# Bosch Carbon Catalyst Regeneration in Water II: Analysis of Bosch Catalyst Beads Throughout the Initial Regeneration Process

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Presenter

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Authors

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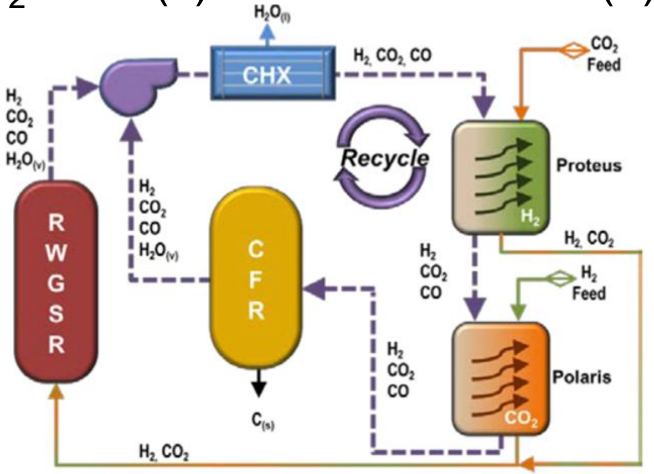
# Introduction

| | | |
|---|---|---|
| **RWGS** | $CO_2 + H_2 \leftrightarrow H_2O + CO$ | (1) |
| **CO Hydrogenation** | $CO + H_2 \leftrightarrow H_2O + C(s)$ | (2) |
| **Boudouard** | $2CO \leftrightarrow CO_2 + C(s)$ | (3) |
| ***Total Bosch Process*** | $CO_2 + 2H_2 \leftrightarrow 2H_2O + C(s)$ | (4) |

- Bosch Process reduces carbon dioxide and hydrogen into water and solid carbon catalytically
  - Candidate for closing the loop for long distance space travel by eliminating the need to resupply water
  - The S-Bosch system makes the Bosch process more efficient by dividing the reactions into different reactors with the RWGSR housing the RWGS reaction and the other reactions occurring in the CFR
- Need a method for carbon build up removal from CFR to prevent over-pressurization and regenerate catalyst
  - Method investigated was removal of carbon via water




*Credit: Stanley et al. (2017)*

# Introduction (Continued)

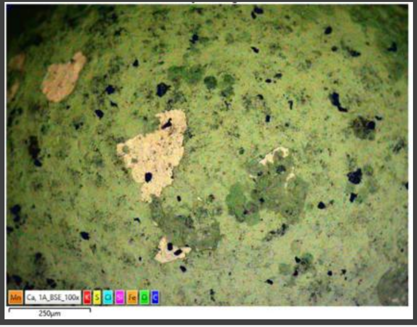
- In previously discussed experiments random samples of carbon dust and carbon-coated catalyst beads were taken from the COR-CaTS reactor and agitated in room temperature water
  - Optimal parameters for catalyst bead regeneration were experimentally determined
  - Samples of catalyst beads from different experimental stages were taken for SEM analysis to gauge catalyst degradation from the carbon formation reaction as well as the regeneration process.

# Methodology

- Samples of catalyst beads from different experimental stages were taken for SEM analysis to gauge catalyst degradation from the carbon formation reaction as well as the regeneration process.
  - Unreacted catalyst beads
  - Carbon-coated catalyst beads
  - Carbon-coated catalyst beads that had been agitated in 100 mL of room temperature water at a spin setting of 12 (1200 rpm) for a duration of 18 hr followed by repeated washings at 30 min intervals under the same conditions
- Samples were fixed to the sample holder with carbon tape for surface imaging
  - Images were taken using an SEM microscope
- Samples were mounted in epoxy pucks and ground to reveal the bead cross-sections
  - Images were taken using an optical microscope and an SEM microscope
- Sample cross-sections were etched with 2% Nital
  - Images were taken using an optical microscope and an SEM microscope
- Samples of the wastewater were taken and imaged under an optical microscope
- Wastewater samples were collected with a pipette, deposited on an aluminum stub with carbon tape, dried out, and then imaged using a SEM microscope

SLIDE TITLE

# Bead Surface Images



**Unreacted Catalyst Beads Material Composition**

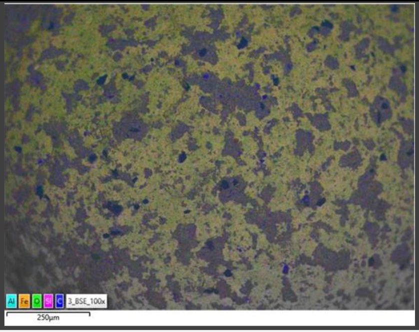
Magnification of 100x with a 250 μm scale bar

- Uneven iron-oxide surface layers
  - Removed during Carbon formation process
- Smooth overall but has small pores and crevices

**Carbon-Coated Catalyst Beads Material Composition**

Magnification of 100x with a 250 μm scale bar

- No surface oxidation
- Evenly coated in carbon



**Repeatedly Washed Carbon-Coated Catalyst Beads Material Composition**

Magnification of 100x with a 250 μm scale bar

- Surface oxidation present
- Small patches of carbon on surface
- Rough surface and has scattered pits and crevices

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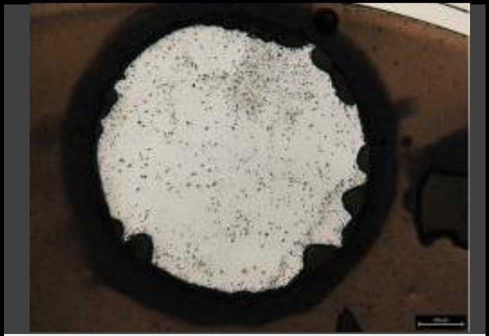
# Bead Cross-Section Images

**Unreacted Catalyst Beads**

Magnification of 50x with a 250 μm scale bar

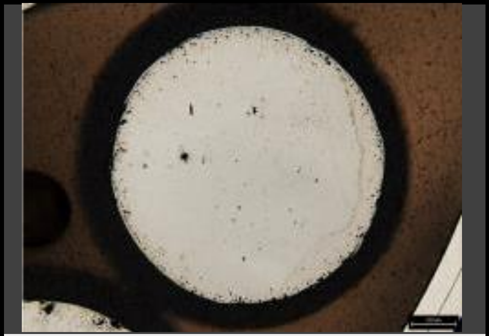
- Contains crevices and pits



**Carbon-Coated Catalyst Beads**

Magnification of 50x with a 250 μm scale bar

- Contains deep crevices and pits filled with carbon and further carbon deposits deeper in the bead
  - Catalyst displacement from the Boudouard reaction

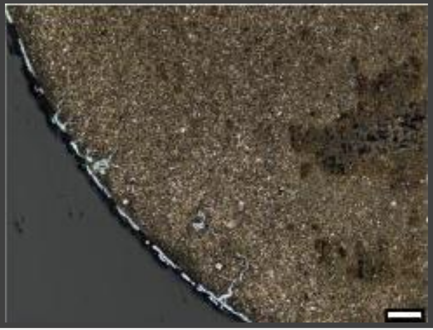


**Repeatedly Washed Carbon-Coated Catalyst Beads**

Magnification of 50x with a 250 μm scale bar

- Contains traces of pitting and carbon deposits
  - Not as severe as carbon-coated samples due to regeneration process

# Bead Cross-Section Images Cont.

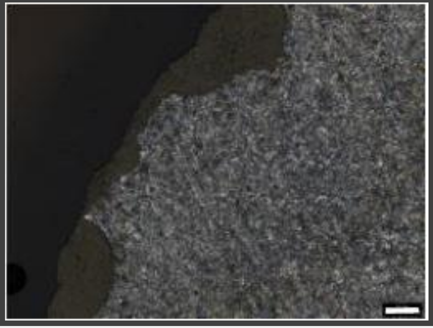


**Unreacted Catalyst Beads**

Etched with 2% Nital

Magnification of 200x with a 50 µm scale bar

- Contains dendritic regions inside bead
- Tempered martensite/bainite structure
- Outer edges are oxidized

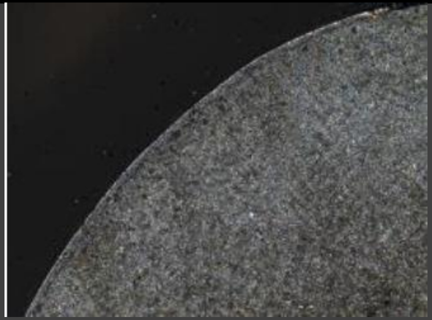


**Carbon-Coated Catalyst Beads**

Etched with 2% Nital

Magnification of 200x with a 50 µm scale bar

- Oxidized dendritic regions
- Annealed martensite structure
- No oxidation at edges



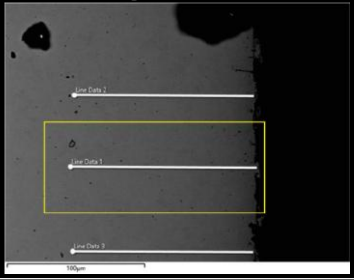
**Repeatedly Washed Carbon-Coated Catalyst Beads**

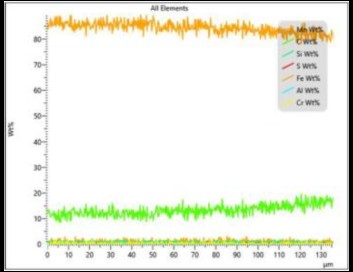
Etched with 2% Nital

Magnification of 200x

- Oxidized dendritic regions
- Annealed martensite structure
- Outer edges are oxidized

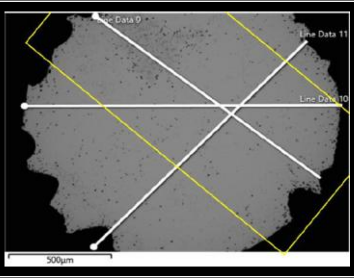
# Material Composition of Beads Cross-Section Images

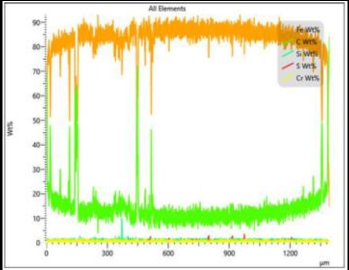



**Unreacted Catalyst Beads**
EDS line scan and graph of weight percentage of materials detected
Magnification of 500x with a 100 μm scale bar

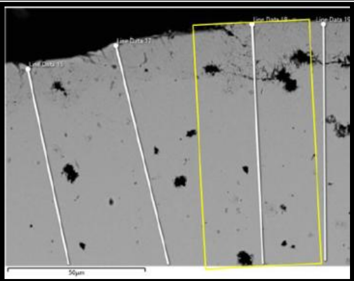
- Materials not unevenly distributed around dendritic regions
- Higher concentrations of carbon on exterior of beads due to manufacturing process

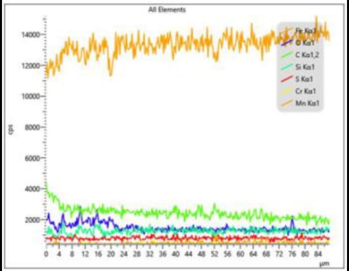



**Carbon-Coated Catalyst Beads**
EDS line scan and graph of weight percentage of materials detected
Magnification of 75x with a 500 μm scale bar

- Spikes of carbon throughout bead interior particularly around dendritic regions
- Majority of catalyst remains viable despite deep pitting and carbon deposits
- Oxidation around dendritic regions

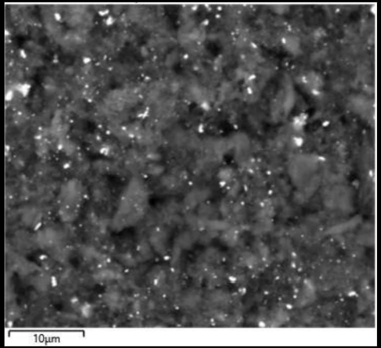


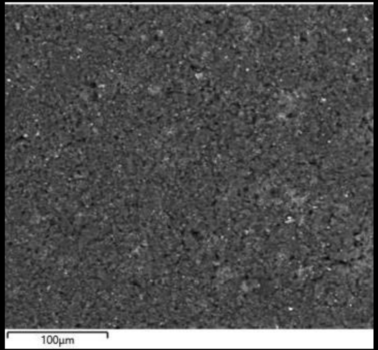

**Repeatedly Washed Carbon-Coated Catalyst Beads**
EDS line scan and graph of weight percentage of materials detected
Magnification of 1000x with a 50 μm scale bar

- Small carbon deposits in bead interior
- Oxidation around dendritic regions
- Minimal carbon on edges of beads

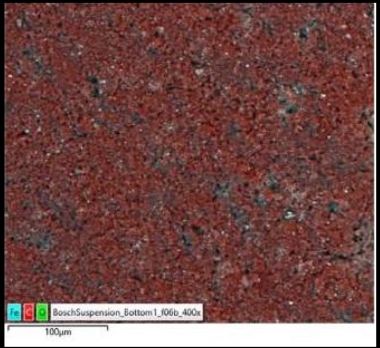
# Waste Water Images



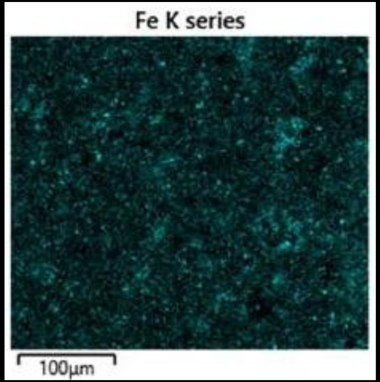

**Iron Catalyst and Carbon Wastewater Sample**
Magnification of 3000x with a 10 µm scale bar




**Iron Catalyst and Carbon Wastewater Sample**
Magnification of 400x with a 100 µm scale bar




**Material Composition of Iron Catalyst and Carbon Wastewater Sample**
Magnification of 400x with a 100 µm scale bar




**Iron Composition of Iron Catalyst and Carbon Wastewater Sample**
Magnification of 400x with a 100 µm scale bar

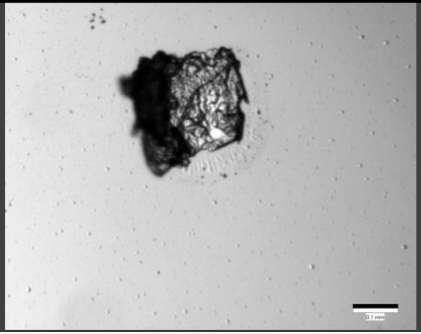
- Samples were taken from the wastewater used in the carbon-coated catalyst regeneration experiments.
  - Small shiny particles were observed floating in the water resulting in further investigation
- SEM images show iron particles interspersed through the carbon
  - Evidence of the metal dusting that occurs during the Boudouard reaction
- Material composition scans further confirm the presence of flakes of the iron catalyst in the carbon waste water sample

# Bead Cross-Section Images Cont.

**Carbon Mass Cross-Section with Embedded Iron Catalyst Particles**

Magnification of 100x with a 100 μm scale bar

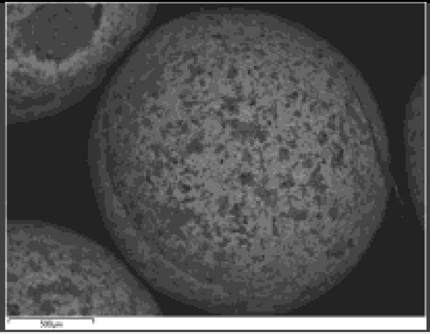
- Iron Catalyst particles were embedded throughout the mass of carbon
- Further confirms metal dusting during the Boudouard reaction



**Iron Catalyst Particle from Wastewater**

0.1 μm scale bar

- More iron catalyst particles were found floating in the collected wastewater from the regeneration process
- Further SEM analysis of samples of wastewater was conducted after this discovery



**Repeatedly Washed Carbon-Coated Catalyst Beads**

Magnification of 50x with a 500 μm scale bar

- Regenerated beads had a warped overall shape from either reaction or agitation process
- Some cracking was observed on the surfaces

# Conclusions and Future Work

- SEM imagining showed that the catalyst is oxidized before loading into the CFR but this does not affect the ability of carbon to form on the surface of the catalyst.
- Images show that while there is some degradation of the catalyst in the form of metal dusting throughout the reaction the majority of the catalyst remains intact and viable for further use.
- Regeneration of the beads leaves trace amounts of carbon. However, the surface is able to oxidize indicating the presence of available activation sites for further reaction.
- Lab-scale devices are being developed to separate the carbon and water waste stream and to agitate the catalyst within a reactor.
  - These will eventually be placed in a 4-crew member reactor which will undergo cyclical testing.

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