diameter of 70 mm (Fig. 7, left). The gaseous mixture of hydrogen and air is fed at the bottom end of the tube at a well-defined flow rate by means of mass flow controllers. The gaseous mixture which may be preheated passes through the catalyst section containing single or several catalyst samples leaving the tube at the top end. Part of the outlet gas is fed into a gas analyser in order to measure the hydrogen concentration. The catalyst temperature is measured by means of pyrometry through a glass window.

During test preparation, the catalyst samples are unpacked and separately mounted in a catalyst sheet holder (Fig. 7, right top). The holder is placed inside the flow tube right in front of the glass window (Fig. 7, right bottom) in order to allow for optical measurement of the catalyst temperature. As the surface emissivity of the different catalyst types is unknown, the corresponding emissivity parameter of the pyrometer is set to 100 %. Hence, the catalyst temperature measurement is only qualitative.

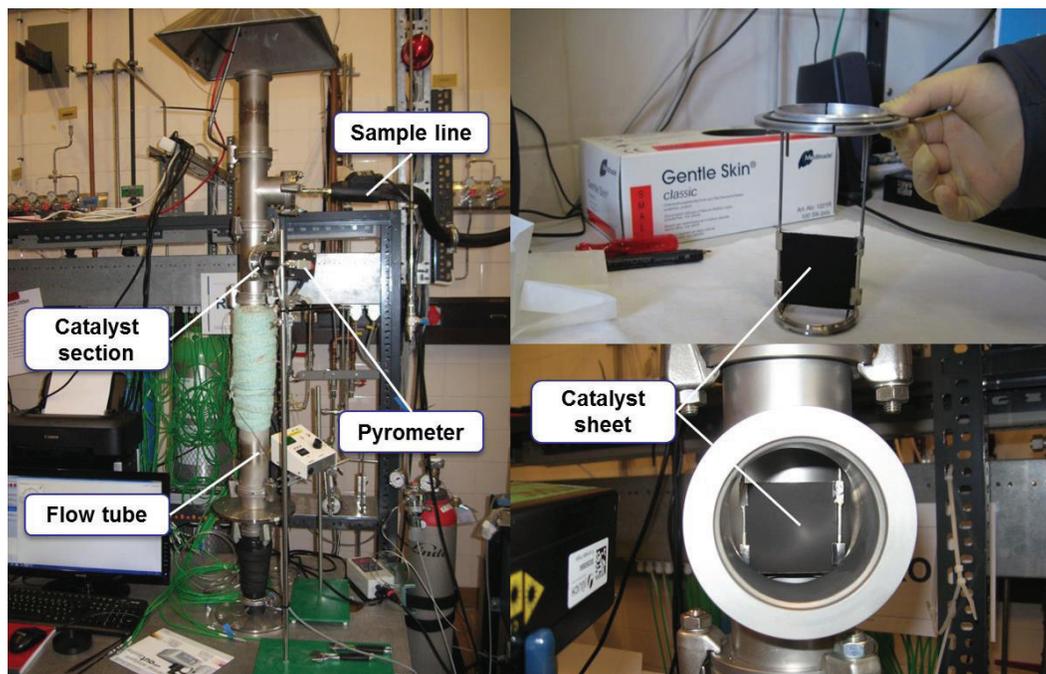


Figure 7. REKO-1 test facility.

During the performance tests with the samples from cable fire test CFS-2, the weight of the samples is determined before and after the test. By doing this, the amount of soot removed (“burned-off”) during operation is determined. The measurement yields approx. 9.5 mg of soot for samples located at the sectional wall sheets of the sample holder and approx. 13.1 mg of soot for the remaining samples.

4.2. Test Procedure

After mounting the catalyst sample into the REKO-1 test tube, a test procedure consisting of two phases is applied. In phase one, the start-up behaviour of the contaminated sample is investigated. In the second phase, the start-up behaviour of the catalyst sample after burn-off of the deposits is investigated. The data from the performance test with reference sample '2R' is given in Fig. 8.

The entire test is performed at a flow velocity of 0.1 m/s and 20°C inlet temperature. It starts with injection of a mixture of 1.0 vol.% hydrogen in air (light blue, 'Hydrogen inlet') at 110 s. The measurement of the gas concentration above the catalyst sample shows the same value (dark blue, 'Hydrogen outlet'). Due to the length of the gas sampling line, the concentration signal has a time lag of approx. 45 s. The catalyst temperature remains below the measurement threshold value of 75 °C (red, 'Temperature catalyst'). Consequently, no start of the reaction is observed for a time period of 15 min. After that time, the inlet hydrogen concentration is increased to 1.5 vol.% (1020 s). Again, no indication for any catalytic reaction is visible. After another 15 min, the inlet concentration is increased to the maximum value of 2.0 vol.% (1920 s). As there is still no reaction observed after further 15 min, the gas pre-heater is started (2880 s). The light green curve represents the gas temperature at the tube inlet while the dark green curve represents the gas temperature approx. 5 cm below the catalyst sheet. As the gas temperature reaches approx. 29 °C, the reaction starts. The start of the reaction is indicated by a steep increase of the catalyst temperature and by a simultaneous decrease of the hydrogen outlet concentration. After the start of the reaction, the gas pre-heater is switched off causing the gas temperature as well as the catalyst temperature to decrease. After the reaction has proven to run stable, the hydrogen flow is stopped and the catalyst sample cools down (4120 s).

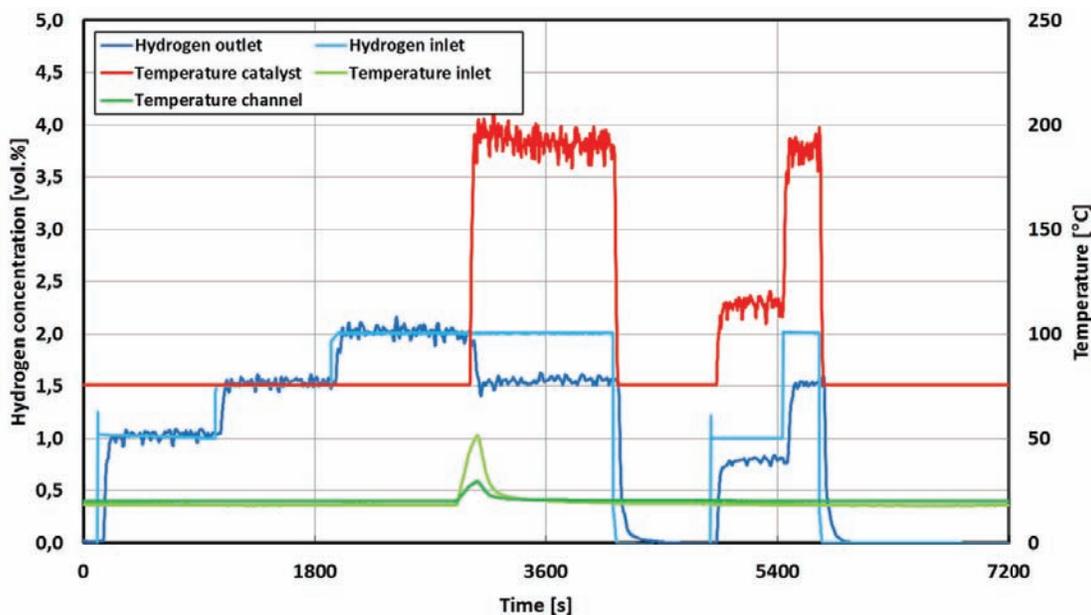


Figure 8. Performance test with reference sample (Type 2).

The second test phase principally follows the same procedure as the previous phase 1. Again, the gas injection starts with an inlet hydrogen concentration of 1.0 vol.% in air at a constant flow velocity of 0.1 m/s (4880 s). This time, the catalytic reaction starts immediately after hydrogen injection, as indicated by

the catalyst temperature and the outlet concentration. Afterwards, the inlet concentration is directly increased to the maximum value of 2 vol.% (5440 s). Outlet hydrogen concentration and the catalyst temperature show approximately the same values as observed during the first test phase.

The catalyst samples contaminated with soot during the cable fire tests show a different performance, as demonstrated here with sample '2C' from test CFS-1 (Fig. 9). Compared to the reference sample, the sample needs more pre-heating until the reaction starts (here approx. 50 °C). After start-up of the catalytic reaction (3490 s), the maximum conversion is not reached immediately. It takes approximately 1000 s until the steady-state conversion rate is reached. After cooling down, the performance in the second test phase is comparable to the reference sample.

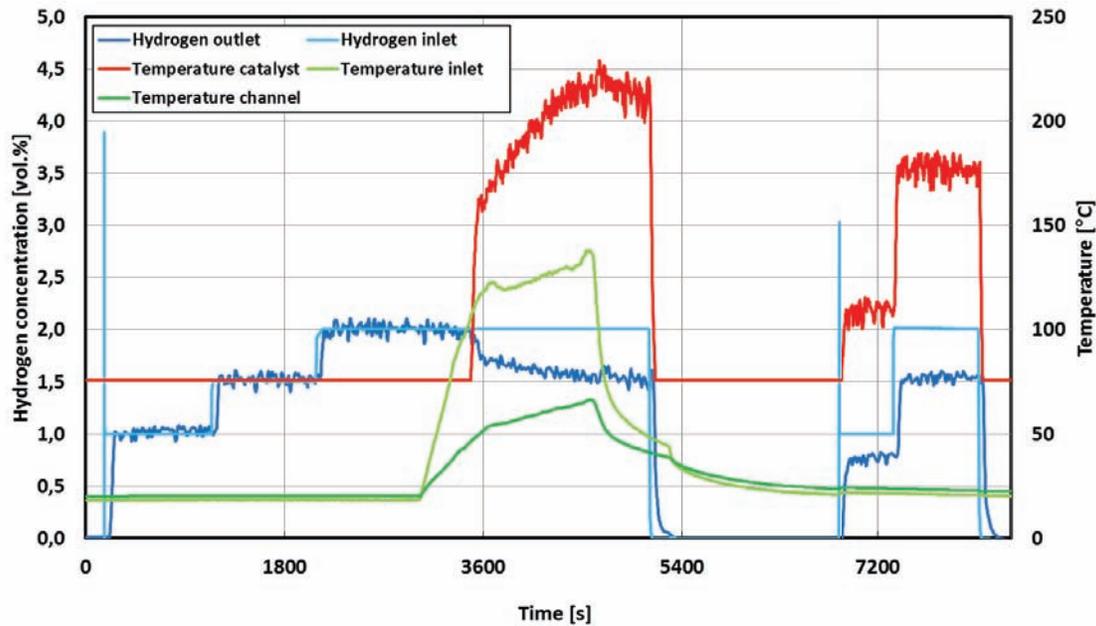


Figure 9. Performance test with contaminated sample (Type 2).

Performance tests were conducted with all available catalyst samples according to the test procedure described above. In order to quantify the influence of the cable fire products on the catalyst performance, two key indicators are considered (Fig. 10):

- The delay time between the start of the catalytic reaction (indicated by the catalyst temperature exceeding the measurement threshold value of 75 °C) and full catalytic operation (indicated by the outlet hydrogen concentration reaching the minimum steady-state value)
- The final conversion efficiency (after deposition burn-off) compared to the conversion capacity of the reference samples

For the determination of the delay time value, the time lag of the hydrogen concentration signal due to the length of the gas sampling line needs to be taken into account. The time lag is determined for each test separately and amounts to values between 43 s and 49 s. The conversion efficiency η is calculated with the measured values for the inlet and outlet hydrogen concentration (y_{in} and y_{out}) according to

$$\eta = \frac{y_{in} - y_{out}}{y_{in}} \quad (1)$$

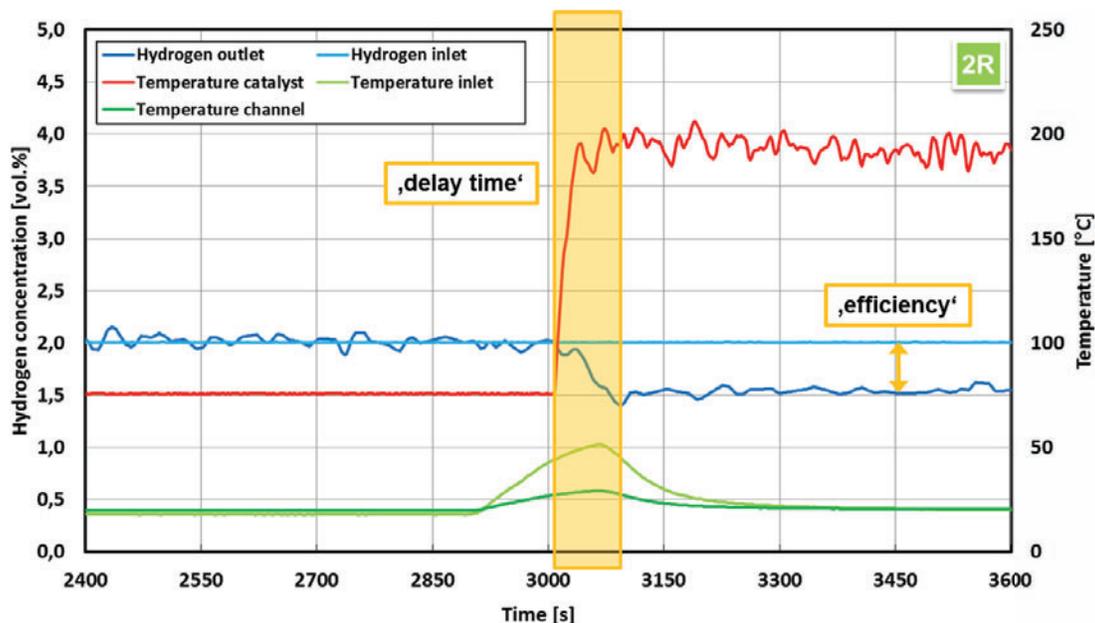


Figure 10. Key indicators for the performance assessment (reference sample).

4.3. Performance Test Results

The performance test results for all catalyst samples of Type 2 are summarised in Table III. The table includes the hydrogen concentration at which the catalytic reaction starts (with or without pre-heating) and the conversion efficiency which was observed at 2 vol.% hydrogen for both test phases. Furthermore, for phase 1 the observed delay time quantifying the impact of the soot contamination on the start-up behaviour is given. For phase 2, the start-up time indicates how fast the catalytic reaction starts after hydrogen injection.

Table III. Performance test results

	catalyst sample	Phase 1				Phase 2		
		start concentration	pre-heating	delay time	efficiency @ 2 vol.%	start concentration	start-up time	efficiency @ 2 vol.%
		vol.%	°C	s	%	vol.%	s	%
CFS-1	2-R	2,0	27,4	29	22,0	1,0	46	27,5
	2-B	2,0	66,1	2839	23,0	1,0	36	22,5
	2-C	2,0	46,3	1001	24,1	1,0	45	22,8
CFS-2	2-R	1,5	19,6	38	23,4	1,0	159	23,3
	2-B	2,0	36,0	2755	19,0	1,0	96	20,6
	2-C	2,0	19,6	1944	19,9	1,0	54	21,3

In phase 1, almost all catalyst samples start at a hydrogen concentration of 2 vol.% with more or less intense pre-heating. The only exceptions are samples 2-R and 2-C of test CFS-2 which start without pre-heating (2-R at 1.5 vol.%). Direct comparison of the catalyst sample characteristics (e.g. the pre-heating

temperature needed for start-up) would be misleading as all catalyst samples (even the reference samples) have different histories and have not been activated at the beginning of the present investigation. However, the delay time observed during test phase 1 differs largely between the samples. While all reference samples immediately achieve maximum efficiency after reaction start (delay time between 29 s and 38 s), the soot-contaminated sheets need significantly more time (between 1001 s and 2839 s). Independent of the delay time, all catalyst sheets achieve very similar steady-state conversion efficiencies between 19.0 % and 24.1 %.

For illustration purposes, Figure 11 compares the start-up behaviour of the type 2 catalyst samples from test CFS-1. The reference catalyst sample 2-R (red line) reaches full conversion efficiency (dotted green line) 29 s after start-up. For sample 2-C (blue line), it takes approx. 16 min longer until the steady-state conversion rate is reached. The impact of the cable fire soot on sample 2-B (black line) is even more pronounced. It takes more than 45 min for the catalyst to regain full conversion efficiency.

In test phase 2, all catalyst samples which have been activated during test phase 1 start recombination at 1 vol.% hydrogen without pre-heating. Although at very low hydrogen concentration, all samples reach full conversion efficiency immediately. In phase 1, this has only been the case for the reference sample sheets. The conversion efficiency for 2 vol.% hydrogen amounts to 20.6 % - 27.5 % which is comparable to the values obtained in phase 1.

Due to the small number of samples, no clear statement on the correlation of the soot mass deposited on the catalyst sheet with the delay time can be made. Although samples of type 2 (CFS-1 and CFS-2) suggest a direct correlation, this is not confirmed by samples of different types. Furthermore, there is no clear indication of a different impact for the cable types used in the cable fire tests.

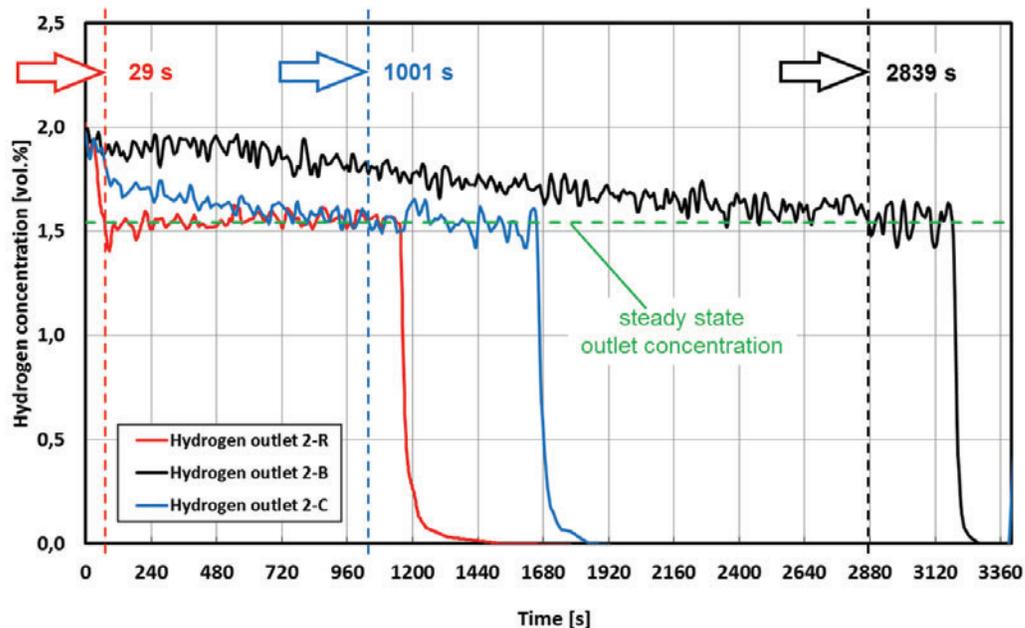


Figure 11. Hydrogen concentration measurements after start of the catalytic reaction ($t = 0$ s) for type 2 samples from cable fire test CFS-1.

5. CONCLUSIONS

The goal of the present joint investigation between IRSN and JÜLICH was to assess the necessity to further studying the impact of severe accident-relevant fire products on the catalytic activity of PARs. The work programme included three steps:

- Two cable fire tests with different cable types and sets of different catalyst samples in the DIVA facility (CFS-1 and CFS-2)
- Subsequent surface analyses of selected catalyst sheets in order to characterise the deposition of fire products
- Performance tests with the remaining catalyst samples in the REKO-1 facility

During the cable fire tests, the catalyst samples were exposed to max. temperatures of approx. 400°C (CFS-1) and 280°C (CFS-2) for a duration of several hundred seconds. The CO concentration measured in the vicinity of the catalyst sheets amounted to approx. 2 vol.% in CFS-1 and 4 vol.% in CFS-2. The combustion of cables produced significant amount of soot on the catalyst surfaces. The total mass of soot on the sheets was only quantified for test CFS-2 and yields approx. 9.5 mg for samples located at the sectional wall sheets of the sample holder and approx. 13.1 mg for the remaining samples.

For the catalyst samples from both cable fire tests, surface analyses show significant amount of carbon, chlorine (constituent of PVC), zinc, and antimony (flame retardant) compared to reference samples. For samples from test CFS-2, copper and lead were identified as well. Corrosion effects as observed after the Ringhals fire event were not observed in this investigation, which could be expected due to the absence of steam in the investigation.

The performance tests show that all catalyst sheets from the cable fire tests start with a significant delay. While reference samples reach full conversion capacity within 29 s – 95 s after start-up of the catalytic reaction, contaminated samples need between 7 min – 45 min. However, after burning off the soot deposition, the catalyst samples reach full conversion capacity and show immediate start-up behaviour in a subsequent test. Hence, no permanent degradation effect can be observed. Based on the available results, no influence of the cable type on the start-up characteristics can be detected. Whether or not the amount of soot deposited on the catalyst surface has an immediate influence on the start-up delay time remains an open question due to the small number of catalyst samples.

In the light of the present results, the adverse effect of cable fire products on the efficiency of hydrogen conversion in a PAR is clearly demonstrated. In order to further understand and quantify the impact of cable fire products and to assess the relevance for severe accident scenarios, further experimental as well as theoretical investigations are required. The present investigation provides a profound basis for the justification of further research on this topic. The combined influence of cable fire products and humidity, which had been intentionally omitted in the present activity, should be investigated in the future due to its possible corrosive impact on the catalyst. Humidity is also likely to play an important role for the nature of the soot deposition due to clogging effects.

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