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CO_2 reforming of methane in a solar driven volumetric receiver–reactor

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Abstract

CO_2 reforming of methane in a solar driven volumetric receiver–reactor has been investigated. This reactor was successfully tested on the Solar Tower Test Facility of the Weizmann Institute, Israel. The power absorbed by the reactor was between 200 and 300 kW. Typical operating temperatures ranged from 700°C to 860°C, with an absolute pressure of 3.5 bars, reaching methane conversions over 80%. Two solar-specific, catalytically-active absorber systems have been developed. These being based on ceramic foam structures, made from α-Al_2O_3 and SiC ceramics, respectively. γ-Al_2O_3 was used as support material and Rh was applied as active metal. The absorber systems were characterised by pre- and post-test analysis. Both inserts showed coke deposition after solar testing, this resulting from problems with the methanator part of the test loop. Some sections in the irradiated front side of the first absorber system, being located close to the gas inlet, were subjected to catalyst poisoning by Na deposits. The second absorber insert has not yet been fully characterised after solar testing. Despite local degradation effects, both absorber systems performed well during solar operation. © 1998 Elsevier Science B.V. All rights reserved.

Keywords: Reforming; Receiver–reactor; Solar testing; Irradiated catalyst; Aging

1. Introduction

In the chemical industry the reforming of methane or other low hydrocarbons with steam is one of the major material-transforming processes, being used for the production of a hydrogen-rich synthesis gas. CO_2 reforming plays a minor role in oxosynthesis, where a synthesis gas with a lower H_2/CO ratio is desired [1]. About 30–40% of the fuel fed into either process is used to provide the energy required for the highly endothermic reactions. Activities at the German Aerospace Research Center (DLR) on methane reforming are concerned with an approach to providing this energy by concentrated solar radiation. In a closed thermochemical loop, the solar energy is converted into chemically bonded energy: the latter can then be stored and transmitted before it is released again via the reverse exothermic reaction for use in power production or as process heat. In an open loop application, solar reforming is used for upgrading fossil fuels by 20–30% with respect to their energetic values: the upgraded fuel can then be fed into the gas turbine of a modern fossil power plant, thus introducing a solar contribution of about 30%.

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Due to the specific properties of a 'concentrated solar radiation' energy source modifications of the conventionally used process technology and reactor design are necessary. As a solar-specific approach, a direct absorption concept was used in which the receiver and the reformer are the same unit. This is realised by using a reactor with a quartz window as aperture. The concentrated solar radiation passes through this window and is absorbed by a catalytically active absorber system (CAS) which is mounted behind the window. Besides giving catalytic activation, this absorber system has to fulfil the additional functions of solar absorber and heat transfer unit. Ceramic foams, combining high gas permeability and turbulence of flow with a geometry suitable for effective and uniform absorption of solar radiation, are preferably used instead of conventional honeycomb structures.

Reforming of methane with CO_2 in a directly irradiated volumetric receiver–reactor was first demonstrated in the 'catalytically enhanced solar absorption receiver (CAESAR)' experiment conducted by DLR and Sandia National Laboratories, USA in 1990 [2]. Within this project, the process control strategy of adapting the feed flow to the unsteady solar input was demonstrated to be feasible [3]. A second generation solar chemical receiver–reactor was designed and built in 1993–1994 for operation as part of a closed thermochemical storage and transportation loop within a joint project between the Weizmann Institute of Science (WIS) in Israel and DLR [4]. A schematic diagram of the receiver–reactor is shown in Fig. 1. It is designed for a power input of up to 300 kW, being operated at pressures about 3.5 $\text{bars}_{\text{absolute}}$. Reaching reforming temperatures of over 800°C, methane conversions above 80% can be achieved. Operating conditions are controlled by adjusting the receiver mass flow and the feed gas composition by partially recycling the receiver exit gases. In a first test period, the solar chemical receiver–reactor was tested for 100 h under solar conditions. A second test period with a modified CAS insert was conducted, accumulating about the same number of hours of testing [5].

The paper reports on the development of the two catalytically active absorber systems and their performance during solar testing. Stability and degradation aspects are discussed in as far as post-test analysis has been as yet completed.

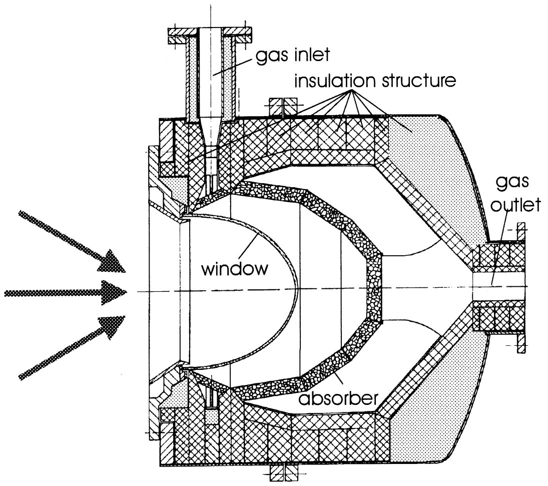

Fig. 1. Schematic of the solar chemical receiver–reactor.

2. Experimental

2.1. Catalytically active absorber system

In contrast to reforming catalysts being used in conventionally heated reactors [6–10] the catalytically active absorber system of a solar-driven volumetric receiver–reactor must be a new, solar specific component. Due to the direct energy input via concentrated solar radiation, it has to fulfil additional requirements as solar absorber. Therefore, its reflectivity has to be as low as possible in order to minimise energy losses by reflection. The system is composed of several components:

- A *ceramic matrix material* is used which must have the following characteristics: good heat transfer behaviour, high mechanical strength, high thermal shock resistance, a geometry suitable for effective and uniform absorption of solar radiation, and high gas permeability together with high turbulence and mixing of the gases as well as low pressure drop. Reticulated ceramic foams have proven to fulfil all these requirements [11].
- A *catalyst support* (or *washcoat*) is applied to increase the surface area and to ensure catalytic activation of the metal catalyst, if a non-oxidic ceramic matrix material is used.
- An *absorber material* is added which darkens the CAS and is responsible for good absorption of the

concentrated radiation. Therefore, its hemispherical reflectivity has to be as low as possible. It has to be temperature resistant and chemically inert towards the other absorber components and the reaction gases.

- An *active catalytic loading* is finally added which must have a sufficient dispersion for high activity and selectivity. Furthermore, this component should show little degradation as a result of temperature effects, coking or other poisoning.

In many cases the catalytic loading is taking over the function of the absorber material as well.

2.2. *Preparation*

The sponge-like, porous ceramic foams are made by filling polymer foams with slurries of ceramic particles. The plastic is then burned off and the ceramic sintered to yield a positive image, with hollow struts, of the original foams. By appropriate cutting of the polymer foam, any desired shape and size of ceramic foam can be fabricated. The megapores of the foam, being 0.4–1.5 mm in diameter, result in an open porosity of 80–95%. The surface being available for convective heat transfer ranges from 500 m^2/m^3 to over 3000 m^2/m^3, depending on the pore size.

There exist two variations for applying the other components: In the first case a white catalyst support is used (γ-Al_2O_3). Afterwards the catalytically active noble metal is applied, which darkens the absorber. A much higher loading than necessary for catalytic activation is then needed in order to achieve good absorption values. In contrast to this a pigmented support is used in the second case. Therefore, the white washcoat material (γ-Al_2O_3) is mixed with a metal oxide pigment, that is stable at high temperatures under reducing conditions. The active metal catalyst is afterwards applied on this pigmented support. As now the active metal only has to serve as a catalyst, a much lower loading is necessary.

The procedures for applying the components are the same, whether a pure Al_2O_3-support or a pigmented support is used. The ceramic matrix is immersed into an aqueous slurry of the support material. The density of this solution has to be kept low enough to prevent a clogging up of the megapores of the foam structure, as this would lead to hot spots during solar operation. After gentle drying at 120°C, the coated structure is calcined and sintered at 1150°C for 8 h. This results in a temperature-stable BET surface area of 25 $m^2/g_{washcoat}$, mainly arising from pores in the range of 150 to 500 nm in diameter. Thereafter the active catalyst is loaded by the method of incipient wetness impregnation, using a metal salt as precursor compound. Table 1 gives an overview on the catalytically active absorber systems that were prepared and investigated.

2.3. *Laboratory equipment*

For the development of both the absorber sets to be used in the receiver–reactor during solar testing, a

Table 1
Overview on the absorber systems investigated

Ceramic matrix	Support/washcoat	Catalyst
α-Al_2O_3 92% Al_2O_3, 8% mullite 20 ppi, 92% porosity	γ-Al_2O_3 30 wt.%[a]	Rh 0.05–90 wt.%[b] Ru 0.05–90 wt.%[b] Pd 0.5–5 wt.%[b]
	γ-Al_2O_3+ metal oxide pigment 10 wt.%[a] (Al_2O_3:pigment =9:1 to 1:1)	Rh 0.05–0.5 wt.%[b]
SiC 97% SiC, 3% Al_2O_3 (with a protective SiO_2 layer 10 ppi, 94% porosity	γ-Al_2O_3 1–20 wt.%[a]	Rh 0.2–20 wt.%[b]
		Ru 0.2–40 wt.%[b]
	γ-Al_2O_3+metal oxide pigment 10 and 17 wt.% (Al_2O_3:pigment=9:1 to 1:1)	Rh 0.05–0.5 wt.%[b]

[a] With respect to the mass of the matrix.

[b] With respect to the mass of the washcoat.

conventionally heated, double walled quartz reactor was used for catalytic investigations, having a length of 465 mm and an o.d. of 40 mm. The reaction feed gases were fed to the outer annulus, where they are preheated. The inner tube of the reactor was equipped with cylindrical absorber test pieces, 28 mm in diameter and 35 mm long. Reactor feed and product gases were analysed by an infrared detector. Catalyst temperatures ranged from 700°C to 1000°C with an operating pressure of 1.1 $\text{bars}_{\text{absolute}}$ and a CO_2:CH_4 ratio of 1.4 in the feed. For the pre- and post-test analysis of the absorber, an isothermal mini-reactor was employed which uses crushed and sieved absorber pieces (1.15–2 mm in diameter) as sample. The reaction feed gases were diluted with nitrogen in a ratio of 1:8 to achieve isothermal conditions and to keep the conversion below equilibrium. A temperature range between 500°C and 700°C was investigated with this set-up. Further characterisation of the catalysts was achieved with BET surface analysis, X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDAX), X-ray photoelectron spectroscopy (XPS) and optical analysis. A pulse chemisorption device is currently being installed to determine the dispersion of the active metal particles.

2.4. Set-up for solar tests

For solar testing, the assembled receiver was installed on top of the tower of the WIS Solar Test Facility Unit in Rehovot, Israel. A flow diagram of the main parts of the test loop is shown in Fig. 2. The methanator system consists of two adiabatic stages and a cooled third stage, the product of each stage being cooled in a water boiler. Due to problems with the methanator part of the cycle, steam had to be injected into the system upstream of the receiver–reactor, resulting in a mixed CO_2/steam reforming for both test periods. During solar testing, the total receiver mass flow, the recycle mass flow, the receiver pressure, the temperature and composition of the feed and exit gases as well as the absorber temperature at various locations were recorded. After the end of the test campaign, the power input of the receiver was measured, using a cooled black target.

3. Results

3.1. Development of the two absorber systems

For the first solar testing period, an absorber system like the one successfully tested in the CAESAR experiment was used. It consisted of an α-Al_2O_3 foam matrix with 20 megapores per inch and 92% porosity, which was coated with approximately 30 wt.% of γ-Al_2O_3 washcoat. Rhodium was used as the catalytically active metal, its loading being optimised with respect to activity and optical aspects. Investigations using the integral reactor set-up showed that a catalyst loading of 2 wt.% with respect to the mass of the washcoat was sufficient for catalytic activation (see Fig. 3). Due to the design of the quartz lab reactor a small bypass between the cylindrical catalyst samples and the inner tube wall was tolerated in sake of the ease of catalyst installation. Therefore, the reforming reaction never reached equilibrium. Nevertheless a higher loading was needed to attain good absorptivity values. As can be seen from Table 2, a catalyst loading of 11 wt.% resulted in the lowest reflectivity. For

Table 2
Hemispherical reflectivity of absorber test pieces loaded with rhodium

Rhodium loading (%) (With respect to the mass of the washcoat)	Hemispherical reflectivity (%)		
	Untested	After 200 h at 1000°C under N_2	After 100 h at 950°C under CO_2 reforming
1.6	13.7	–	–
2.1	9.1	–	–
4.6	4.7	–	–
7.8	3.1	–	–
11	2.2	3.7	2.5
22	3.1	–	–

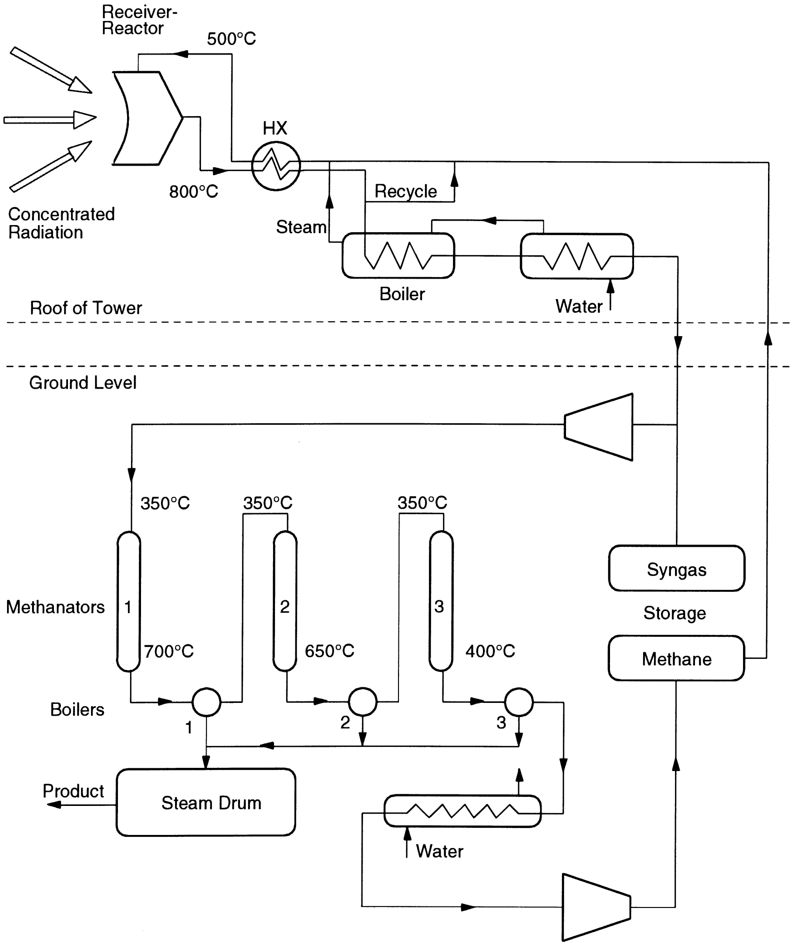

Fig. 2. Flow diagram of the main parts of the reforming test loop at the WIS solar test facility in Rehovot, Israel.

higher loadings, as the dispersion of the metal catalyst decreased and crystals started to be formed, the reflectivity increased once more. The long term temperature influence on catalyst stability was, investigated by keeping sample probes under N_2 for 100 h at 1100°C. Even though the BET surface area and the metal dispersion decreased, the catalytic properties remained unchanged as a result of the high loading. The reflectivity increased from 2.2% to 3.2% (see Table 2). Long-term testing at 900°C under reforming conditions (CO_2:CH_4 =1.4, dilution with N_2 in a ratio of 1:8) did not cause any degradation effects either. A catalyst loading of 11 wt.% was therefore, chosen for use as the first absorber insert.

As the thermal shock resistance of α-Al_2O_3 is not very high, SiC was used as the ceramic material for the foam matrix of the absorber system being tested during the second solar test period. Before further coating, the absorber segments were kept for 100 h at 1400°C in an air atmosphere so that a protective SiO_2 layer could built out on the surface of the struts. To simplify the coating procedure, a matrix with larger megapores was used (10 pores per inch). Another improvement was the reduction of the cost of the catalytic loading. Absorber test pieces coated with cheaper noble metals (Ru, Pd) either did not show the same catalytic activity and stability towards coking as did Rhodium or their reflectivity increased significantly during long-term testing. The use of a pigmented washcoat together with a low Rh loading failed because the metal oxides of the black pigment were reduced by the reaction product gases (H_2 and CO):

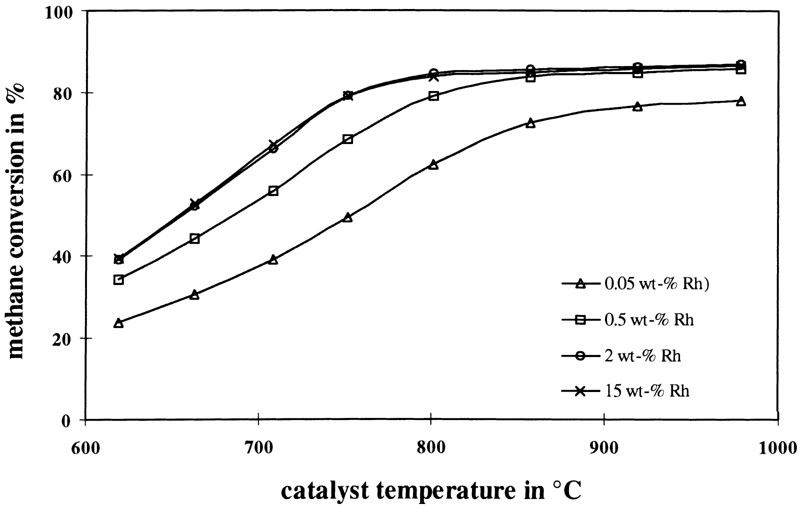

Fig. 3. Methane conversion of cylindric catalyst samples with different Rhodium loading, measured in the quartz tube reactor at 1.1 $\mathrm{bar_{abs}}$ with a CO_2:CH_4 ratio of 1.4 in the feed and a total flow of 1 l/min.

the resulting base metals (Fe, Co, Ni) lead to coke formation. The same washcoat (γ-Al_2O_3) and catalyst (Rh) therefore, had to be used as was used for the first absorber system. Keeping the relative loading of Rh constant (11 wt.%), the minimum amount of γ-Al_2O_3 washcoat necessary for high catalytic activation was determined. It was found that the use of a 10 wt.% washcoat coating reduced the cost of the catalytic loading by two-thirds. Table 3 gives an overview of the absorber systems used for the two solar test periods.

3.2. *Solar testing*

During both solar test periods the catalytically active absorber inserts performed well. During the first test campaign, 100 h of solar testing were accumulated, including 36 h of running the loop with inert gas (CO_2) during start-up and component testing and 64 h of testing under reforming conditions. The maximum power absorbed reached 290 kW. The average absorber temperature ranged from 700°C to 830°C, with a local maximum of 925°C. Methane conversions of 84% were reached, resulting in a CH_4 content of 3.2% in the receiver–reactor product gas. Recycling ratios were between 100% of the receiver exit gases during start-up and 25% during full solar operation [12].

The second test period accounted for 80 h of reforming and 17 h of CO_2 testing. The maximum absorbed power was with 300 kW slightly higher than during the first test period. Methane conversions of

Table 3
Catalytically active absorber systems tested in the solar chemical receiver–reactor

	CAS I	CAS II
Ceramic matrix	α-Al_2O_3 92%, 8% mullite 20 ppi, 92% porosity surface area: 990 m^2/m^3	SiC 97% SiC, 3% Al_2O_3 (protective SiO_2 layer) 10 ppi, 94% porosity surface area: 550 m^2/m^3
Support/washcoat	γ-Al_2O_3 30 wt.% [a]	γ-Al_2O_3 10 wt.% [a]
Catalyst	Rh 11 wt.%[b] 9760 gRh/m^3	Rh 11 wt.%[b] 1930 gRh/m^3

[a] With respect to the mass of the ceramic matrix.

[b] With respect to the mass of the washcoat.

88% were reached, with a maximum of the average absorber temperature of 858°C and a local maximum of 977°C. After the end of the solar tests, the receiver–reactor was shipped to DLR and dismantled for post-test analysis.

3.3. *Pre- and post-test analysis of the absorber systems*

In this section, the Al_2O_3-based absorber of the first test period will be referred to as CAS I and the SiC based absorber of the second test period will be referred to as CAS II. Not all of the post-test analyses of the second absorber insert have yet been finished, so that for some items only the results for CAS I are available.

3.3.1. *Ceramic matrix structure*

After dismantling the receiver–reactor, no major damage to the absorber segments was observed, apart from some cracks in the aluminium oxide absorber due to the low thermal shock resistance of the material. Examination with a light microscope showed that there was no further change or damage to the megapore structure of either of the ceramic matrices.

3.3.2. *Coke deposition*

Almost all the segments of both absorber systems showed the effects of coking, especially on the irradiated front side. The coke was burned off under CO_2 atmosphere at 800°C. The resulting amount of coke depositions ranged from 0.2% of the total weight for segments with little coking to 5 wt.% for segments with intensive coking. The differences arise from the different locations of the segments in the receiver–reactor being subject to a non-uniform flux distribution resulting in temperature differences.

3.3.3. *Optical properties*

The average hemispherical reflectivity of the untested segments was determined to be 2.4% for CAS I and 2.2% for CAS II. After the end of the tests, these values were almost unchanged. Depending on the amount of coke deposited on the structure, the reflectivities were between 1.4% for segments with intensive coking and 3% for segments with little coking.

3.3.4. *Ceramic phase structure*

X-ray diffraction of CAS I did not show any changes in the phase structure of the various components. For CAS II, a reaction of the protective SiO_2 layer of the ceramic matrix and the Al_2O_3 washcoat resulted in the formation of a mullite layer ($3Al_2O_3 \cdot 2SiO_2$). This layer started to built up during catalyst preparation and became more pronounced after solar testing.

3.3.5. *Surface area*

For the untested absorber systems, N_2 absorption isotherms resulted in BET surface areas of 50 $m^2/g_{washcoat}$ and 35 $m^2/g_{washcoat}$ for CAS I and CAS II, respectively. These values were higher than the value of 25 m^2/g measured for the pure γ-Al_2O_3 washcoat substance before the catalytic metal was applied. This increase in surface area is due to the micropore system, that is built by agglomerates of single catalytic metal praticles. After solar testing, the BET surface area of most of the segments of CAS I had decreased, reaching values between 5 and 15 $m^2/g_{washcoat}$. Some segments located close to the gas inlet, however, showed an increase in surface area values of about 60 $m^2/g_{washcoat}$ being found. Porosimetric measurements with mercury showed that the macropore structure, mainly arising from the pore structure of the washcoat, did not change for either absorber system during solar testing. This means, that the washcoat does not show any further sintering during solar testing. This assumption could be proved by ageing washcoat test samples under N_2 atmosphere for 100 h at different temperatures. Sintering started at a temperature of 1200°C, that was never reached during the solar tests.

3.3.6. *Catalyst poisoning*

Deposits of bare metals – mainly Ni and traces of Fe – were found on the irradiated front side of the absorber segments of the CAS I by EDAX. These deposits originate from the tubing system and from the other devices of the loop. As the reaction gases are cycling through the loop, contaminations are enriched with time. A possible mechanism for the deposition of bare metals on the absorber is the metal dusting phenomenon or the dissociation of carbonyl compounds. A white layer being observed on some sections of the outer ring close to the gas inlet could be identified by XPS analysis to be Na species.

3.3.7. Catalytic properties

For these investigations, the following test program was carried out in the isothermal mini-reactor: activation under N_2 at 800°C for 1 h, measurement of the methane conversion between 700°C and 500°C, then removal of the coke deposits with CO_2 at 800°C for 1 h, further measurements of the methane conversion between 700°C and 500°C. The conversion measurements were carried out under CO_2 reforming conditions, with a CO_2:CH_4 ratio of 1.4 in the reactor feed and a ratio of N_2: (CO_2+CH_4) of 8. Catalyst samples were taken from the irradiated front side of the absorber segments. The results of all the segments tested were compared with the values reached with an untested sample of the same material. For CAS I, most segments showed very little degradation (see Fig. 4, segment A2061), their activity being almost the same as before solar testing. However, segments exhibiting intensive coking (e.g. A000) showed a decrease in methane conversion rates, see Fig. 5. In this case, after removal of the coke depositions, the catalytic activity was partly restored. Samples taken from the outer ring (e.g. from segment A3081) which had Na deposits gave low conversion rates, even after coke removal (see Fig. 6). For CAS II, similar effects were observed, although none of the tested segments regained the initial conversion level of the untested spare piece.

4. Discussion

A final evaluation of the second absorber insert is not yet possible, as post-test analysis is still in progress. Only the results of the CAS I will therefore, be discussed in detail. Table 4 gives an overview on the most important properties for selected segments, representing typical behaviours. Segments (e.g. A2061) which were subject to little coking and had good conversion rates after solar testing showed no degradation effects. Small amounts of Na and Fe deposited on the surface of the structure did not affect the catalytic activity. Intensive coking resulted in a decrease of methane conversion rates (e.g. segment A000). This effect was partly reversible following removal of the coke depositions. Irreversible degradation occurred by the deposition of Na (segment A3081). Segments showing these depositions were located on the outer ring, close to the gas inlet.

As no coking was observed for the Rh based absorber systems during any long-term laboratory testing, the specific operating conditions of the Weizmann loop must be responsible for these effects. Due to the problems with the methanator part of the system already mentioned, the reverse methanation reaction

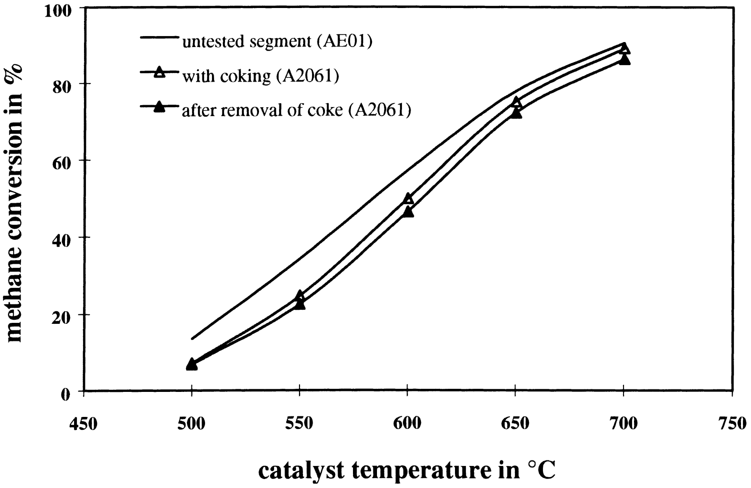

Fig. 4. Methane conversion of a segment with little coking of the CAS I. Results obtained in the mini-reactor of samples of CAS I before and after testing in the solar reactor.

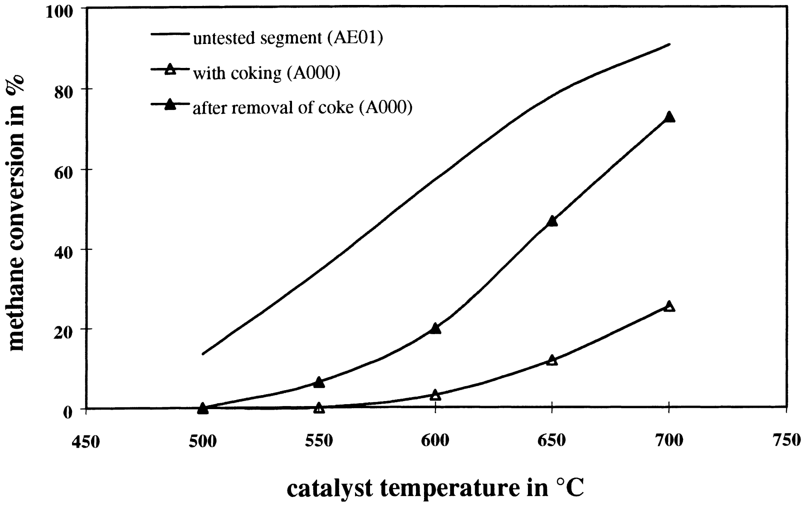

Fig. 5. Methane conversion of a segment of the CAS I exhibiting intensive coking. Results obtained in the mini-reactor of samples of CAS I before and after testing in the solar reactor.

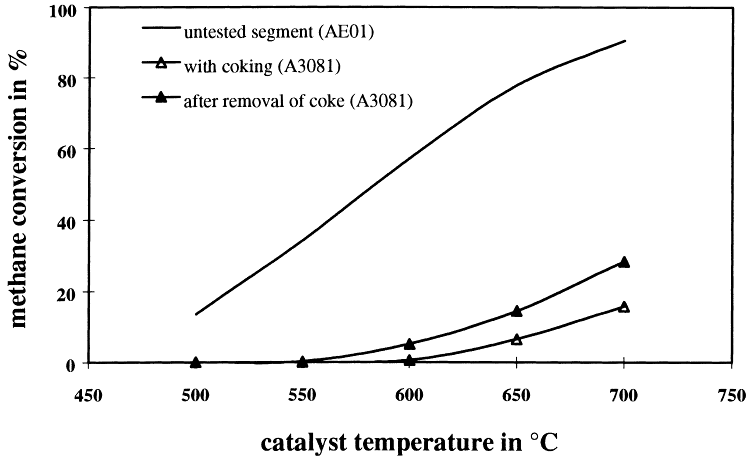

Fig. 6. Methane conversion of a segment of the CAS I exhibiting deposition of Na species. Results obtained in the mini-reactor of samples of CAS I before and after testing in the solar reactor.

Table 4
Results of characterisation of selected segments of the CAS I

Segment No.	Location	Reflection (%)	Coke deposition (wt.%)	Surface area		Other deposits (SEM, XPS)
				N_2-adsorption ($m^2/g_{washcoat}$)	Hg-porosimetry ($m^2/g_{washcoat}$)	
AE01	Untested segment	1.9	–	51	7.1	–
A000	Center disk	1.8	1.43	4	6	Ni, Fe
A2061	Segment of 2nd ring	2.9	0.33	15	5.7	Ni, Fe
A3081	Segment of 3rd ring (close to inlet)	2.1	1.32	60	5.4	Ni, Fe, Na

never achieved equilibrium. As a result a distinct fraction of synthesis gas (H_2 and CO), was always found in the feed gases to the receiver–reactor. A high CO content leads to coking problems, since CO disproportionates to CO_2 and C according to the Boudouard equilibrium. Especially during shut-down of the system, when temperatures in the receiver–reactor fall below 500°C, this mechanism appears to have come into account. Another problem of the high CO concentration in the methanator exit gas is the formation of carbonyl compounds and metal dusting effects. Both mechanisms might be responsible for the deposition of Ni and Fe on the front side of the absorber leading to additional coke formation. Coking by the decomposition of CH_4 can be ruled out due to the specific design of the receiver–reactor, even though local absorber temperatures of over 950°C were reached. The heating of the feed gases is done by convective heat transfer from the directly irradiated catalytically active solar absorber. This means, that as soon as the feed gases reach reaction temperature, the active metal is present to catalyse the reforming reactions and suppress decomposition of CH_4.

Even though both absorber systems showed local degradation effects, the overall performance during solar operation in the receiver–reactor was not affected. It was successfully demonstrated that both systems are suitable as catalytically active absorber inserts in a solar-driven receiver–reactor for CO_2 reforming with low CO_2/CH_4 ratios.

5. Conclusions

Two solar specific absorber systems for pure CO_2 and mixed CO_2/steam reforming were developed and successfully tested in a solar driven volumetric receiver–reactor. As the reforming reaction is performed under energetic aspects rather than under material aspects, a low ratio of $CO_2/(CO_2+H_2O)$ to CH_4 is needed in order to achieve high process efficiencies. Both systems can be operated with ratios below 1.8, which is lower than typically used in industrial reforming processes (2.4–3). Up to an average operating temperature of 850°C both CAS only showed degradation effects, that were due to the specific conditions and problems with the loop system. For solar open- and closed-loop applications these absorber systems have to be further developed in the future to withstand higher operating temperatures and variable feed gas compositions.

The ceramic foams used as matrix structures for the catalytic system proved to be very well suited for solar receiver–reactors, due to their ease of preshaping, their good solar absorption and heat transfer properties as well as their low pressure drop. Apart from the use within solar applications, ceramic foams also have potential to be used in conventional catalytic processes, as their improved heat transfer and high effectiveness factors result in catalyst beds with high conversion rates, being more compact than conventionally used fixed beds.

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