

# Fuel processing in integrated micro-structured heat-exchanger reactors

G. Kolb\*, J. Schürer, D. Tiemann, M. Wichert, R. Zapf, V. Hessel, H. Löwe

*Institut für Mikrotechnik Mainz GmbH, Carl-Zeiss-Str. 18-20, D-55129 Mainz, Germany*

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## Abstract

Micro-structured fuel processors are under development at IMM for different fuels such as methanol, ethanol, propane/butane (LPG), gasoline and diesel. The target application are mobile, portable and small scale stationary auxiliary power units (APU) based upon fuel cell technology. The key feature of the systems is an integrated plate heat-exchanger technology which allows for the thermal integration of several functions in a single device. Steam reforming may be coupled with catalytic combustion in separate flow paths of a heat-exchanger. Reactors and complete fuel processors are tested up to the size range of 5 kW power output of a corresponding fuel cell. On top of reactor and system prototyping and testing, catalyst coatings are under development at IMM for numerous reactions such as steam reforming of LPG, ethanol and methanol, catalytic combustion of LPG and methanol, and for CO clean-up reactions, namely water-gas shift, methanation and the preferential oxidation of carbon monoxide. These catalysts are investigated in specially developed testing reactors. In selected cases 1000 h stability testing is performed on catalyst coatings at weight hourly space velocities, which are sufficiently high to meet the demands of future fuel processing reactors.

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**Keywords:** Fuel processing; Micro-structured reactors; Plate heat-exchanger reactors; Catalyst coatings

## 1. Introduction

Within the scope of hydrogen generation for fuel cells systems space demand and weight are critical issues for small and medium sized applications ranging from few watts to some ten kilowatts. Thus, the process intensification benefits of micro-technology [1] are currently within focus of the world-wide research related to fuel processing. Numerous reactors and even complete fuel processor systems have been realised applying micro-technology [2], which is documented by a recently dramatically increasing number of publications.

Generally, a fuel processor is composed of a reformer reactor and – at least in case of small scale systems – catalytic gas purification reactors to purify the reformate from carbon monoxide, which is poisoning the catalyst of low-temperature PEM fuel cells, the most common fuel cells for small scale applications (see Fig. 1). Additionally, fuel and/or water evaporators and heat-exchangers are required, the latter to adjust the feed temperatures of the individual reactors and to improve overall system efficiency.

The fuel determines the operating temperature of the reforming process. Methanol steam reforming is performed in micro-reactors at temperatures not exceeding 300, 600–650 °C are required for ethanol, whereas hydrocarbon steam reforming takes place at even higher temperatures (700–750 °C for LPG and gasoline and even higher for methane and diesel).

Integrated heat-exchangers/reactors open the door to novel processing concepts. As shown in Fig. 1, the endothermic steam reforming reaction may be coupled to an exothermic catalytic combustion in such reactors, which was first time published by Eigenberger and co-workers for methane steam reforming in meso-scaled reactors made from ceramic monoliths and steel foils [3–7].

Reuse et al. demonstrated the plate heat-exchanger concept in micro-structures for methanol steam reforming [8]. The power for the endothermic steam reforming was generated by the exothermic combustion of hydrogen, which would be provided by the off-gas of the fuel cell anode in a future fuel cell/fuel processor system. This off-gas still contains some 10–15 Vol.% hydrogen or more.

However, for all fuels apart from methanol the evaporation of water and liquid fuels requires additional fuel to be combusted in a combined catalytic burner/evaporator as shown in Fig. 1. Another alternative would be to reduce the hydrogen

\* Corresponding author. Tel.: +49 6131990341; fax: +49 6131990305.  
E-mail address: [kolb@imm-mainz.de](mailto:kolb@imm-mainz.de) (G. Kolb).

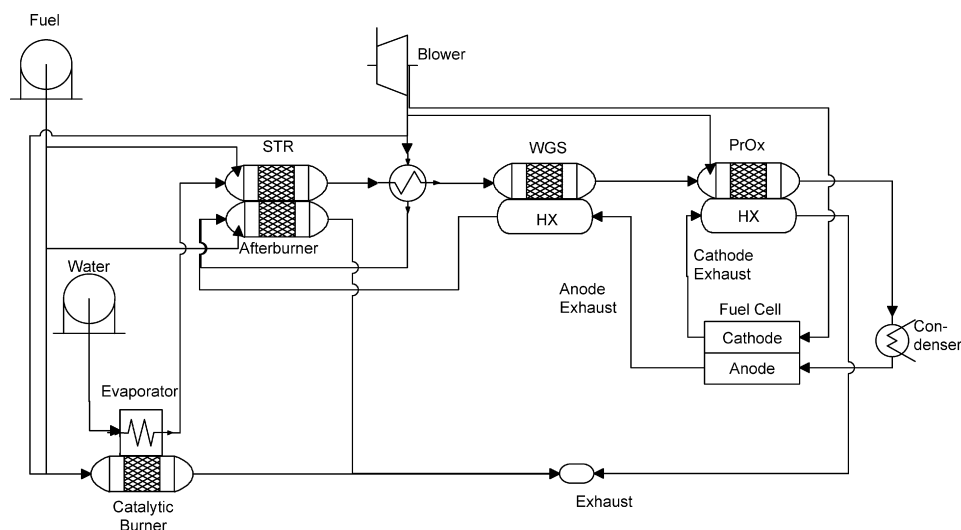


Fig. 1. Flow scheme of a fuel processor based upon catalytic CO-clean-up.

utilisation of the fuel cell and supply more residual hydrogen via the anode off-gas to the catalytic burners. Simulation work performed by Cutillo et al. for diesel fuel processing [9] revealed, that steam reforming applying anode off-gas as heat source generates higher system efficiency compared to autothermal reforming. For a methane steam reforming fuel processor, more than 15% higher fuel processor efficiency was determined experimentally by Heinzl et al. [10] when utilising fuel cell anode off-gas.

Furthermore, the gas purification processes are all exothermic and the removal of heat out of these reactors improves their performance. The water-gas shift reaction (WGS) is operated close to its thermodynamic equilibrium. Thus, the process is performed in conventional systems with an intermediate cooling step applying two separate reactors (high-temperature (HT) and low-temperature (LT) water-gas shift). On the other hand the space demand of the shift reactors is a crucial problem in fuel processors relying on conventional technology. Here micro-technology may well bring substantial benefit, as integrated micro-structured heat-exchangers/reactors offer the opportunity to work with a single device and an internal temperature gradient, which moves the thermodynamic equilibrium in the desired direction [11]. In Fig. 1, fuel cell anode off-gas is applied as cooling agent to generate the required temperature gradient in the WGS reactor. Numerical calculations revealed, that it is possible to achieve a temperature profile very close to the optimal one in a counter-current heat-exchanger by cooling. The configuration has several definite advantages over the two-step process. The single step process can be made more compact as the total reactor length is shorter by up to 50% and furthermore water-gas shift is performed in a single housing eliminating the space required for connections and fittings between different components. As neither a water injection nor a heat-exchanger is required the plant design is significantly simplified. It could be demonstrated that the single step process offers the advantage of a lower water consumption of the system which in turn opens the possibility of higher plant efficiency because the water removal required downstream increases the heat losses from the system [12]. The

effect of diffusion limitation was investigated and revealed that channels of more than 400  $\mu\text{m}$  diameter require higher reactor size to achieve the same degree of conversion [12]. Because the water-gas shift reaction has low reaction rate compared to hydrocarbon reforming, diffusion limitations are an even more stringent factor in the latter case.

Finally the selective or preferential oxidation of carbon monoxide (PrOx) is an exothermic reaction as well, where the operating temperature of the catalyst needs to be kept in a narrow range to achieve optimum conversion. Thus, removing the heat generated by the chemical reaction provides potential for improving the reactor performance, and decreasing its size. In Fig. 1, fuel cell cathode off-gas is applied as cooling agent to remove the heat of reaction out of the PrOx reactor in a co-current arrangement.

## 2. Catalyst development

Catalyst development at IMM covers the field of methanol, ethanol and propane reforming, water-gas shift, selective oxidation, methanation and catalytic combustion of hydrogen and various fuels such as hydrocarbons and alcohols. Below some recent results of this research are discussed. Basically, the wash-coating procedure is currently the main deposition method at IMM [13]. Both home-made catalysts are prepared and commercial catalysts are coated into the micro-channels. Much effort has been spent in dedicated research projects to achieve mechanically and thermally durable coatings [14], which are evenly distributed both from channel to channel and over the channel length.

Home-made Rh/Pt/CeO<sub>2</sub> catalyst on alumina wash-coat basis was identified as a good candidate for propane steam reforming. The combination of Rh generating stability and high activity towards reforming, Pt improving the dispersion of Rh and catalyst stability against coking, CeO<sub>2</sub> supplying oxygen to the noble-metals and improving the water-gas shift functionality thus reducing the CO content of the products revealed an excellent performance of the catalyst. Full conversion was achieved

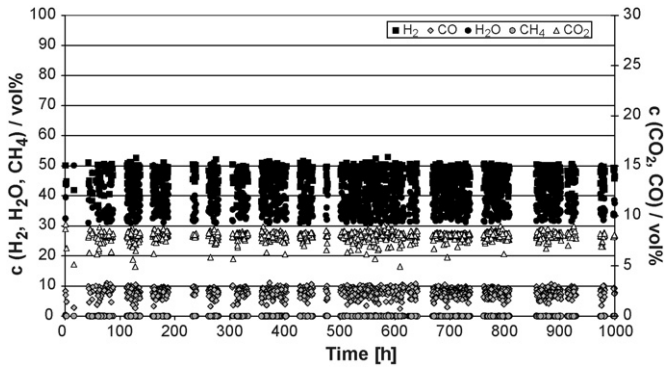


Fig. 2. One thousand hours stability test of a catalyst coating for propane steam reforming in micro-channels; reaction conditions—temperature: 750 °C, pressure: ambient; S/C = 4.0; WHSV: 375 Ndm<sup>3</sup> (h g<sub>catalyst</sub>)<sup>-1</sup>.

for a steam/carbon ratio (S/C) of 3.2 at a temperature of 750 °C and 10 ms residence time. Under these conditions, merely CO and CO<sub>2</sub> were found as carbon species in the product. A WHSV of 375 Ndm<sup>3</sup> (h g<sub>catalyst</sub>)<sup>-1</sup> was achieved at full conversion for the reformer reactor [15]. More recently, an improved, proprietary catalyst formulation proved 1000 h stability at S/C 4.0 for the same WHSV (see Fig. 2).

Total oxidation of fuels is an important reaction when steam reforming is applied, because both the evaporation process and steam reforming require energy supply. The oxidation process needs to be complete (ideally 100% conversion), robust against air surplus, which is necessary to adjust the appropriate operating temperature, and long-term stable. The catalytic combustion of hydrocarbon fuels, however, remains a challenging task. Pt/Mo and Pt/W catalyst formulations were identified as highly active catalyst formulations for the complete catalytic combustion of LPG at moderate temperature and low catalyst loading. The long-term stability of these formulations was proven by 1000 h stability tests, one of them shown in Fig. 3.

For water-gas shift, numerous catalyst systems were under investigation at IMM to come up to the novel demands of fuel processing in micro-structured reactors. For example, there are reformer concepts existing, where air is used for heating the reactors during start-up, which deteriorates the performance of

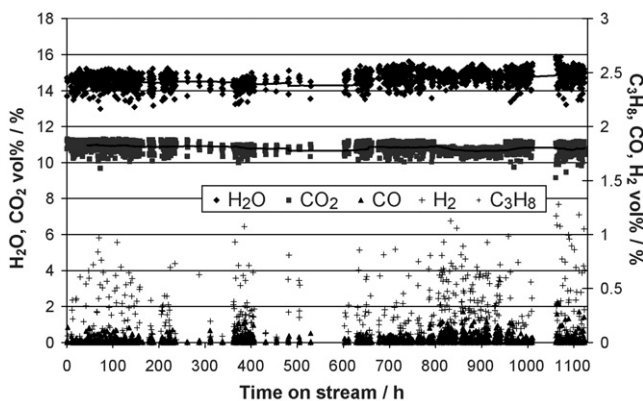


Fig. 3. One thousand hours stability test of a catalyst coating for propane total oxidation in micro-channels; reaction conditions—temperature: 750 °C, pressure: ambient; O/C = 3.3; WHSV: 750 Ndm<sup>3</sup> (h g<sub>catalyst</sub>)<sup>-1</sup>.

Cu/Zn low-temperature water-gas shift catalysts dramatically. Noble-metal based systems are the proper alternative in this case. The water-gas shift catalyst should be capable of covering the whole operating range from high-temperature water-gas shift (about 10 Vol.% CO in feed) to low-temperature water-gas shift (about 3 Vol.% CO in feed). This is important when running an integrated heat-exchanger/reactor which covers both stages. Self developed Pt/CeO<sub>2</sub> catalyst formulations showed significant activity [16] and proved to be stable for 1000 h even in the presence of considerable amounts (5 Vol.%) of methane in the simulated reformat.

The preferential oxidation of carbon monoxide, which is the last step of catalytic CO-clean-up, is also under investigation at IMM [17]. Rh/Pt catalysts proved to be the most active species under investigation and, once more, 1000 h stability could be demonstrated for the Rh/Pt system [18]. Further investigations on the detailed kinetics of the PrOx reaction system are on their way.

### 3. Integrated reactor concepts

The design concepts of micro-structured reactors require adoption to the specific field of application. Testing devices normally have the option to exchange the catalyst carrier plates and they are commonly electrically heated. Thus, gaskets are applied and heating may be done by heating cartridges. This makes the reactors relatively bulky compared to the size of the micro-structured plates incorporated.

However, small fuel cell APU systems are future mass products. Thus, automated fabrication procedures are required for future reactor production. On top of that, fuel processing devices need to have minimum mass to reduce their start-up time demand. Consequently the devices have to be as porous as possible and wall material needs to be minimised as well by applying suitable sealing techniques. Currently, wet chemical etching of the micro-structures into the metal foils and laser welding as sealing technique are applied at IMM for prototype reactors. These techniques are suitable for mass production already. However, other micro-structuring techniques such as embossing, punching and alternative sealing techniques are under investigation in parallel.

Generally, the simplest design of a plate heat-exchanger is a cross-flow arrangement. However, uneven temperature distribution and limited efficiency are major drawbacks of the cross-flow in principle and thus either the co- or counter-current design is favoured for heat-exchangers and reactors at IMM. As soon as the plates are coated with catalyst, the heat-exchanger gets a reactor. One flow path may be utilised to supply or remove heat from the chemical reaction taking place in the second flow path. Alternatively, catalyst may be coated onto both sides of each plate and two chemical reactions performed in the reactor such as steam reforming coupled with catalytic combustion. The simplest appearance of such a reactor is shown in Fig. 4. It is a combined methanol steam reformer/catalytic afterburner designed for a 100 W fuel cell system. Because both steam reforming and combustion take place to a large degree at the inlet of the reactor, a co-current operation of the device is required.

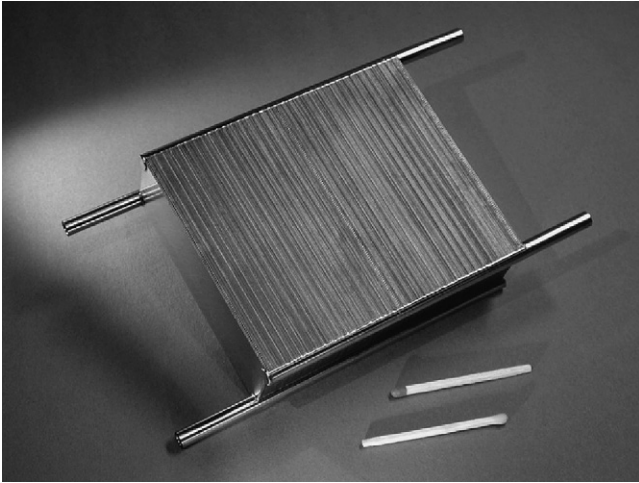


Fig. 4. Combined methanol steam reformer/catalytic afterburner designed for a 100 W fuel cell system.

In the reactor shown in Fig. 4, both feed gas mixtures enter via half-tubes into the device. The gas distribution is then arranged inside the plate geometry. In case minimisation of pressure drop is required, external diffusers may act as gas distributors alternatively. However, up to four functions may be integrated into such a plate heat-exchanger. One further requirement is frequently a dedicated start-up function, which allows immediate supply of heating gas to each device of the fuel processor, a measure which reduces time demand for system start-up considerably compared to conventional technology.

An important feature of micro-channels is their inherent safety because they act as flame arresters. This gets important when explosive gases are processed. Fig. 5 shows a coupled diesel steam reformer/catalytic afterburner which carries dedicated inlets for both anode off-gas and air for burning the residual hydrogen from the fuel cell at temperatures exceeding 800 °C. The reactor, into which catalyst from Johnson–Matthey Fuel Cells was coated, has two inlets for anode off-gas, two inlets for the air supply to the burner, one inlet for the diesel/steam supply,

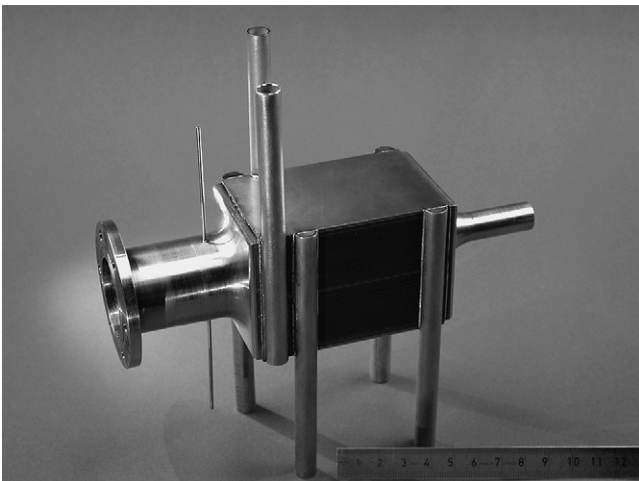


Fig. 5. Combined diesel steam reformer/catalytic afterburner operated at temperatures exceeding 800 °C.

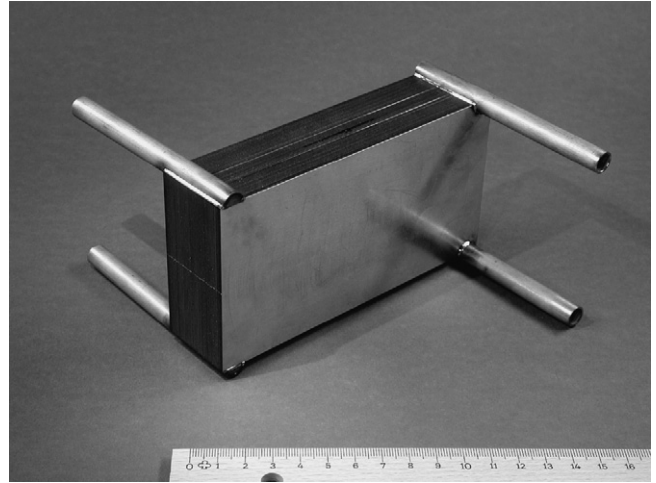


Fig. 6. Integrated water-gas shift reactor/heat-exchanger designed for 2 kW fuel cell system.

and two outlets for the combustion gases apart from the outlet for the reformat. By this arrangement, the feed gases are mixed after entering the micro-structured device, which prevents them from uncontrolled reaction upstream the reactor. Full conversion of the diesel fuel was achieved for a total operation time of 40 h with this reactor to-date, which has a power equivalent of 2 kW thermal energy of the hydrogen produced.

The implementation of a declining temperature gradient into the water-gas shift reactors by counter-flow cooling has been realised for various systems at IMM already. One of these reactors is shown in Fig. 6. It is designed for a power equivalent of 2 kW of the corresponding fuel cell. The temperature profile, which was experimentally determined in the reactor is shown in Fig. 7. After a slight temperature rise at the reactor inlet, which is required to get high initial rate of the reaction, downstream the reactor temperature decreases gradually allowing the content of carbon monoxide to be reduced from 10.6 to 1.05 Vol.%, which corresponds to a conversion of 91%. Somewhat lower temperatures were observed at the shell of the reactor, especially in the inlet section, which has to be attributed to heat losses to the environment.

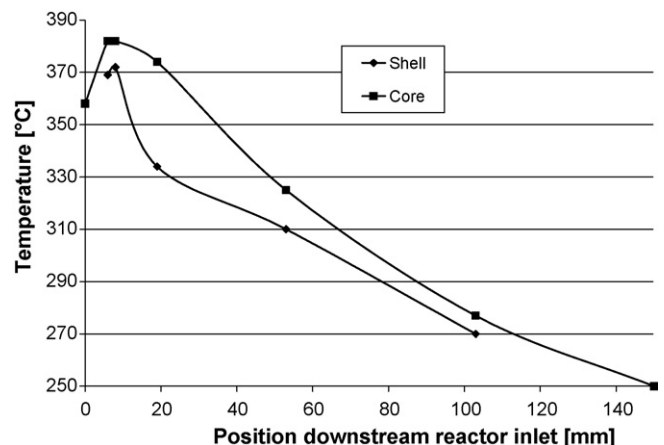


Fig. 7. Internal temperature profile achieved during operation of the reactor shown in Fig. 6.



The principle of integrated cooling of the reactor has been realised for the PrOx reaction as well. However, rather a co-current operation of the reactor is required in this case owing to the fast kinetics of the hydrogen and carbon monoxide oxidation reactions.

#### 4. System design and testing

A complete fuel processor was developed and put into operation, which was designed to supply a 5 kW fuel cell with purified reformat applying *iso*-octane as fuel. *Iso*-octane had been chosen as a model substance for gasoline. Owing to the early stage of this development, the reactors were built either as monoliths or as cross-flow plate heat-exchangers.

Fig. 8 shows a flow scheme of the fuel processor, which was installed on a bread-board level. The test-rig itself is shown in Fig. 9. It was composed of the autothermal reformer reactor (ATR), a heat-exchanger for cooling the reformat downstream the ATR, a cross-flow cooled high-temperature water-gas shift reactor, a low-temperature water-gas shift reactor and a PrOx reactor again cooled by a cross-flow arrangement.

The autothermal reformer (ATR) is shown in Fig. 10. It is composed of 200 micro-structured metal foils of 400  $\mu\text{m}$  thickness carrying a total of 25,000 channels each 400  $\mu\text{m}$  wide and 250  $\mu\text{m}$  deep. The reactor has a total width and height of 80 mm at a total length of 150 mm. It has a monolithic design. The water/steam mixture generated externally by a conven-

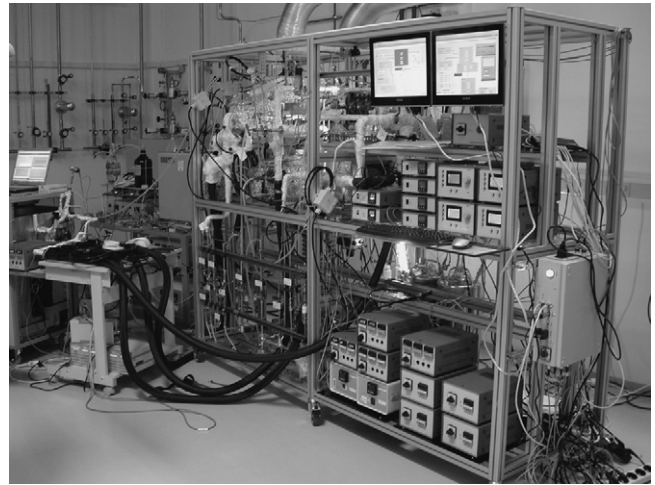


Fig. 9. View of the bread-board fuel processor; the reactors of the fuel processor are placed in the top left area.

tional 8 kW evaporator and superheated to 650–720 °C in a 3 kW micro-structured gas heater (not shown here) was introduced into the reactor inlet. *Iso*-octane vapour was fed directly at the inlet diffuser of the reactor. This arrangement avoided the occurrence of homogeneous reactions in the equipment upstream the reactor itself (i.e. in the gas heater). A 1 wt.% Rh on alumina sol carrier catalyst was coated onto the micro-structured plates, which were then sealed by laser welding and put into a stainless steel housing to assure mechanical stability at the maximum operating

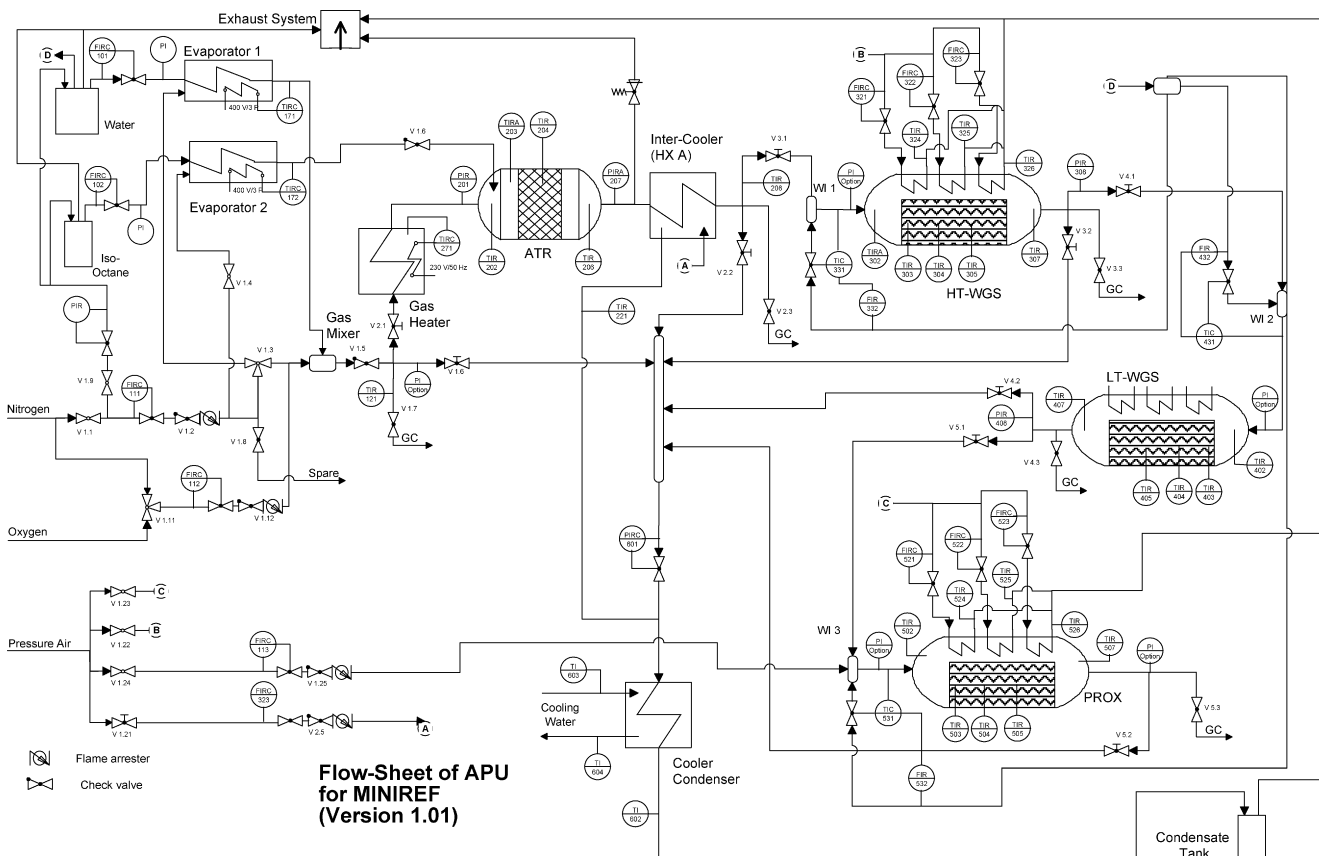


Fig. 8. Flow scheme of the 5 kW fuel processor based upon micro-structured reactors.



Fig. 10. Five kilowatts autothermal reformer for *iso*-octane.

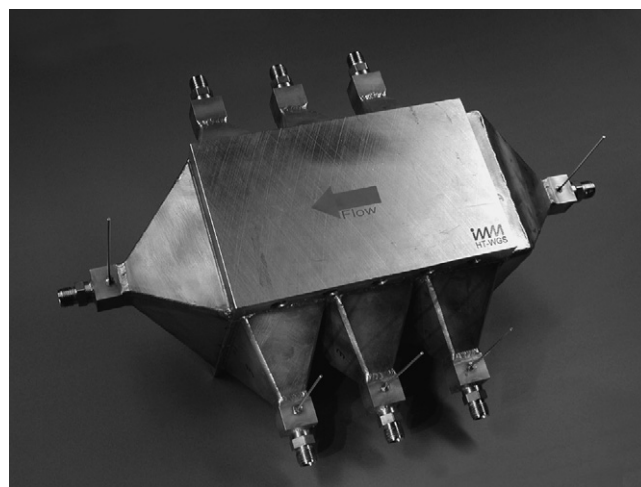


Fig. 11. Five kilowatts high-temperature water-gas shift reactor.

temperature of 800 °C and the maximum operating pressure of 4 bar. The total amount of catalyst incorporated into the reactor was 19.8 g, which corresponds to 0.2 g of the active Rh species. The reactor was operated at a WHSV of  $330 \text{ Ndm}^3 (\text{h}_{\text{cat}})^{-1}$ .

Additionally, a micro-structured heat-exchanger was incorporated between the ATR and the HT-WGS reactor to cool down the reformat from the exit temperature of the ATR to the desired feed temperature of the HT-WGS. It had a heat-exchange capacity of 2 kW under its operating conditions. Pressurised air was applied as cooling agent.

Fig. 11 shows the high-temperature water-gas shift reactor (HT-WGS), which has cross-flow cooling capabilities. The key data of the HT-WGS and of the other CO clean-up reactors discussed below are provided in Table 1. As catalyst for water-gas shift, a 1 wt.% Pt/CeO<sub>2</sub> on alumina carrier developed by Institute de Recherche sur la Catalyse (IRC), Villeurbanne was incorporated into the reactor. The same catalyst was applied for the low-temperature water-gas shift reactor (LT-WGS, not shown here). However, the latter reactor was not designed as a heat-exchanger because of the minor heat-generation by low-temperature water-gas shift.

This does not apply for the preferential oxidation of carbon monoxide (PrOx) and thus cross-flow cooling capabilities were

introduced into the corresponding reactor (not shown here), similar to the reactor for HT-WGS. A catalyst composed of 2 wt.% Pt on zeolite X/Al<sub>2</sub>O<sub>3</sub> developed by Polytechnico Torino was incorporated into the reactor.

The clean-up reactors were switched in series after the ATR.

Prior to the incorporation of the reactors into the test-rig, they had been tested individually for their performance applying octane/steam/air feed for the ATR and simulated off-gas of the reactor upstream for each of the CO clean-up reactors.

Fast on-line (micro-)GC analysis was applied for chemical analysis of the reformat. Samples could be taken after each of the four reactors. The advantage of this analysis system is its low time demand of about 2 min per analysis. Thus, an overview of the performance of all reactors of the fuel processor could be gained within less than 15 min. However, the drawback of the analytical equipment was, that only a water content of less than 15 vol.% could be tolerated by the instrument. Thus, all water had to be removed out of the samples before entering the GC by a tube filled with molecular sieve and results of analysis had to be reported on a dry basis (d.b.).

As shown in Table 2, the gas composition of the reformat changed from reactor to reactor during the operation of the fuel processor until a CO concentration of 120 ppm (d.b.) base was

Table 1  
Key data of the CO-clean-up reactors

Reactor	HT-WGS cooled (three-stage cross-flow)	LT-WGS un-cooled (monolithic)	PrOx cooled (three-stage cross-flow)
Maximum operating pressure (bar)	4	4	4
Maximum operating temperature (°C)	450	450	300
Reactor dimensions ( $L \times W \times H$ ) (mm)	200 × 120 × 120	200 × 120 × 130	200 × 120 × 120
Number of foils	110	130	110
Foil thickness (mm)	1	1	1
Channel dimensions (reactor side) ( $W \times H$ ) (μm)	600 × 800	600 × 800	600 × 800
Channel density (channels in. <sup>-2</sup> )	720	720	720
Total number of channels	16,000	19,000	16,000
Coated channel surface (m <sup>2</sup> )	1.5	1.8	1.5
Catalyst	1 wt.% Pt/CeO <sub>2</sub> on Al <sub>2</sub> O <sub>3</sub>	1 wt.% Pt/CeO <sub>2</sub> on Al <sub>2</sub> O <sub>3</sub>	2 wt.% Pt on zeolite X/Al <sub>2</sub> O <sub>3</sub>
Total catalyst mass (g)	232	274	138
WHSV (Ndm <sup>3</sup> (h <sub>cat</sub> ) <sup>-1</sup> )	38	33	63

Table 2  
Gas composition of the ATR feed and product composition as determined experimentally for the four reactors of the fuel processor

Feed ATR								
S/C 3.3		O/C 0.7						
		calculated [100% Conversion]			experimentally determined			
		feed ATR	product ATR	product ATR [d.b.]	product ATR	product HT-WGS	product LT-WGS	product PrOx
H <sub>2</sub> O	[mol %]	67.3	40.4	0.0	-	-	-	-
N <sub>2</sub>	[mol %]	23.4	18.1	30.4	33.7	30.4	30.4	34.9
O <sub>2</sub>	[mol %]	6.8	0.0	0.0	0.0	3.0	0.0	0.0
C <sub>8</sub> H <sub>18</sub>	[mol %]	2.5	0.0	0.0	-	-	-	-
CH <sub>4</sub>	[mol %]	0.0	1.7	2.9	2.6	3.0	3.0	2.2
C <sub>2</sub> H <sub>4</sub>	[mol %]	0.0	0.1	0.2	0.2	0.2	0.2	0.2
C <sub>3</sub> H <sub>6</sub>	[mol %]	n.d.	n.d.	n.d.	[0.03]	[0.05]	[0.04]	[0.04]
CO	[mol %]	0.0	5.3	8.9	9.4	1.0	0.6	0.0
CO <sub>2</sub>	[mol %]	0.0	8.5	14.2	15.2	21.0	21.3	21.2
H <sub>2</sub>	[mol %]	0.0	25.8	43.3	41.7	46.5	47.2	44.4

achieved after the PrOx reactor. In Table 2, the feed composition of the ATR is shown at the left column. Next the results of a calculation of the ATR product composition are shown, which were determined assuming full octane conversion and establishment of equilibrium conditions for the water-gas shift and methanation reactions at the outlet temperature of the ATR reactor. In the third column of Table 2, the composition of the ATR product as calculated is shown on dry basis. A good agreement with the data as determined experimentally during operation of the fuel processor was achieved (fourth column of Table 2). Besides the desired and expected products CO, CO<sub>2</sub>, H<sub>2</sub> and CH<sub>4</sub>, also small amounts of ethylene and propylene were found in the reformer product. Different to methane, these gases damage the fuel cell catalyst with course of time. The same applies for the catalyst of the CO clean-up reactors downstream the ATR. However, over the total short-term operation of about twenty hours of the fuel processor, a degradation of the reactor performance was not yet observed.

The next columns show the product composition as determined downstream the HT-WGS, LT-WGS and CO-PrOx, respectively.

The CO content of the reformat of 120 ppm would be low enough for a CO-tolerant low-temperature PEM fuel cell system. A second stage PrOx reactor (not shown here), which had been built, did reduce the CO-concentration of the reformat below 50 ppm, which had been demonstrated separately for the single reactor applying simulated reformat. This value is generally regarded as acceptable for reformat tolerant membranes of PEM fuel cells. About 150 Ndm<sup>3</sup> min<sup>-1</sup> reformat were processed in the micro-structured reactors.

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