



# Dynamics in environmental catalysis: from surface coverages to reactor operation

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## **Dynamics due to varying inlet conditions**

### **Cold start-up of behavior of a Three-Way-Catalyst**





## Dynamics due to varying inlet conditions Cold start-up of behavior of a Three-Way-Catalyst







Tischer et al. SAE 2007-01-1072

Simulation by DETCHEM<sup>MONOLITH</sup>

Appl. Catal. B: Env. 156–157 (2014) 153.

# Dynamics due to varying inlet conditions Removal of NO<sub>x</sub> emissions by NH<sub>3</sub>-SCR



Bottom

Preparation of the reducing agent NH<sub>3</sub> by thermolysis and hydrolysis of urea-water solution (AdBlue, DEF)



Very transient preparation of  $NH_3$  due to pulsing spray, film and deposit formation as well as interaction with varying exhaust gas

Prog. Energy Combst. Sci. 87 (2021) 100949



DC: 0.9

Top

t = 3.06 ms

Cortesy of Robert Bosch GmbH

# **Dynamics due to varying inlet conditions**

# Aftertreatment system causes temporal and spatial fluctuation



Chem. Eng. Sci. 246 (2021) 116876.





Prog. Energy Combst. Sci. 87 (2021) 100949.

- Pulsing, start and stop of UWS spraying lead to spatially and temporally fluctuating NH<sub>3</sub> and HNCO profiles
- Varying inlet conditions to the catalyst may affect SCR performance and NO<sub>x</sub> removal, even lead to particles





## From surface coverages to reactor operation

- Dynamic operation often inherent to catalytic reactors due to varying
  - inlet conditions
  - short- and long term catalyst aging
  - catalyst structure and morphology changes





Appl. Catal. B: Env. 340 (2024) 123241.

- Dynamic operation also great potential for boosting activity by
  - optimized operating conditions
  - reactivation of catalysts
  - control of oxidation states
  - control of surface coverages



Catalysts 11 (2021) 300



real-world operatio



1. Emission control of local pollutants



2. Reduction of GHG emissions











### 3. Circular energy economies





1. Emission control of local pollutants





- 2. Reduction of GHG emissions
- 3. Circular energy economies

- Three-way catalyst
- NO reduction by CO over Pt/Al<sub>2</sub>O<sub>3</sub>
  - Dynamics in surface coverages
- Lambda-dithering at low temperatures
  - Forced dynamic operation



# Emission control by Three-Way Catalysts - NO reduction by CO

## Space- and time-resolved mapping of activity by laser spectroscopy

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# Emission control by Three-Way Catalysts - NO reduction by CO What causes activity decline at stoichiometric NO – CO conditions?



800.0 ppm CO



- Formation of isocyanate (NCO) already known from DRIFTS

#### Packed bed measurements

400.0 ppm CO

200.0 ppm CO



#### T. Häber et al., 2025

# Emission control by Three-Way Catalysts - NO reduction by CO Surface coverage studied by time-resolved DRIFTS



Simultaneous measurements of time-resolved DRIFTS and integral conversion using FTIR



- NCO absorption always increases over time
- Amount of NCO depends on inlet CO concentration but formation rate mostly governed by temperature (not CO)

#### Pt–CO

Appears within a few seconds & remains almost constant

Both, the formation <u>and</u> the decomposition of  $Al_2O_3$ -NCO occur via the noble metal

initially no Pt-CO

 $\tilde{\nu}$  [cm<sup>-1</sup>]

*x<sub>j</sub>* [ppm]



1. Emission control of local pollutants





- 2. Reduction of GHG emissions
- 3. Circular energy economies

- Three-way catalyst
- NO reduction by CO over Pt/Al<sub>2</sub>O<sub>3</sub>
  - Dynamics in surface coverages
- Lambda-dithering at low temperatures
  - Forced dynamic operation

# Forced dynamic operation by lambda-dithering of TWC Enhancement of low-temperature performance



# Hybrid electric vehicles

- Gasoline-fueled IC engine
- TWC; stoichiometric conditions
- Lower exhaust temperature
- Frequent cold start-ups
- Rapid light-off requested

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Compact designs needed





# Can we overcome the rate limitations due to high surface coverages?



Appl. Catal. B: Environ. 345 (2024) 123657

# Forced dynamic operation by lambda-dithering of TWC A wide variety of process parameters can be considered





# Forced dynamic operation by lambda-dithering of TWC Significant increase of catalyst activity at low temperature





#### Influence of split cycle



Appl. Catal. B: Environ. 345 (2024) 123657

# Forced dynamic operation by lambda-dithering of TWC Significant increase of catalyst activity at low temperature



#### **Operating conditions**

**Temperature** influences the optimal frequency

**Gas velocity** influences the optimal frequency

Gas mixing is influenced by the **geometry** of the exhaust gas line

#### **Dithering parameter**

**Amplitude** increases maximal conversion and is influenced by frequency

#### Asymmetric periodic operation

- Mechanistic insight on periodic operation
- Trade-off between conversion of different species

#### Activity boosting through surface coverage effects



Appl. Catal. B: Environ. 345 (2024) 123657 Chem. Eng. J. 499 (2024) 155852.

 $\bigvee$ 

- 1. Emission control of local pollutants
- 2. Reduction of GHG emissions









## 3. Circular energy economies



- Oxidation of CH<sub>4</sub> over PGM catalysts
  - Dynamics in coverage and oxidation state
- Avoiding CH<sub>4</sub> slip by oxidation catalysis
  - Forced dynamic operation for catalyst reactivation
- Carbon capture by autocatalysis of carbon
  - Dynamics of the reactor characteristics
- Methanol formation in a two-component system
  - Dynamics in inlet and temperature leads to product

# Start-up of reactor for catalytic partial oxidation of natural gas Dynamics of surface coverage and catalyst oxidation state

- CPOx of CH<sub>4</sub> over Rh-based catalyst at stoichiometric conditions
- Fundamental understanding due to safety concerns

 $CH_4 + \frac{1}{2}O_2 \rightarrow 2H_2 + CO$   $CH_4 + 2O_2 \rightarrow 2H_2O + CO_2$ 









XAS-based Rh-oxidation state

J.-D. Grunwaldt et al., JPC C, 113 (2009) 3037.



**Temperature-dependent catalyst oxidation state** 

- Methane slip can ruin better CO<sub>2</sub>-power-balance of natural gas vs. oil
- Challenges in low-temperature catalytic conversion

 $CH_4 + 2 O_2 \rightarrow 2 H_2O + CO_2$ 

Hysteresis behavior of conversion related oxidation state

Exp.: Groppi, Lietti, Forzatti, Stud. Surf. Sci. Catal. 130 (2000) 3801.

J. Catal. 370 (2019) 152.



20

**Total oxidation of GHG methane over Pd-based catalysts** 





#### DFT: H. Grönbeck et al., J. Am. Chem. Soc. 137 (2015) 12035.

# Total oxidation of GHG methane over Pd-based catalysts

**Temperature-dependent catalyst oxidation state** 

- Methane slip can ruin better CO<sub>2</sub>-power-balance of natural gas vs. oil
- Challenges in low-temperature catalytic conversion

 $CH_4 + 2 O_2 \rightarrow 2 H_2O + CO_2$ 

DFT as basis for the development of reaction mechanisms



## **Total oxidation of GHG methane over Pd-based catalysts**

**Temperature-dependent catalyst oxidation state** 

 $CH_4 + 2 O_2 \rightarrow 2 H_2O + CO_2$ 

**Surface reaction mechanism** 



- 1. Emission control of local pollutants
- 2. Reduction of GHG emissions









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#### 100 100 100Phase content [%] PdC 1.0 80 conversion / a.u. • () CH<sub>4</sub> conversion / % 75 ●Pd 0.8 50 0.6 CH₄ 20 Pd/CeO 0.4 Pd-Pt/CeO<sub>2</sub>-ZrO<sub>2</sub>-10 20 30 50 0 1000 2000 3000 100 200 300 **400** 500 4000 5000 Temperature / °C Time [min] time on stream / s Ind. Eng. Chem. Res. 58 (2019) 12561. K.A. Karinshak, PL, M.P. Harold, OD, ChemCatChem 12 (2020) 3712. T. Franken, O. Kröcher et al., ACS Catal. 11 (2021) 4870.

**Total oxidation of GHG methane over Pd-based catalysts** 

## Forced dynamic operation for catalyst reactivation

- Lean-operated NG-engines: effective way for improving fuel efficiency and reducing NO<sub>x</sub> emissions
- Methane slip requires exhaust-gas after-treatment by oxidation catalysts, which loose activity rather quickly under steady operation
- In situ re-activation of catalyst by short rich pulses

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XAS monitors transient electronic state of catalyst during dynamic operation 

### $CH_4 + 2 O_2 \rightarrow 2 H_2O + CO_2$









# Controlling methane slip by catalytic total oxidation over Pd: Forced dynamic reactor operation induces gradients in Pd/CeO<sub>2</sub>





#### Evolution of axial gradients induced by forced dynamic operation

- Spatial profiling (SpaciPro) uncovers two zones
- X-ray photoelectron spectroscopy (XPS) uncovers electronic state variations

26-28 -

28-30

0.00

8-10. 0-12.

ဖစ

2-2 4-2 14-16 16-18 18-20 20-22 22-24 22-24 24-26

Axial section (mm)

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-14

# Feed modulation for coupled methane and NO<sub>x</sub> conversion Forced dynamic operation boosts catalytic activity



#### PGM+Spinel

PGM / Al <sub>2</sub> O <sub>3</sub> (Pt:Pd :: 19:1, 30g/ft <sup>3</sup> )				
Spinel / Al <sub>2</sub> O <sub>3</sub> (25wt.% Mn <sub>0.5</sub> Fe <sub>2.5</sub> O <sub>4</sub> )				
Cordierite				

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#### **PGM-Only**

PGM / Al <sub>2</sub> O <sub>3</sub> (Pt:Pd :: 19:1, 30g/ft <sup>3</sup> )
Alumina
Cordierite

#### PGM+Spinel (Single Layer)



#### PGM-Only (Single Layer)





- Dual-layer catalyst outperforms single-layer samples
- Close proximity of the PGM and MFO functions
- Mixed-layer catalyst lowers the CH<sub>4</sub> conversion at high temperature
- Separation of PGM and spinel layers with Al<sub>2</sub>O<sub>3</sub> intermediate layer does not
- Pretreatment matters; modulation increases activity



K. Karinshak, P.W. Chen, R.F. Liu, M. P. Harold. Appl. Catal. B. Environ. 304 (2021) 120607.

# Feed modulation for coupled methane and $NO_x$ conversion Forced dynamic operation boosts catalytic activity

 $GHSV = 90k hr^{-1}$ 

40 °C/min

f = 1 Hz

 $<\lambda> = 0.996$ 

A =  $2\Delta\lambda$  varied

200

100%

**6** 80%

**Conversio** 40%

**Н**2 20%

0%

100

b) PGM+Spinel



reeds composition with and without modulated reed condition.					
	Full Feed w/o modulation $\lambda = 0.992$	Full Feed w/ modulation $\langle \lambda \rangle = 0.992$ (0.978–1.006 at varying Hz)	CDTI Feed w/ o modulation $\lambda = 0.996$	CDTI Feed w/ modulation $\langle \lambda \rangle = 0.996$ (0.982–1.009 at 1 Hz)	
CH <sub>4</sub> CO H <sub>2</sub> NO CO <sub>2</sub> H <sub>2</sub> O O <sub>2</sub>	1500 ppm 8000 ppm 1000 ppm 1000 ppm 10 % 10 % 5650 ppm	1500 ppm 8000 ppm 1000 ppm 1000 ppm 10 % 10 % 7900 ppm / 3400	1500 ppm 8000 ppm 2000 ppm 1000 ppm 10 % 10 % 6900 ppm	1500 ppm 8000 ppm 2000 ppm 1000 ppm 10 % 10 % 8900 ppm/ 4500	
		ppm		ppm	

composition with and without modulated food condition

- Inlet lambda modulations increase CH<sub>4</sub> and NO conversion
- Impact of varying oscillation amplitude on performance

-A=0.000

-A=0.00350

-A=0.0150

-A=0.014

-A=0.007

-A=0.021

-A=0.028

-A=0.042

600

-A=0.056

700

- Linked to O<sub>2</sub> concentration
- Transient activity spikes







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300

400

Inlet Temperature (°C)

500

- 1. Emission control of local pollutants
- 2. Reduction of GHG emissions









### 3. Circular energy economies



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# High-temperature pyrolysis of hydrocarbon streams: Technology for hydrogen production and carbon capture



- Idea: Removing carbon from hydrocarbon streams (CCU and CCS)
- Sources
  - Fossil fuels such as natural gas
    - $CH_4 \rightarrow H_2 + C(s)$ and hydrocarbon feedstocks
    - $C_xH_y \rightarrow y/_2H_2 + x C(s)$
  - Biogas
    - $2 \text{ CH}_4 + \text{CO}_2 \rightarrow 4 \text{ H}_2 + 2 \text{ CO} + \text{C(s)}$
  - Plastics, waste
    - $(C_xH_y)_n \rightarrow n \frac{y}{2}H_2 + n \times C(s)$
- Products
  - Hydrogen for energy and chemistry
  - Synthesis gas for chemistry
  - Carbon for electrodes, composite materials, tires, fertilizers

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Carbon sequestration



ChemSusChem 16 (2022) e202201720.

# Pyrolysis of natural gas for $H_2$ production and carbon capture

## Autocatalytic process over carbon

- $CH_4 \rightarrow 2 H_2 + C(s)$
- Carbon deposition on carbon particles
- Moving bed of carbon particles
- Recirculation of growing carbon
- 1200-1600°C, 1-10 bar



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Chem. Eng. J. 485 (2024) 149684.



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# Pyrolysis of natural gas for H<sub>2</sub> production and carbon capture Complex dynamics on micro and macroscale





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- Gas-phase reactions in and outside carbon bed <sup>[1]</sup>
- Deposition of smaller hydrocarbons and aromatics <sup>[2]</sup>
- Molecular growth processes and formation of soot particles <sup>[3]</sup>
- Deposition of soot on/between carbon particles and on reactor walls <sup>[4]</sup>
- Spatial and temporal variation of external and internal porosity
- Flow through bed, external and internal diffusion
- Heat balances

J. Appel, H. Bockhorn, M. Frenklach, Combust. Flame 121 (2000) 122; Chem. Eng. J. 479 (2024) 147556.
 Chem. Eng. Sci. 62 (2007) 4976.
 M. Frenklach, H. Wang, Proc. Combust. Inst. 23 (1990) 1559; J. Phys. Chem. A 127 (2023) 2136.
 Carbon (2024) 231 (2025) 119689.

# Pyrolysis of natural gas: Interplay of fundamental understanding and industrial design considerations



- Only very small amounts of intermediates/by-products
- Analysis of carbon formed reveal usage options
- DETCHEM<sup>PBR</sup> used for scale-up towards pilot plant





3d bed porosity X-ray tomography

Grunwaldt group



SEM picture

**D** • **BASF** 



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Carbon deposits on reactor wall



BASF's pilot plant (Ludwigshafen, Germany)

#### Chem. Eng. J. 485 (2024) 149684.

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# Methanol synthesis from green $H_2$ and industry $CO_2$ at mild conditions



## Dynamics on two-components catalyst



- 1. Emission control of local pollutants
- 2. Reduction of GHG emissions
- 3. Circular energy economies



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- Reversible operated solid-oxide fuel/electrolysis cells
  - Dynamics in inlet conditions
- NH<sub>3</sub> as chemical energy carrier
  - Dynamics of electro- & thermo-catalysis
  - Forced dynamic operation to avoid NH<sub>3</sub> slip
- Reactive metals as chemical energy carrier
  - Dynamic reactor operation

# Challenge of the energy transition towards renewables Need for energy storage materials substituting fossil fuels





Net electricity production by solar (PV) and wind in Germany in October 2024

Renewable energy production highly volatile

- Renewable energies production cannot be provided at a scale where and when needed
- Difficult storage & transportation at large scale
- Need of chemical energy carriers (100s TWh)
  - Long-term (months, years) storage capacity
  - High (volumetric) energy density
  - Available, easy to handle and transportable, non-toxic and safe

# Challenge of the energy transition towards renewables Materials for circular energy economy substituting fossil fuels





Options for future chemical energy carriers being more climate-friendly:

- Synthetic hydrocarbons from electrolysis hydrogen
- Electrolysis hydrogen
- Ammonia
- Metal hydrides (e.g. KBH<sub>4</sub>)
- Metals



J. M. Bergthorson et al., Applied Energy 275 (2020) 115112.

- 1. Emission control of local pollutants
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# Syngas and methane as energy storage materials

# Energy circle using reversible solid-oxide cells technologies

- Scalable and distributed
- High roundtrip-efficiency and fuel flexibility





#### Efficient thermal management

 $\Rightarrow$  Presentation *OR92* by Oscar Furst (Thu, June 5th)

#### Appl. Energy 317 (2022) 119143.



Power to CH<sub>4</sub>

Tank (H<sub>2</sub>O,

 $CO_2$ rich)

# Reversible solid-oxide cells technologies for energy storage Dynamics in coupling of thermo- and electrocatalysis



Solid oxide cell operated with methane containing fuels at high temperature (600 - 900 °C)



# Solid-oxide electrolysis technologies for energy storage Dynamics on stack level due to mode switching and power variation





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Challenges at varying power:

- Small temperature gradients in stack
- Keep product composition constant

#### Solution:

Forced dynamic operation (inlet fluxes, air ratio)



- 1. Emission control of local pollutants
- 2. Reduction of GHG emissions
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# Ammonia as energy carrier can use existing infrastructure Alternative, decentralized routes to NH<sub>3</sub>



 $N_2 + 3 H_2 \rightleftharpoons 2 NH_3$ 

Proton-conducting ceramic cell

Green H<sub>2</sub> for Haber-Bosch





# Electrochemically supported synthesis of $NH_3$ in Fe/BCZY - PCCC Impact of protons and electric fields not understood yet







- 1. Emission control of local pollutants
- 2. Reduction of GHG emissions
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# Controlling ammonia slip by selective catalytic oxidation

Forced dynamic reactor operation makes Pd more active and selective



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#### Spatially resolved data (Pd/Al<sub>2</sub>O<sub>3</sub>)

#### Forced dynamic reactor operation enhances selective oxidation of NH<sub>3</sub>

- Exploit unique Pd/PdO redox dynamics
- Pd-based catalyst more active and selective than Pt-based benchmark catalyst
- Efficient catalyst operation strategies reduce noble metal demand and offer possibility to reduce catalyst size

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## Metals as carbon-free energy carriers

Promising substitution for C-fuels for energy retrieval on demand

- Huge amounts of energy can be stored due to high volumetric energy density and released by oxidation
- Potential metals: Al, Si, Na, Mg, Zn, Fe



### Example Iron (Fe)

- High energy density (16 kWh/l)
- Easily storable
- Very long retrieval time (years)
- Safety class as conventional fuels
- Non-toxic
- Long-distance transport established

# Fossil fuels 1.36 TWh 1.22 TWh

Coa



J.M. Bergthorson, Prog. Energy Combust. Sci. 68 (2018) 169.



# Iron as carbon-free energy carrier in a wet cycle: Energy release by oxidation with steam at mild temperatures







Prototype of full cycle with PV panel, PEM fuel and electrolyzer cells, and e-engine at KIT



- Oxidation with steam in a fixed bed reactor at mild conditions producing H<sub>2</sub>
- Reduction with electrolysis H<sub>2</sub> in the same compartment

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Reversible SOC promising device for solar power to H<sub>2</sub> to electricity

### $\rightarrow$ Potential for temporal and seasonal H<sub>2</sub> and energy storage

ChemSusChem 17 (2024) e202400351

Knapp et al., Fuel (2025)

## Iron as carbon-free energy carrier: Challenges of the steam-iron process



Sintering of the iron powder particles after cycling





- Dynamic operation leads to fast loss in activity due to loss of porosity of Fe particles and powder bed
- Additives can prevent conversion loss
- Particle morphology maintained (acts as catalyst) for repeated reaction cycles



#### 5% Mo modified Fe particles

fresh



#### after 50 redox cycles at 600°C



A. Knapp et al., Fuel (2025)

# Dynamics in environmental catalysis: from surface coverages to reactor operation



Dynamics appear in catalysis on many scales







Dynamic operation: great potential for boosting activity





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LEIPZIG

# Thank you for your attention and

# stay dynamic as catalyst!

Amsterdam

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oAntwerp

Dijon

**Brussels** 

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Copenhagen

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Munich

Bremen

Frankfurt

Zürich

Switzerland

Stuttgart

Dortmund

oDüsseldorf

Esseno

Luxembourg

Coloane

Strasbour

Hanover



MODEGAT VIII September 21-23, 2025 Bad Herrenalb • Germany



8<sup>th</sup> International Symposium on Modeling of Exhaust-Gas After-Treatment

Submission Deadline: June 15, 2025 E-Mail: mail@modegat.org





Chair Chemical Technology Olaf Deutschmann

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