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Fast temperature cycling in microstructure devices

J.J. Brandner[a,*], G. Emig[b], M.A. Liauw[c], K. Schubert[a]

[a] Forschungszentrum Karlsruhe GmbH, Institut für Mikroverfahrenstechnik IMVT, P.O. Box 3640, 76021 Karlsruhe, Germany
[b] Universität Erlangen-Nürnberg, LTC I, Egerlandstrasse 3, 91058 Erlangen, Germany
[c] RWTH Aachen, ITMC, Worringerweg 1, 52074 Aachen, Germany

Abstract

For a couple of years, unsteady state processing has been discussed to be possibly advantageous for chemical process engineering. Specifically periodic changes of reaction parameters are expected to enhance the rates of certain reactions [Chem. Eng. Commun. (1973) 111]. For different reaction systems, concentration cycling has been performed [P.L. Silveston, Composition Modulation of Catalytic Reactors, Gordon and Breach, Amsterdam, 1998]. In contrast, a fast periodic modulation of the reactor temperature, even though potentially beneficial, has long been considered to be beyond reach because of the large thermal masses and the poor surface-to-volume ratio which restrict the heat exchange between conventional reactor systems (e.g. fixed bed reactors) and heating/cooling systems. Recently, microstructure devices for fast temperature cycling have been described [J.J. Brandner, G. Emig, M. Fichtner, M.A. Liauw, K. Schubert, A new microstructure device for fast temperature cycling for chemical reactions, in: M. Matlosz, W. Ehrfeld, J.P. Baselt (Eds.), Proceedings of the Fifth International Conference on Microreaction Technology, IMRET 5, Springer, Berlin, 2001, pp. 164–174; Chem. Eng. Sci. 56 (2001) 1419; Gen. Eng. News 22 (11), 42]. The devices make it possible to obtain a periodic temperature change by 100 K in the second to subsecond range. A method for catalyst integration into microstructure devices will be described briefly. First results on the stability of catalyst carrier layers with respect to fast temperature changes are very promising. The oxidation of carbon monoxide was chosen as model reaction. Under fast temperature cycling conditions, a considerably higher yield compared to the steady state could be observed.

Keywords: Microstructure reactor; Unsteady state; Periodic process; Temperature modulation

1. Introduction

Running chemical reactions in unsteady state mode have been extensively discussed in literature in the last decades. Especially the change of the reactant composition was shown to be useful [1,2,6]. Therefore, the periodic change of reactant composition was subject of many research activities.

Less attention has so far been paid to modulating the temperature, another interesting reaction parameter which has large influence on the reaction behaviour. A theoretical study of possible effects of a fast periodic change of the reaction temperature, which may positively affect certain heterogeneously catalysed reactions heavily depending on adsorption–desorption equilibria, was given in [7]. In this publication, the influence of fast periodic changes of the temperature to the preferential oxidation of CO in a H_2-rich gas mixture was described. The effect postulated to arise by fast periodic changes of the temperature is to generate a residual behaviour of adsorbed reactant molecules within temperature ranges they normally are not adsorbed to the catalyst surface. Therefore, the frequency of the temperature changes has to be high enough to prevent desorption of the reactants, but low enough to allow adsorption and not to run into quasi-steady state by switching the temperature very rapidly.

Beside this so-called PROX-reaction, other heterogeneously catalysed gas phase reactions may benefit from rapid periodic temperature modulations by improving the coverage of a catalyst, and therefore maybe provide a higher yield. Having competitive reactions, fast temperature changes of the catalyst surface may lead to a preferential adsorption of one reactant, while the reaction product may be desorbed more easily compared to the steady state running. In this case, a higher selectivity of the reaction may be possible. Other reactions in which the yield is limited by a heavily adsorbed product may benefit by a burn-off of this product at high temperatures without keeping the high temperature range for a long time and prohibiting the adsorption of reactants.

However, with conventional reactors it seemed not to be possible to achieve a temperature modulation with sufficiently short periods of down to a few seconds or less, which

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* Corresponding author. Tel.: +49-7247-82-3963;
fax: +49-7247-82-3186.
E-mail address: juergen.brandner@imvt.fzk.de (J.J. Brandner).

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Nomenclature

c_P	specific heat capacity
m_{th}	thermal mass
$\dot{Q}$	overall thermal power
Δt	half-cycle time, heating/cooling time
ΔT	temperature difference

we shall call fast temperature cycling (FTC). One reason for this can easily be seen from

$$\dot{Q} = m_{\mathrm{th}} c_P \frac{\Delta T}{\Delta t} \qquad (1)$$

In (1), $\dot{Q}$ denotes the overall thermal power applied to a reactor device, m_{th} is the overall thermal mass of this device, c_P the specific heat capacity of the material the reactor is made of, ΔT the temperature difference and Δt the time to heat up or cool down the reactor device. It is obvious that the thermal mass has to be as small as possible to reach sufficiently short Δt, which is not easy to obtain with conventional reactor devices. Beside this, limitations in heat transfer and small surface-to-volume ratios of conventional devices prohibit reaching high temperature differences in short times and, moreover, to obtain those temperature changes in a periodic manner.

The superior heat transfer capabilities of microstructure devices have been shown [8–10]. Combined with the low thermal mass and the excellent mechanical stability [11], the use of microstructure devices should permit fast reproducible periodic changes of the device temperature, keeping the period time in the range of a few seconds or below. With regard to the proven possibility of catalyst integration into microstructure devices [12], microstructure devices may be excellent tools to obtain FTC.

2. Design of the microstructure devices

Different design approaches for microstructure devices suitable for FTC are possible. An extensive discussion of the different opportunities to obtain fast periodic temperature changes with microstructure devices as well as a full description of specifications, design criteria and manufacturing methods for metallic FTC microstructure devices is given in [13]. In this paper only an abbreviated description shall be shown in the following.

One possibility to obtain temperature changes of a reaction fluid mixtures is by generating a combination of several zones kept at different temperatures [14,15]. In both references, the reaction device was based on silicon with all its known advantages and limitations. The temperature cycling obtained in these experiments for the reactant was reached by a change of its position in the device but not by modulating the temperature of the device itself.

For a periodic change of the temperature of a reaction device, a metallic microstructure device was described having a passage for a reaction gas flow and another passage used for both a heating and a cooling liquid in an alternating manner [4]. The temperature changes are reached by switching the liquid passage from a hot to a cool fluid loop and back, leading to a temperature change of the reactor device itself. The microstructure device was manufactured of single microstructure foils made of stainless steel which had been structured by wet chemical etching, stacked and clamped together.

Another design was presented in [3]. Here, the microstructure device was specifically designed to combine high heating and cooling capability, a large reaction surface area and a considerably small pressure drop within the reaction passage with a small thermal mass as well as stability against high temperatures and pressure up to about 2.0 MPa. Moreover, manufacturing process limitations like the maximum channel width, bonding process limitations as well as the possibility of integrating catalytically active materials had to be considered.

To improve the steepness of the temperature changes, cooling and heating was done in different manner. While the heating was performed by high-power resistor cartridges, a liquid cooling was used. This combination was chosen to avoid the generation of a mixed temperature between two fluids kept on different temperatures and therefore improving the heating and cooling power at the same time.

The device consists of a small central heater plate with six holes to insert high-power resistor heater cartridges and four grooves to insert thermocouples. On both sides of this central heater plate, six microstructure foils for a cooling passage and four microstructure foils for a reaction passage are arranged in alternating order. All these parts are made of stainless steel. While the central heater plate has been structured mechanically, the microstructure foils have been obtained by wet chemical etching. The stack was completed by a top plate and a base plate. It was assembled by a diffusion bonding process. Adaptation for fluids was realized with standard fittings.

The described device (so-called Type 1 FTC-device) was continuously heated with electrical power and periodically cooled by a fluid stream. Electrical heating was realized using six high-power resistor cartridges providing a maximum power of 1050 W each. Excellent heat transfer results and good, reproducible controllability with a similar microstructure device used for heating and evaporation of liquids have been described [9]. The device is, without fittings, 44 mm long, 33 mm wide and 12 mm high. The thermal mass including the adapter fittings is about 120 g. A photo of such a device is shown in Fig. 1.

The design of the Type 1 FTC-device could be improved by changing the microstructure shape and dividing the single heater plate into three plates controlled independently. This redesign led to Type 2 FTC-device which is shown in Fig. 2. This Type 2 FTC-device has the same external dimensions

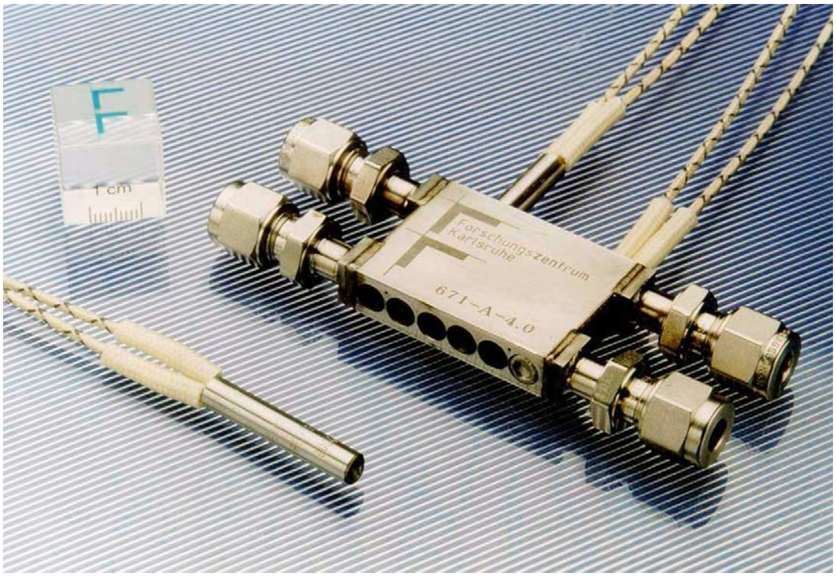

Fig. 1. Photo of a Type 1 FTC-device with high-power resistor heater cartridges [3,13].

as Type 1 but is 17 mm high and has a thermal mass of about 240 g [16].

In Type 1 devices, the channel length was around 810 mm, while it was about 760 mm for Type 2 devices. The channel width was set to 350 μm at a depth of about 150 μm for both device types. The channels of the cooling passage were 350 μm wide, about 150 μm deep and of different length. For Type 1 devices, the channels have been arranged to meet a nearly crossflow design to the reaction passage. The microstructure used for Type 2 devices was changed in shape to an ideal crossflow design to reduce the pressure drop in the cooling passage.

3. Test facility

To obtain periodic temperature changes, a computer controlled test facility was built. For measurement and control of the test facility, a specifically designed control program was developed. A full description can be found in [13].

In the control program, the duration of the heating and the cooling half-cycle as well as waiting times, mass flow of the reaction gases, mass flow of the cooling fluid and the applied electrical power could be controlled independently of each other. Therefore, it was possible to split the heating and cooling half-cycle to different lengths, obtaining, e.g.

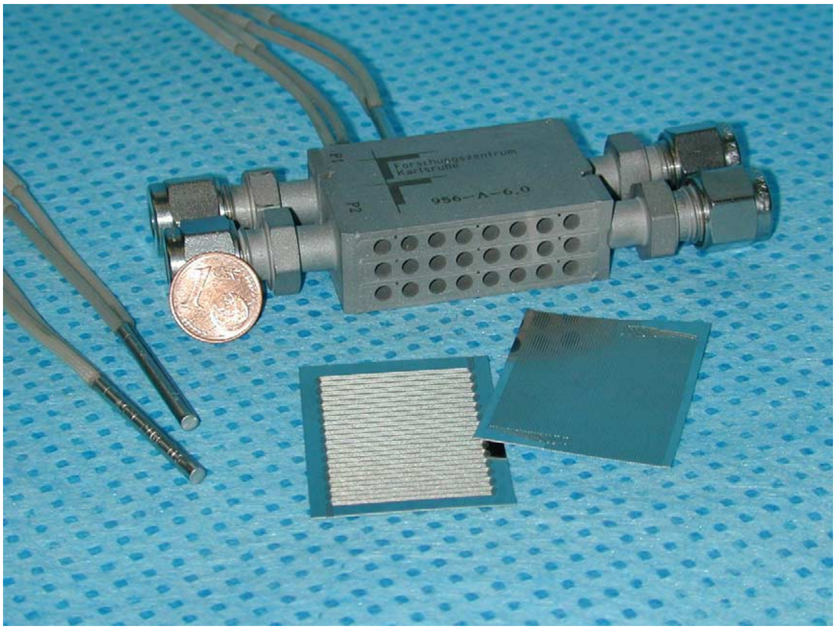

Fig. 2. Type 2 FTC-device with heater cartridges and microstructure foils compared in size to an Eurocent coin [13].

long heating times with slow temperature changes and short cooling times with fast temperature changes, vice versa or equal cooling and heating times (time controlled program mode). As a second opportunity, a control system based on preselected temperature limits (i.e. an upper and a lower temperature limits) was integrated (temperature controlled program mode). The temperature limitation automatically leads to a split of the cycle period into different heating and cooling half-cycle times. The minimum runtime duration was limited by the data acquisition hardware to around 0.3 s.

A computer controlled bypass-to-cooling-passage valve combination was integrated into the test facility. The pressurized-air-driven magnetic valves are controlled with precise timing independently of each other. For the heating half-cycle, a bypass around the FTC-device was opened 10 ms before the cooling passage through the device was closed. For the cooling half-cycle, the cooling passage was opened 10 ms before the bypass was closed. As cooling fluid, deionized water was used, provided by a pressure pulse reduced multi-membrane pump at a pressure of 1.2 MPa. The applied mass flow was adjusted up to 45 $\mathrm{kg\,h^{-1}}$, depending on the desired temperature change. A mass flow of 28 $\mathrm{kg\,h^{-1}}$ was found to be sufficient for most experiments.

To obtain sharper temperature changes, especially in the heating half-cycle, a third valve was integrated into the test facility which was opened 10 ms after the cooling passage valve was closed. The opening duration could be adjusted by the control program. This valve was used to apply a short high-pressure pulse of air to clear the cooling passage from remaining water and therefore reduce the overall thermal mass of the microstructure device.

Due to the specific structure of the control program, the maximum delay time of a switch process between a cooling half-cycle and a heating half-cycle is in the same range than the minimum runtime duration. This led to a maximum control uncertainty of one minimum runtime duration for the half-cycle time, leading to slightly different temperatures in each cycle at the upper and lower switching point for both cases, the time controlled and the temperature controlled program mode.

For the experiments on thermal behaviour, the reaction gas was replaced by a continuous N_2 flow of 200 $\mathrm{ml\,min^{-1}}$ (STP), leading to a pressure drop of around 0.1 MPa in the gas passage.

4. Results

4.1. Results on thermal tests

First thermal tests have been done with a Type 1 FTC-device to obtain experimental data on the overall thermal behaviour and the performance of the device. At a full cycle time of around 60 s and an electrical power of 420 W, a half-cycle temperature difference of 193 K could be measured. At a full cycle time of only about 5 s, still a temperature difference of 100 K could be observed within each half cycle. Here, an electrical power of 1675 W was applied. During the cooling half-cycle, a thermal power of 1550 W was drained. Therefore, the heat transfer efficiency is around 93%.

The obtained temperature gradients in time are quite high compared to those reported for other devices for FTC. Other presented microstructure devices were able to reach temperature gradients of 0.2 or 2 $\mathrm{K\,s^{-1}}$, respectively [4,17]. The device presented here is able to manage a temperature gradient of more than 40 $\mathrm{K\,s^{-1}}$, which gives enough room for real high frequency cycling of the temperature with large temperature differences.

However, the temperature profile generated on the FTC-device was not fully homogeneous. During the heating half-cycle, a temperature gradient of about 20 K between the centre and the outer parts of the device have been determined using online IR thermography combined with measurements of thermocouples integrated into the reactor itself. This temperature gradient could not be observed during the cooling half-cycle. Here, a temperature difference between inlet and outlet of the cooling fluid of only 5 K could be observed.

In Figs. 3 and 4, the temperature of the reactor device, measured close to the cooling fluid inlet, and the temperature of the N_2 gas flow, measured roughly 1 mm behind the gas outlet in the centre of the fluid stream, are displayed. While the data in Fig. 3 have been obtained at a cycle time of 60 s, Fig. 4 shows the results obtained at a cycle time of around 5 s. The results presented here are exemplary for the potential of the devices to provide high temperature differences in a periodic manner, combined with a good reproducibility and controllability of the temperature.

A remarkable side effect was observed during the cooling half-cycles with short time periods: the gas temperature at the outlet could not follow the fast temperature changes of the device. The reason for this behaviour was examined with an IR thermographic system. It was found that the inlet and outlet region of the reaction passage of the FTC-device were cooled by heat dissipation only, leading to a certain delay time. Those areas were acting as temperature low pass filters, leading to a nearly constant mean temperature of the gas flow at short cycle times, which can be seen in Fig. 4.

4.2. Catalyst layer integration

There are several possibilities to integrate catalyst carrier layers into microstructure devices. A possibility to obtain Al_2O_3 layers inside the microchannels of ready-to-use assembled microstructure devices was shown in [18]. With devices made of Al, an anodic oxidation step could be used to generate a porous alumina layer inside the channels. A sol–gel method was described in [12]. This method is also applicable on microstructure devices not made of aluminium. It was used to generate an alumina layer on microstructured foils for the reaction passage of a Type 1

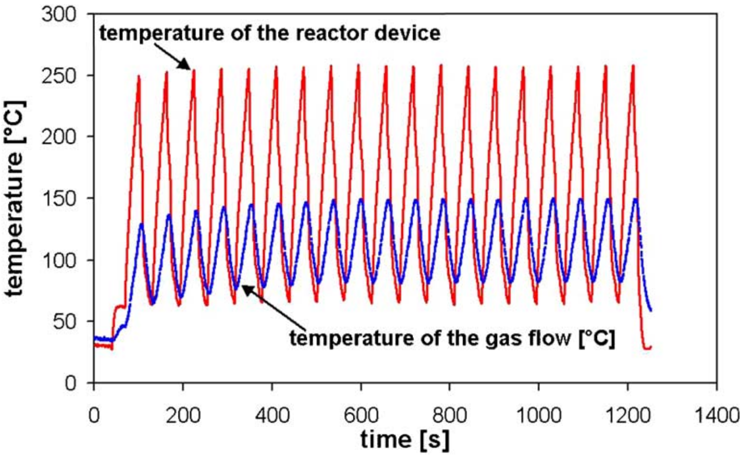

Fig. 3. Temperatures of the reactor device and the N_2 gas flow at a cycle time of 60 s. A constant electrical power of about 400 W was applied. A flow of 15 kg/h deionized water was used for periodic cooling. A delay time between the temperature changes of the FTC-device and the gas temperature could be observed.

FTC-device. In Fig. 5, a SEM photo of a single microstructured stainless steel foil covered with an alumina layer based on sol–gel technique is shown. In Fig. 6, a SEM photo of some microchannels inside of a completely manufactured microstructure device covered with an alumina layer, generated by sol–gel technique, is shown. The alumina layer is several μm thick and quite homogeneous, covering all sidewalls of the microchannels. The thickness of this layer can be adjusted below 20 μm.

A surface enhancement factor could be measured by physical adsorption method (BET) with Krypton as adsorbing gas. The enhancement factor, defined as the ratio of the surface of the porous system to the geometrical surface of the microstructured foil, was measured to be $100\,m^2\,m^{-2}$ [12].

Several experiments have been conducted to obtain data on the stability of the alumina layers with regard to fast temperature changes. At a period time of 10 s, several thousand temperature changes of about 80 K have been applied to an alumina-coated Type 1 FTC-device. The mass of the device was measured before and after the experiments. Moreover, a metal sinter filter (mesh width: 5 μm) was integrated into the gas flow behind the device to detect alumina particles which may have left the device. The maximum throughput of the device before and after the cycling experiments was measured. No detrimental effect of the temperature changes on the alumina layer has been observed so far.

The Al_2O_3 layer can be used to deposit catalytically active material by, e.g., wet impregnation. A Pt catalyst was incorporated from an aqueous solution of platinum nitrate (59.15 mass% of Pt). The porous system was impregnated four times, then dried at 70 °C for 24 h. After drying, the catalyst was calcined at 500 °C for 5 h, followed by a re-

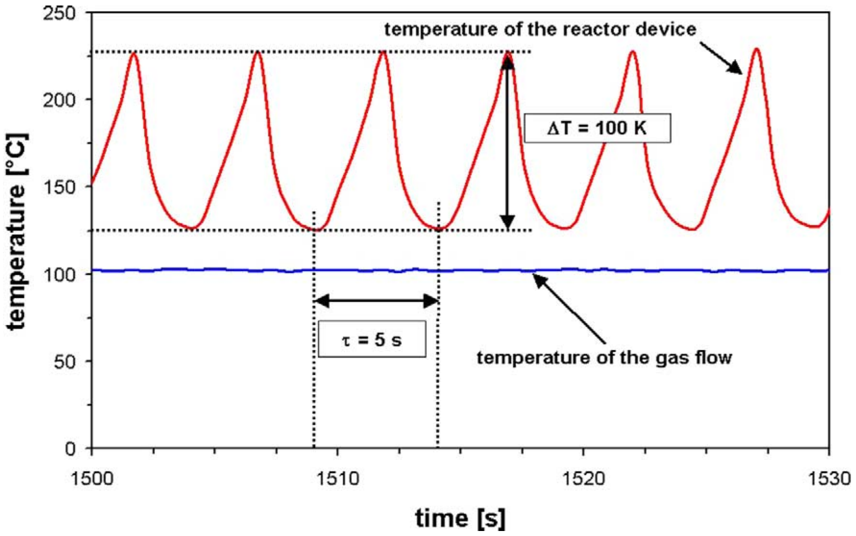

Fig. 4. Temperatures of the reactor device and the N_2 gas flow at a cycle time of 5 s. A constant electrical power of about 1700 W was applied. A flow of $28\,kg\,h^{-1}$ deionized water was used for periodic cooling. Because of the border areas of the gas passage inlet and outlet of the FTC-device are acting as temperature low pass filters, the gas temperature remains quite constant.

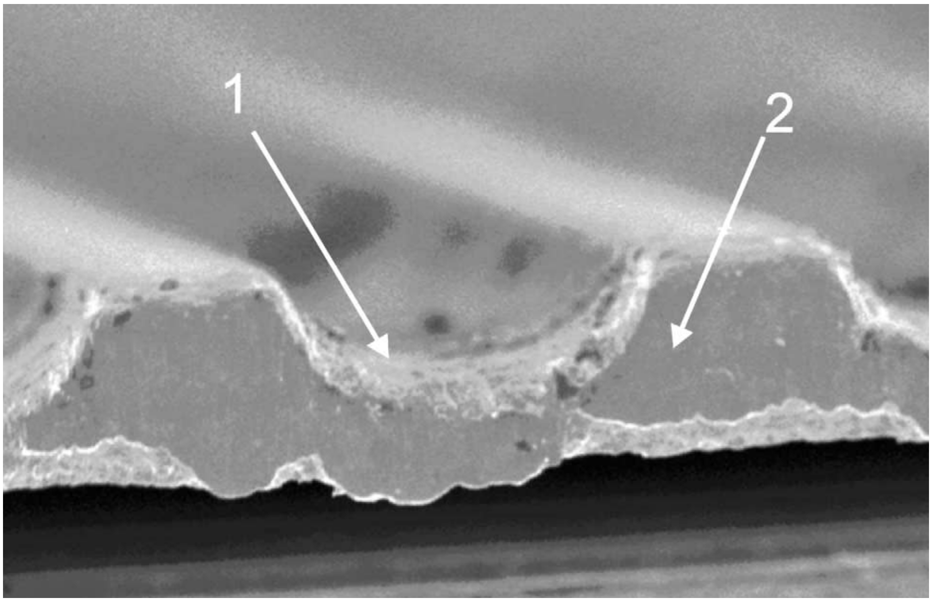

Fig. 5. SEM of a single microstructure stainless steel foil, covered with an alumina layer generated by sol–gel technique: (1) Al_2O_3 layer; (2) stainless steel foil.

duction step. Reduction was done for 2 h with a 0.5 l min^{-1} (STP) flow of Ar, containing 0.5 vol.% of H_2 at 500 °C. The Pt catalyst system has been tested in steady state mode. Some other examples of reactions have been done in microstructure devices with catalyst-carrying layers obtained by sol–gel preparation. High performance of a catalyst applied by sol–gel alumina was demonstrated before [19,20].

4.3. First reaction results

For the very first proof of possible effects of FTC to heterogeneously catalysed gas phase reactions, a simple test reaction, the oxidation of carbon monoxide on a platinum catalyst, was chosen. The reaction is well known, easy to run, and the reactants and products can easily be evaluated by spectroscopic methods. In the case of the CO oxidation, the reason for a possibly increased reaction yield might be a residual catalyst coverage behaviour of adsorbed CO and O_2, as it is described in Section 1. A significant higher yield was postulated, which may be advantageous for, e.g., fuel cell applications [7].

To prove the postulation, a Type 1 FTC-device was tested in steady state mode and unsteady state mode. The temperature limits have been set to $50\,^{\circ}C < T < 150\,^{\circ}C$. The

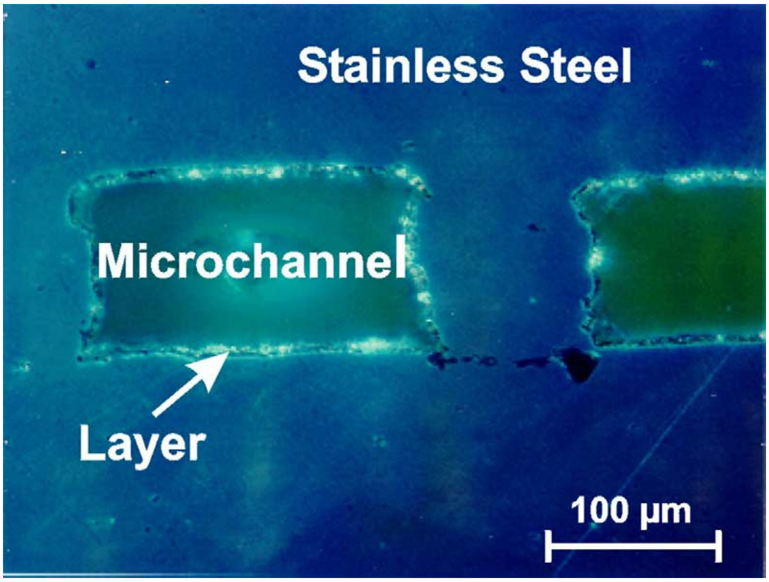

Fig. 6. Photo of a stainless steel microstructure device. The inner side of the microchannels is covered with an alumina layer obtained by sol–gel deposition. The layer is several micrometers thick.

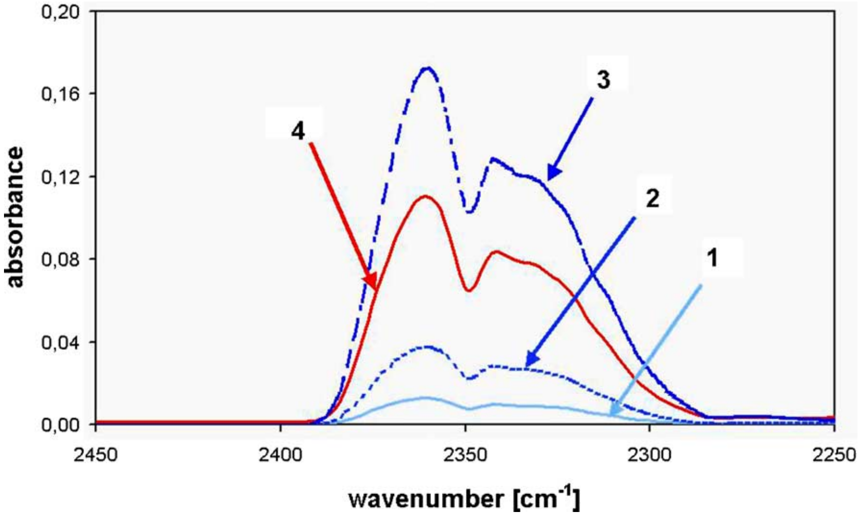

Fig. 7. Absorbance in the wavenumber range around the characteristic CO_2 peak at 2361 cm^{-1}, measured by online FTIR spectroscopy. The lines numbered 1, 2 and 3 denote the mean value of absorbance measured in steady state mode at 50, 100 and 150 °C. Line 4 denotes the absorbance for unsteady state mode in the same temperature range, obtained with a time period of 10 s. The absorbance shown in this graph was calculated by averaging more than 11,000 FTIR spectra. For this experiment, the mean temperature of the unsteady state mode was about 83 °C.

test gas consisted of 20 ml min^{-1} (STP) CO, 80 ml min^{-1} (STP) O_2 and 200 ml min^{-1} (STP) N_2. To determine the generated CO_2, online FTIR spectrometry was used. The absorbance of a characteristic CO_2 peak at 2361 cm^{-1} was measured, which is directly proportional to the reaction conversion.

In Fig. 7, the mean absorbance obtained by experiments in steady state mode (50, 100 and 150 °C) as well as the mean absorbance measured in an experiment under fast temperature cycling conditions are shown. Experiments with period times between 5 and 40 s have been performed, leading to comparable results for all experiments. For the exemplary graph shown in Fig. 7, the period time for the FTC experiment was set to 10 s, the temperature range was approximately $50\,^{\circ}C < T < 150\,^{\circ}C$. No optimisation of the FTC frequency was done so far, but the absorbance measured at a period time of 10 s gives a good idea about the effect of FTC to the CO oxidation. Due to the asymmetric split of the length of heating and cooling half cycle times, the mean temperature for this experiment was calculated to 83 °C. By averaging the measured absorbance of more than 11000 FTIR spectra, a significant higher yield of CO_2 compared to the steady state mode experiments was observed. To obtain the same absorbance value in steady state mode, a temperature above 100 °C would probably be necessary.

5. Conclusions

Two different types of microstructure devices suitable for fast temperature cycling have been presented. The devices were made of stainless steel and assembled by known bonding techniques. They were constantly heated with high-power resistor heaters and periodically cooled with deionized water. With these devices, periodic temperature changes of the complete device including an integrated catalyst carrier layer of several tens of Kelvin in a time range of seconds were possible and reproducible. A computer controlled test facility made it possible to control the heating and the cooling half cycle time independently of each other. All other process parameters were controlled by this program.

The catalyst support layer was integrated by a sol–gel technique. The stability of this layer with regard to the fast temperature changes was tested. No effect of fast temperature cycling on the alumina layer could be observed so far.

First reaction experiments have been conducted, using the oxidation of CO to CO_2 on a platinum catalyst. Online FTIR spectrometry was used to analyse the concentration of the CO_2 generated. A significant increase of the conversion of CO to CO_2 could be observed.

In the future, more experiments on the steady state and the unsteady state behaviour of several heterogeneously catalysed gas phase reactions will be done. It is expected that the fast periodic change of the catalyst temperature will lead to significant changes in the reaction rates and, especially, in the selectivity of several reactions. Beside this, applications in chemical analysis, biomedical research [5] and material research are considered.

Acknowledgements

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