Implementation of O_2/H_2 Catalytic Ignition at -40 °C and -80 °C

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Abstract

Propellant settlement for cryogenic upper stage can be achieved using gaseous H_2 combustion at negative temperatures (-40 and -80 °C) initiated by catalytic ignition. In this study, foam and honeycomb monolith shape-formed catalysts with different active phases (Ir, Pt, Rh, Ir-Rh) have been tested on a lab scale test bench modified recently. Results at -40 °C demonstrated that monolithic catalysts were the most efficient supports with the following order of active phase activity, based on temperature recorded at the reactor outlet: Ir-Rh >> Ir ≈ Rh > Pt. Tests at -80 °C confirmed that the combination of the best support and active phase ranked the bimetallic Ir-Rh/C600cpsi as a very promising catalyst for such application.

1. Introduction

The catalytic ignition of O_2/H_2 or air/H_2 mixtures has been known for almost two centuries when J. W. Döbereiner discovered in 1823 that the ignition of a stream of hydrogen directed at finely divided platinum powder caused the platinum to become white hot [1]. The catalytic ignition of cold O_2/H_2 mixtures for propulsion applications started in the 1960s in the United States. Catalytic materials are pellet shaped and the best catalysts are precious metals supported on alumina [2]. The ignition temperature is related to a balance between the blocking of the hydrogen adsorption sites and of the hydrogen desorption sites. It increases when the O_2/H_2 ratio decreases [3].

Cryogenic upper stage of launcher uses liquid oxygen/liquid hydrogen and needs the propellant settlement during the ballistic phase in order to have an efficient propellant supply to the engine. This could be obtained through the ignition of very cold O_2/H_2 mixtures, by directly taking off the gas phase from the O_2 and H_2 tanks. Gases are then mixed and pass through the catalytic system for combustion ignition. Catalytic starters have been studied to make the propulsion system simple, light, reliable, cheap, energetically independent and not suggested to electromagnetic perturbation. In this system, the adiabatic flame temperature is kept at less than 1100 K by working with H_2 rich mixture. This allows using standard ceramic supports such as cordierite for the conception of catalysts while remaining in a sufficient range of specific impulse to assess the targeted settlement.

Firstly, this study was dedicated to the improvement of a dedicated, home made test bench, including gas line modifications and optimisation of the computer programme used for both the control of the bench actuators and data acquisition using LabVIEW software. The new profile of temperature curves thus obtained allows a better understanding of the combustion behaviour and an easier comparison of the catalytic activity.

Secondly, results of H_2/O_2 ignition obtained with catalysts supported on cellular ceramics using this recently designed, developed and modified test bench are presented. Cylindrical cordierite honeycomb monoliths and foams have been used as cellular ceramic supports for the preparation of M/honeycomb or M/foam catalysts (M = Pt, Ir, Rh, Ir-Rh), where M corresponds to the active phase deposited onto the support after its washcoating. Each catalyst has been tested for the catalytic ignition of the mixture at – 40 °C and – 80 °C.

2. Experimental

2.1 Catalysts preparation and characterization

The supports were washcoated with boehmite colloidal suspension, dried and calcined, then impregnated with the precursor solution and finally dried and reduced [4]. Monolith supports are made of cordierite ($2MgO{\cdot}2Al_2O_3{\cdot}5SiO_2$) and have a nominal channel density of 600 channels per square inch (cpsi). Foams are made of cordierite with a nominal pore density of 20 pores per inch (ppi). Dimensions of the supports are 10 mm in diameter and 20 mm in length. Additional geometrical parameters and loading characteristics of the catalysts (percentage of washcoat and metal on monolithic and foam) are presented in Table 1 and Table 2, respectively.

Table 1: Values of geometric parameters of monolith and foam supports used

Geometric parameters for 20 ppi mullite foam		**Geometric parameters for 600 cpsi cordierite monolith**	
n(mm^{-2})	0.17	n (mm^{-2})	1.00
d_p (mm)	1.50	d_{ch} (mm)	1.00
e_p (mm)	0.60	l_{ch} (mm)	0.20
GSA	1.18	GSA	3.20
OFA	0.59	OFA	0.64
D_h (mm)	2.00	D_h (mm)	0.80
R_f (mm^{-2})	12.74	R_f (mm^{-2})	69.34
		MIF	0.05
		TIF	5.00
H_s (mm^{-2})	2.31	H_s (mm^{-2})	14.40

Nomenclature: d_p =pore size (mm); e_p = wall thickness (mm); n = cell density (mm^{-2}); GSA = geometric surface area (mm^{-1}); OFA = open fraction area; D_h = hydraulic diameter; R_f = resistance to flow (mm^{-2}); H_s = bulk heat transfert (mm^{-2}); d_{ch} = channel size (mm); l_{ch} = wall thickness (mm); MIF = mechanical integrity factor (dimensionless); TIF = thermal integrity factor (dimensionless)

Table 2: Loading characteristics of the catalysts

Support	**Metal**	**Mass support (g)**	**Washcoat (%)**	**Metal/washcoat (%)**	**Metal/catalyst (%)**
Monolith (Cordierite, 600 cpsi)	Pt	0.4796	29.4	20.0	6.8
	Rh	0.4960	29.0	18.8	6.3
	Ir	0.5023	29.7	19.7	6.8
	Ir-Rh	0.5020	28.8	33.7	12.8
Foam (Cordierite, 20 ppi)	Pt	0.8179	20.5	34.8	9.8
	Ir	0.9000	18.3	35.8	9.3
	Rh	0.8993	16.3	40.4	10.0
	Ir-Rh	0.9956	15.2	38.3	8.6

Catalysts are denominated M/C600cpsi and M/Cm20ppi. M is the metallic phase and could be either Ir, Rh, Pt or Ir-Rh. C600cpsi defines the monolith support while the Cm20ppi is related to the foam support. Photos of some of the final catalysts are presented on the Figure 1.

Rh/Cm20ppi Ir-Rh/C600cpsi

Figure 1: Examples of fresh catalysts for combustion tests

2.2 Catalytic tests

The stainless steel reactor used for tests at the lab level, in reaction conditions representative of real ones, is equipped with two pressure sensors placed upstream and downstream of the catalyst, and five thermocouples (TC1, TC2, TC3, TC4 and TC5). The test bench can be divided into three major parts following the gas flow. The first part of the experimental device is the flow regulation system in which N_2, O_2 and H_2 are regulated separately and then mixed. The second part is the cooling gas system composed of a helical coil plunged in an isolated cold bath. The third part is the reactor. TC1 is placed upstream of the cold bath, TC2 downstream of the cooling bath, TC3 at the reactor inlet into the piping (so that the end of the thermocouple lies in the axis of the pipe, that is directly in contact with the stream), TC4 in the middle of the wall of the reactor (in a hole which is of 1.5 mm depth drilled in the wall of about 3 mm thickness), TC5 at the reactor outlet directly in contact with the exhaust gas [5].

Catalysts tests consisted in recording pressure and temperature during O_2/H_2 ignition with the following parameters: O_2/H_2 mass ratio 0.8, gas mixture flow rate 0.08 g s^{-1}, gas flow cooled at –40 °C and –80 °C.

In order to compare tests results obtained before and after modification of the test bench, two catalytic tests corresponding were performed at 0.04 g s^{-1}.

After inserting the catalyst in the reactor, as it is shown on the following pictures (Figure 2), the reactor is cooled down, internally by flowing cooled gaseous nitrogen in the reactor. The nitrogen is cooled down thanks to the helical coil immersed in the cool bath mentioned above. Reactor cooling is aided by pouring liquid nitrogen directly onto the external surface.

The test begins when TC2, TC3, TC4 and TC5 are simultaneously at the desired temperature (-40 °C or -80 °C). The experiment is automatized and piloted using LabVIEW. It consists of five steps. In step 1, N_2 is flown during 120 s, then a flow of N_2 + H_2 is regulated during 120 s (step 2), afterwards the combustion step begins (step 3) when only H_2 and O_2 are flown during 120 s. Step 4 consists in closing H_2 and O_2 feeding valve and in opening N_2 one to stop the reaction, purge the piping, and cool down the catalyst and the reactor. The step 3 is stopped after 120 s in order to avoid an excess of heating which could damage the reactor.

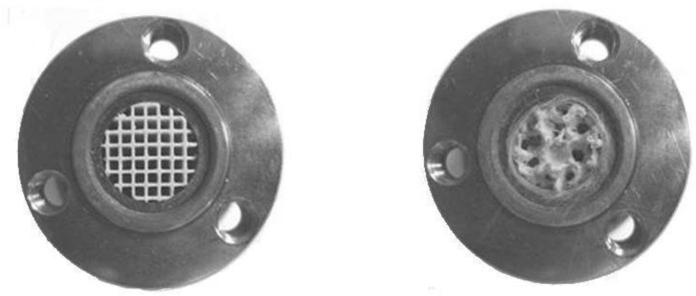

Figure 2: Photo of nude monolith and foam placed in the reactor

Changes made on the experimental bench and its programme concerns the step 2 during which N_2 and H_2 are flown at the same time. This new step of 120 s allows controlling the regulation on the H_2 flow controller and allows also regulating the O_2 flow controller by passing N_2 flow in it. For this, a gas line linking the N_2 line to the O_2 line was added. This pre regulation allows regulating O_2 faster than with the previous experimental bench since molar mass of

O_2 and N_2 are close to each other and that just a slight adjustment is needed to regulate the flowrate at the desired value when N_2 it is switched from N_2 to O_2 at the beginning of the step 3.

3. Results and discussion

3.1 Comparison between temperatures profiles before and after modification of the test bench

Catalytic tests were performed at -40 °C with a total flow rate of 0.04 g s^{-1} and a mass ratio (O_2/H_2) of 0.8.

Figure **3** shows a typical temperatures measurement during a test before and after the experimental bench changes. Temperatures measured on TC3 (reactor inlet) and TC4 (reactor wall) show better profile with much less signal perturbations after modification of the tests bench. It is also the case for TC5 (reactor outlet) since the new settings allow a faster regulation at the desired flow rate. Thus, temperature increases drastically in the very first seconds and the maximum temperature measured is 398 °C (TC5) is higher than the final temperature observed with the old settings (398°C against 248°C). Nevertheless, it is important to highlight here that the sudden decrease of temperature observed on TC5 on Figure **3** is due to thermal exchange and will be explained in the next part of the paper.

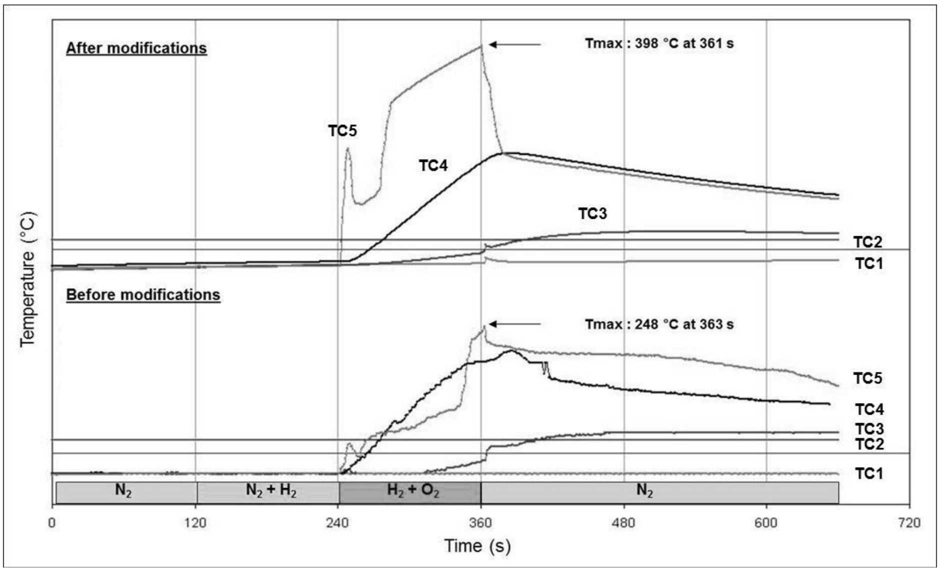

Figure 3: Temperature profiles as recorded from thermocouples TC1 through TC5 before and after the experimental bench modifications. Catalytic tests were carried out on Ir/C600 cpsi, T = -40 °C; Q_{mtot}: 0.04 g s^{-1}; Rm = 0.8; Grey bars at the bottom of the graph depict the nature of the gas flown and detail their duration: Step 1: 120 s; Step 2: 120 s; Step 3: 120 s; Step 4: 300 s. TC1: cooling bath inlet; TC2: cooling bath outlet, TC3: reactor inlet; TC4; middle of the external wall of the reactor, TC5: reactor outlet

Since TC5 recorded directly the temperature of the exhaust gas and consequently measured the maximum temperatures, all the results depicted in the following parts of this paper will be based on TC5 observation.

3.2 Temperature profile at the reactor outlet after modification of the test bench

In order to address the temperature profile observed at the reactor outlet (TC5) and described in the previous section, combustion steps are detailed hereafter supported by the curve profile obtained with the catalyst Ir/C600cpsi and the bimetallic catalyst Ir-Rh/C600cpsi (honeycomb monoliths). These two catalysts were selected as they are representative of the two typical profiles obtained during this study. They are displayed in Figure 4.

For Ir/C600cpsi, the following profile is obtained:

- Start of step 3 (combustion step) at 240 s.

- Start of combustion at 241 s (time needed for H_2 and O_2 to reach the catalyst from the aperture of the electro valves).
- Fast temperature increase up to 288 °C at 246 s.
- Sudden fall of temperature of 100 °C in 4 s.
- Second temperature increase at 250 s.
- Maximum temperature of 529 °C at 361 s.

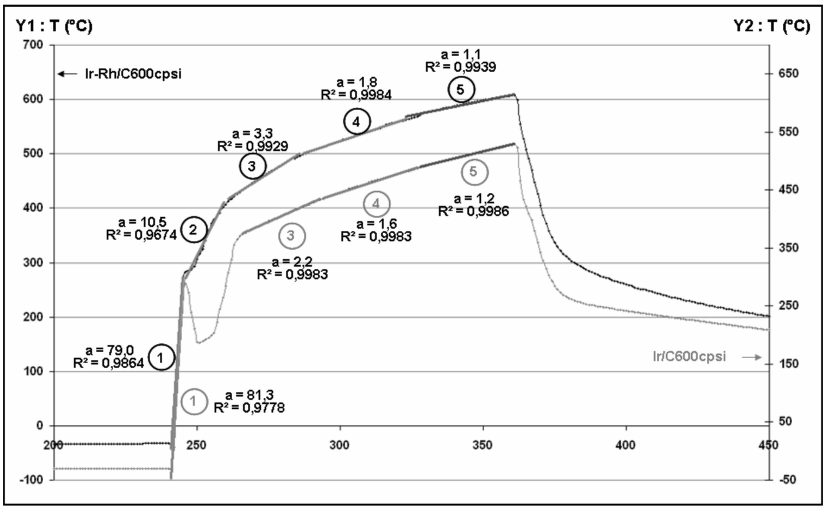

Figure 4: Exhaust gas temperature vs. time during H_2/O_2 catalytic combustion on catalyst monolith shaped. T = -40 °C; Q_{mtot}: 0.08 g s^{-1}; Rm = 0.8; Step 1: 120 s (N_2); Step 2: 120 s (N_2+H_2); Step 3: 120 s (H_2+O_2); Step 4: 300 s (N_2). Segments denoted ① through ⑤ result from the linear regression of different parts of the temperature profiles; "a" and "R²" correspond to the slope obtained from the regression and to the R² value, respectively

The temperature peak observed during 9 s between -40 °C and 250 °C after a maximum temperature of 288 °C could be explained by the cooling effect from the reactor. The energy released by the reaction being high in a short time delay (resulting in a temperature variation of 320 °C in 5 s), the reactor wall cannot be warmed up simultaneously. Thus, the cold wall (-20 °C at 250 °C) could absorb a part of the heat by convection a few seconds before, leading to a sudden decrease of temperature at the outlet of the reactor. Then, temperature increases from 250 s once the wall of the reactor has actually begun to warm up. This phenomenon demonstrates that the heat loss measured on the TC5 is transferred by conduction until the thermal transfer along the reactor wall seems to reach the permanent regime.

This behaviour is less noticeable in the case of the bimetallic catalyst Ir-Rh/C600cpsi for which the second increase begins earlier, so that the temperature loss is not well distinguishable, only a shoulder separating the two thermal events appears. This tendency was retrieved in the case of the foam support, whatever the active phase. Beyond 250 s, it is possible to identify several thermal regimes. The temperature profile latter can be divided in at least five different thermal regimes. Four of them (denoted ①, ③, ④ and ⑤ in Figure 4), in terms of slope value and time, follows punctiliously the profile of the temperature obtained with the catalyst Ir/C600cpsi. This demonstrates that the thermal transfer, leading to the loss of temperature between the regimes ① and ③, might hide another thermal regime, noted ②, only visible on foam temperature profile and on the Ir-Rh phase.

3.3 Ignition at -40 °C

Ignition tests at -40 °C were performed twice for each catalyst with a total flow rate of 0.08 g s^{-1}, a mass ratio O_2/H_2 of 0.8. Figure 5 shows the temperature of the exhaust gas, recorded at the reactor outlet, with Ir, Ir-Rh, Rh and Pt based catalyst deposed on foam. In terms of mass loading of catalyst, the Ir-Rh catalyst is the most active metallic phase for catalytic ignition of cold gas mixture. As it is shown on the Figure 5, the temperature most increases on Ir-Rh since a temperature variation of about 130 °C is obtained in 4 s. Slope of temperature for Ir-Rh, Rh, Ir and Pt as obtained from the linear regression of the points shown in Figure 5 are 32.1, 32.7, 30.9 and 22.4 °C s^{-1}, respectively. Moreover, it can be underlined that the activity order observed 2 min is the same as the one obtained in the first seconds.

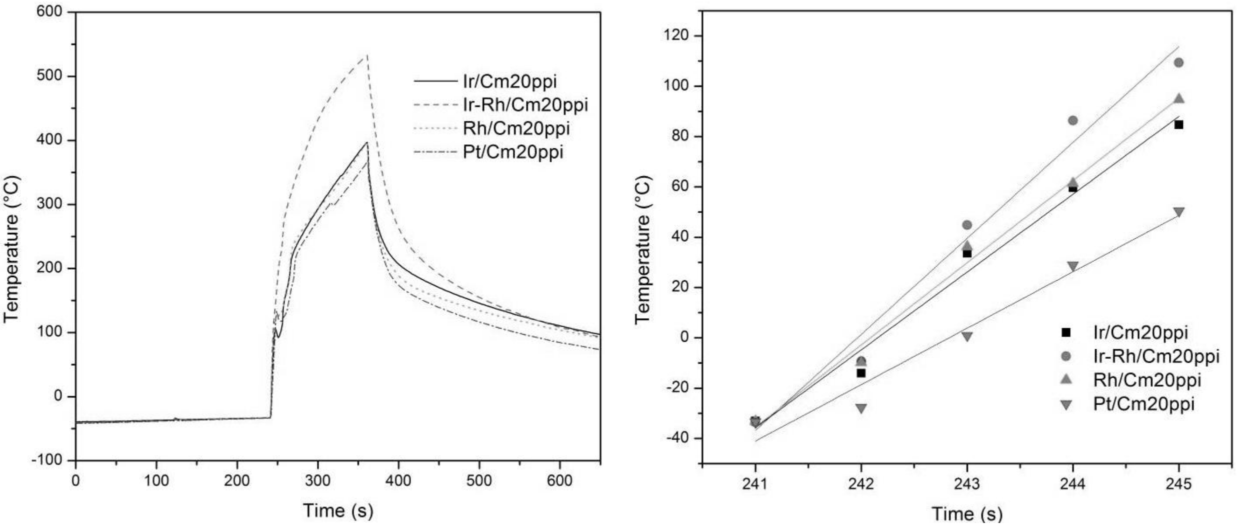

Figure 5: Exhaust gas temperature versus time during H_2/O_2 catalytic combustion on foam (denoted “Cm20ppi”) shaped catalyst with Ir, Pt, Rh or Ir-Rh as active phase deposited on foam for H_2 combustion. T = -40 °C; Q_{mtot} : 0,08 g s^{-1}; Rm = 0.8 ; Step 1: 120 s (N_2) ; Step 2: 120 s (N_2+H_2); Step 3: 120 s (H_2+O_2); Step 4: 300 s (N_2)

Monolith supports have also been tested with the same active phases. Temperature curve profiles obtained with Ir on foam are compared in Figure 6 with the results obtained on monolith. The difference of efficiency between these two supports is observed from the first seconds. In fact, yield of H_2 combustion is higher on monolith than on foam since because of a very important temperature difference. For example, the difference of temperature reached is about 160 °C after 35 s of reaction.

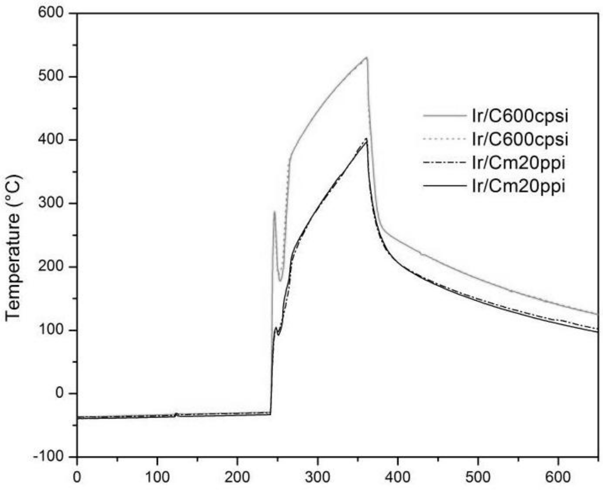

Figure 6: Reactor outlet temperature profiles obtained with honeycomb monolith (“C600cpsi”) and foam (“Cm20ppi”) shaped catalysts with Ir as active phase for H_2 combustion. T = -40 °C; Q_{mtot} : 0.08 g s^{-1}; Rm = 0.8; Step 1: 120 s (N_2); Step 2: 120 s (N_2+H_2); Step 3: 120 s (H_2+O_2); Step 4: 300 s (N_2)

Table 3 summarizes the mean value of the maximal temperature reached which was in all cases observed at 361 s and the thermal efficiency calculated for each catalyst tested. The difference between the time at which the maximal temperature is reached (361 s) and the stop of reactive gas flow (360 s) is due to the dead volume between the electrovalves and the catalyst (same delay observed at the beginning of the step 3 between the aperture of O_2 electrovalve and the ignition of the combustion reaction related to the contact of the reactant with the catalyst).

The adiabatic temperature being equal to 1096 K (determined by calculation at 298 K) for a O_2/H_2 mass ratio of 0.8 and a mass flow rate of 0.08 g s^{-1}, a thermal efficiency of the system can be determined using equation 1.

$$\eta_T = \frac{T_{\exp} - T_{entrance}}{T_{adia} - T_{entrance}} \times 100 \qquad (1)$$

with:

- T_{exp}: Maximal experimental temperature measured (K);
- T_{adia}: Calculated adiabatic temperature (K);
- $T_{entrance}$: Corrective temperature corresponding to the gas mixture temperature at the exit of the cool bath (K);
- η_T: Thermal efficiency of the system (%).

Table 3: Maximum temperature and thermal efficiency obtained at -40 °C with O_2/H_2 mass ratio of 0.8 and a mass flow rate of 0.08 g s^{-1}. “C600cpsi” corresponds to the monolith, “Cm20ppi”, to the foam

Metallic phase	**Support**	**Outlet gas temperature (°C)** **Maximum average measured at 361 s (°C)**	**Thermal efficiency (%)**
Ir	C600 cpsi	529.5	65.9
	Cm20 ppi	399.9	50.9
Rh	C600 cpsi	nd	nd
	Cm20ppi	393.1	50.1
Pt	C600 cpsi	nd	nd
	Cm20 ppi	365.2	46.8
Ir-Rh	C600 cpsi	610.3	75.3
	Cm20 ppi	531.6	66.1

It can be deduced from the description in Table 3 of the maximum average temperature measured at the outlet of the reactor as well as from the thermal efficiency that the most suitable active phase for catalytic ignition of H_2/O_2 mixture at -40°C is Ir-Rh. Nevertheless, Pt is the less active with a maximal temperature of 365.2 °C on foam. The two other metallic phases (Rh and Ir) have similar catalytic activity. Although the foam structure is supposed to cause more flow turbulence than honeycomb monolith and based on maximal temperature reached at the reactor outlet after 361 s, all the foam shaped catalysts tested could be considered as less active than catalyst displaying a monolith structure. This fact could be explained by the limited accessibility of a part of the active phase, even though metal loading is larger in foams than in honeycomb monoliths. All these results allow giving an order of efficiency of the catalytic systems based on the exhaust gas temperature:

- Ir-Rh >> Ir ≈ Rh > Pt
- Monolith > Foam

3.4 Ignition at -80 °C

Catalytic tests at -80 °C were carried out with the same parameters as for the previous experiments using foams and monoliths. Table 4 summarizes the average of temperature measured at the outlet of the reactor at 245 s and the average of the maximal temperature reached at the reactor outlet which was in all cases observed at 361 s as well as the thermal efficiency of each catalyst.

Table 4: Maximum temperature and thermal efficiency obtained at -80 °C with O_2/H_2 mass ratio of 0.8 and a mass flow rate of 0.08 g s^{-1}. “C600cpsi” corresponds to the monolith, “Cm20ppi”, to the foam

Metallic phase	Support	Average temperature on TC5 at 245 s (°C)	Maximal average temperature on TC5 at 361 s (°C)	Thermal efficiency (%)
Ir	C600cpsi		No catalytic ignition	
	Cm20ppi		No catalytic ignition	
Rh	C600cpsi		No catalytic ignition	
	Cm20ppi		No catalytic ignition	
Pt	C600cpsi		No catalytic ignition	
	Cm20ppi		No catalytic ignition	
Ir-Rh	C600cpsi	222.3	500.8	64.3
	Cm20ppi	64.7	497.5	63.9

Table 4 shows that only one active phase, among those which were tested, is suitable for the catalytic ignition at -80 °C, namely Ir-Rh. The two supports allow obtaining similar maximal temperature even if different thermal behaviours are observed as highlighted by Figure 7 which displays the two tests performed on both catalysts.

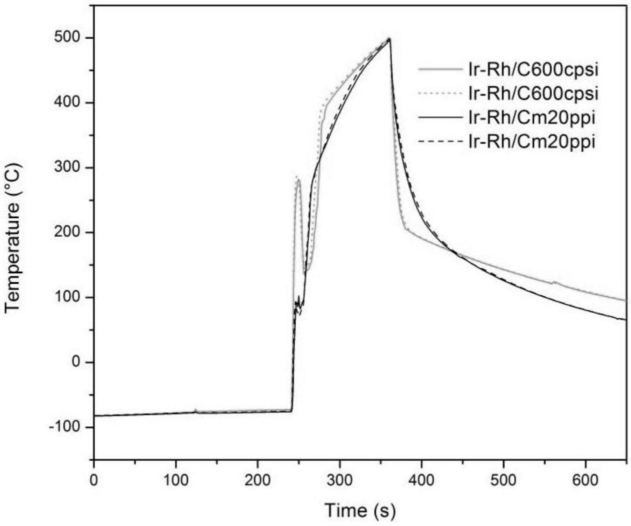

Figure 7: Reactor outlet temperature profiles obtained with a honeycomb monolith (“C600cpsi”) and foam shaped catalysts (“Cm20ppi”) with Ir-Rh as active phase for H_2 combustion. T = -80 °C; Q_{mtot}: 0,08 g s^{-1}; Rm = 0,8; Step 1: 120 s (N_2); Step 2: 120 s (N_2+H_2); Step 3: 120 s (H_2+O_2); Step 4: 300 s (N_2)

Figure 8 depicts the comparison of the temperature profiles obtained at both temperatures (-40 and -80 °C) on both catalysts (honeycomb monolith and foam) on which Ir-Rh active phase was deposited. The maximal temperatures observed at the outlet of the reactor for experiments carried out at -80 °C are below those obtained from experiments at -40 °C (Figure 8a). This difference is higher on honeycomb monoliths than on foams in which just a temperature difference of 34 °C can be underlined. This corresponds to a small loss of thermal efficiency of 2.2 %. In the case of honeycomb monoliths the temperature difference is about 110 °C with a loss of thermal efficiency of 11 %. Otherwise, reaction kinetics, and consequently the ignition delay, is higher on honeycomb monolith (Figure 8, right). It can also be noticed that in the first 5 s of reaction, both temperatures profile are close to each other when comparing results obtained, on the one hand from Ir-Rh/C600cpsi (-40 °C) and Ir-Rh/C600cpsi (-80 °C), and on the other hand, with Ir-Rh/Cm20ppi (-40 °C) and Ir-Rh/Cm20ppi (-80 °C). Noticeable variations are obtained from approximately 75 °C (on foam) and 150 °C (on honeycomb monoliths).

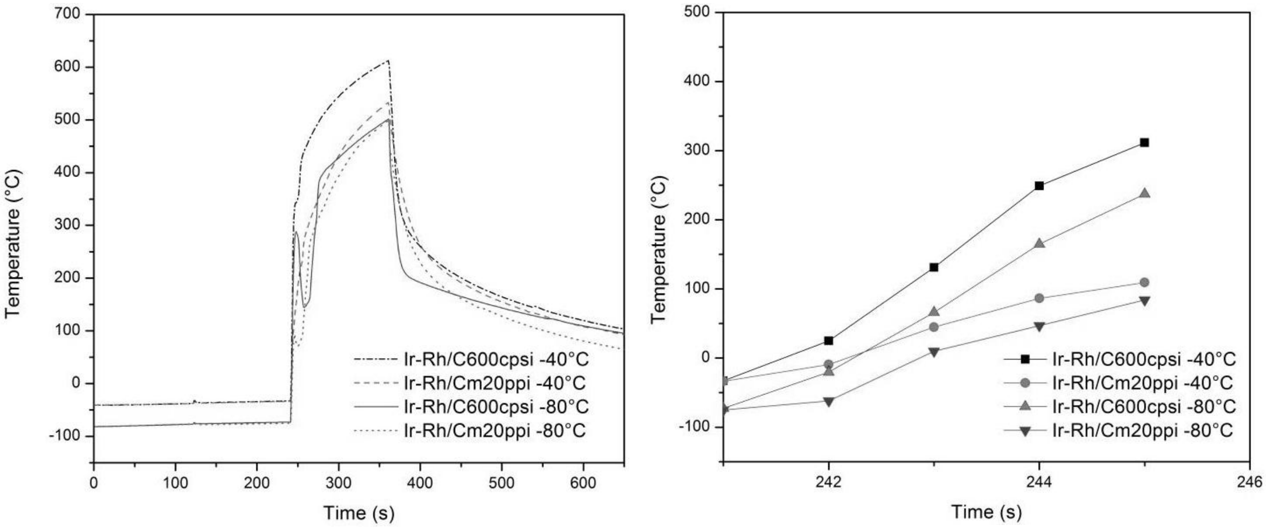

Figure 8: Comparison between honeycomb monolith (“C600cpsi”) and foam shaped catalysts (“Cm20ppi”) with Ir-Rh as active phase for H_2 combustion. T = -40 °C; $Q_{mtot} = 0.08$ g s^{-1}; Rm = 0.8; Step 1: 120 s (N_2); Step 2: 120 s (N_2+H_2); Step 3: 120 s (H_2+O_2); Step 4: 300 s (N_2)

Other metallic phases tested (Ir, Pt, Rh) showed no activity for the catalytic ignition of H_2/O_2 at this temperature. Previous work done by NASA [6] showed that catalytic activity to initiate H_2/O_2 reaction increases with increasing metal content with a maximum passing through 20 wt. % of noble metal for Pt/Al_2O_3 and 29 wt. % for Ir/Al_2O_3. With such content of Ir, an activity was detected at -195 °C. All the catalysts tested in this study contained between 6 and 13 wt. % of active phase. Considering this statement, results obtained with the bimetallic catalysts, even at lower active phase loading (12.8 and 8.6 wt. % on honeycomb monolith and foam, respectively), underline the high performance of such catalysts. A capacity of enhancement by increasing the metal loading is expected.

3. Conclusion

O_2/H_2 ignition tests at -40 °C and at -80 °C were carried out on two series of catalysts defined as M/C600cpsi and M/Cm20ppi in which M is the active phase (M=Ir, Pt, Rh, Ir-Rh). Each catalyst was tested twice for catalytic ignition, using a flow rate of 0.08 g s^{-1} and a O_2/H_2 mass ratio of 0.8.

Results obtained at -40 °C have shown the feasibility of catalytic ignition system for H_2/O_2 mixture and those for all metallic phases (Ir, Pt, Rh, Ir-Rh) and supports (foam or honeycomb monolith) tested. These catalytic tests also allowed discriminating the activity difference of catalysts based on the maximum temperature measured at the reactor outlet and the corresponding temperature slope in the first seconds of the transient: Ir-Rh >> Ir ≈ Rh > Pt. Honeycomb monolith supports led to obtain a better thermal efficiency than foam. Indeed, though foam structure favours the diffusion of species in the catalyst, the accessibility to a part of the active phase might be reduced.

At -80 °C only Ir-Rh metallic phase allowed ignition. Nevertheless, results highlighted a loss of thermal efficiency compared to test at -40 °C. This loss was more important in the case of monolith support with 11 % against 2.2 % for foam support. Other metallic phases (Pt, Rh, Ir) tested did not show catalytic activity at such metal loadings.

Finally, Ir-Rh seems to be a promising catalyst for such application. Further tests at -120 °C should be performed to confirm the high activity of this bimetallic phase.

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