

# Characterization of Catalysts for the Synthesis of Higher Alcohols using Electron Microscopy

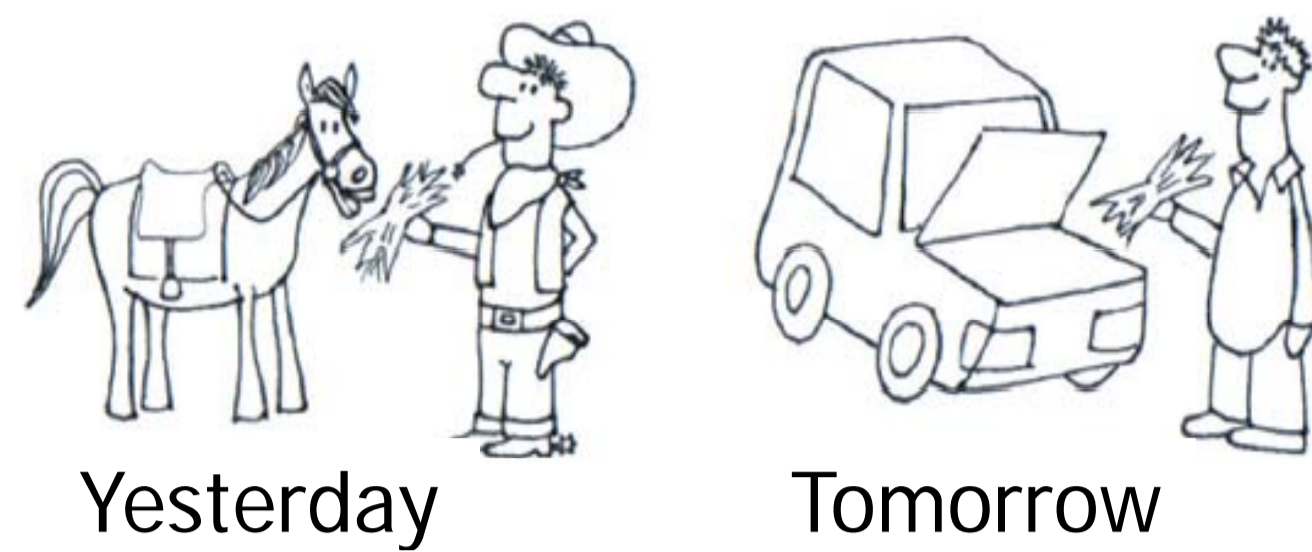
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## Motivation

Increasing interest in the development of transportation fuels from sustainable resources results in the need for new and better production paths for *e.g.* the synthesis of alcohols from biogas. Higher alcohols are favorable due to their higher energy density and ease of use in today's internal combustion engines. However, the poor yield of higher alcohols in chemical reactions requires efficient catalysts for these applications [1].

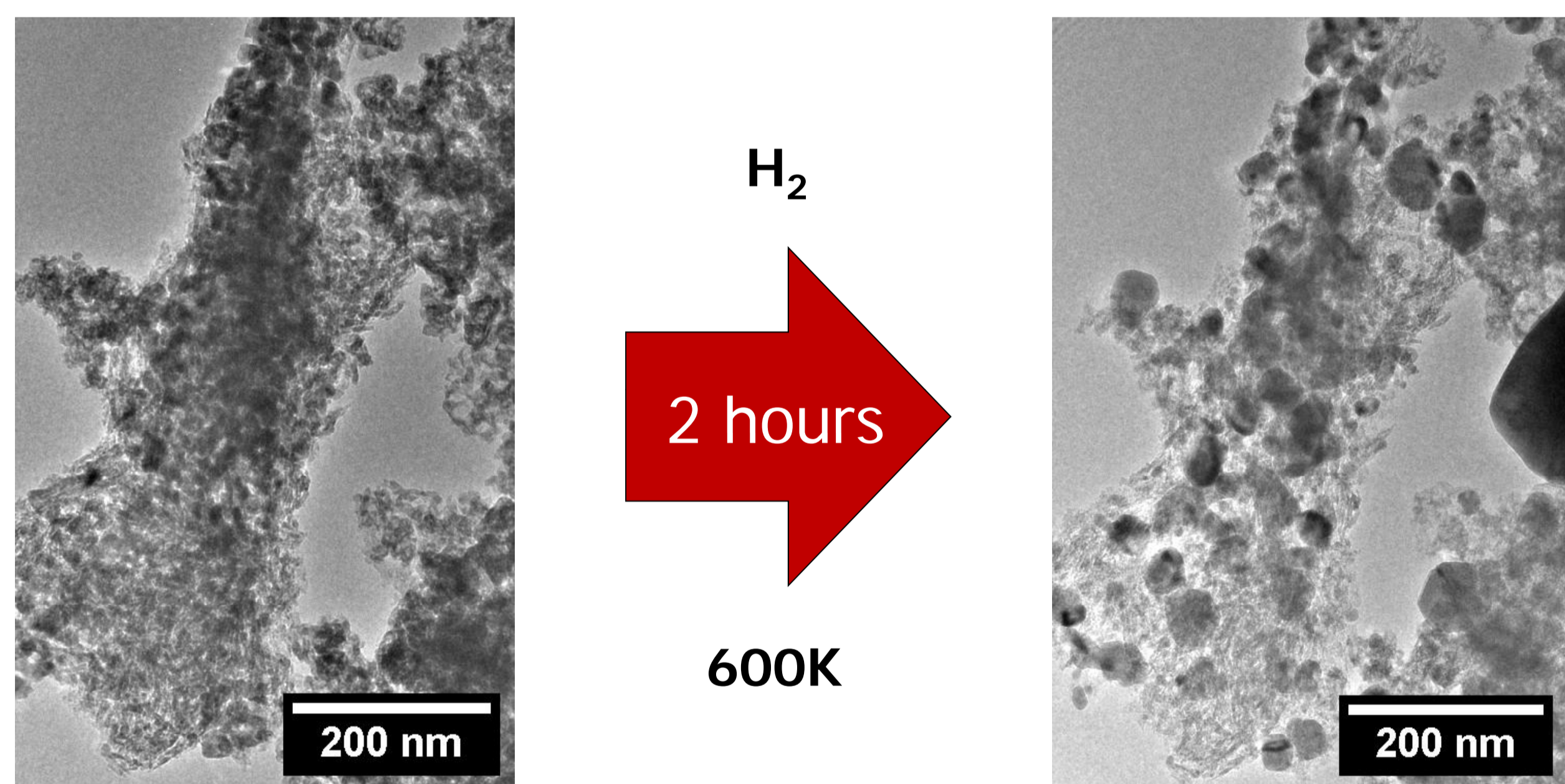


## Requirements for catalysts

- Highly active and selective towards higher alcohols
- Chemically and structurally stable under reaction conditions
- Based on elements that are abundant on Earth

## CuSn supported on Al<sub>2</sub>O<sub>3</sub>

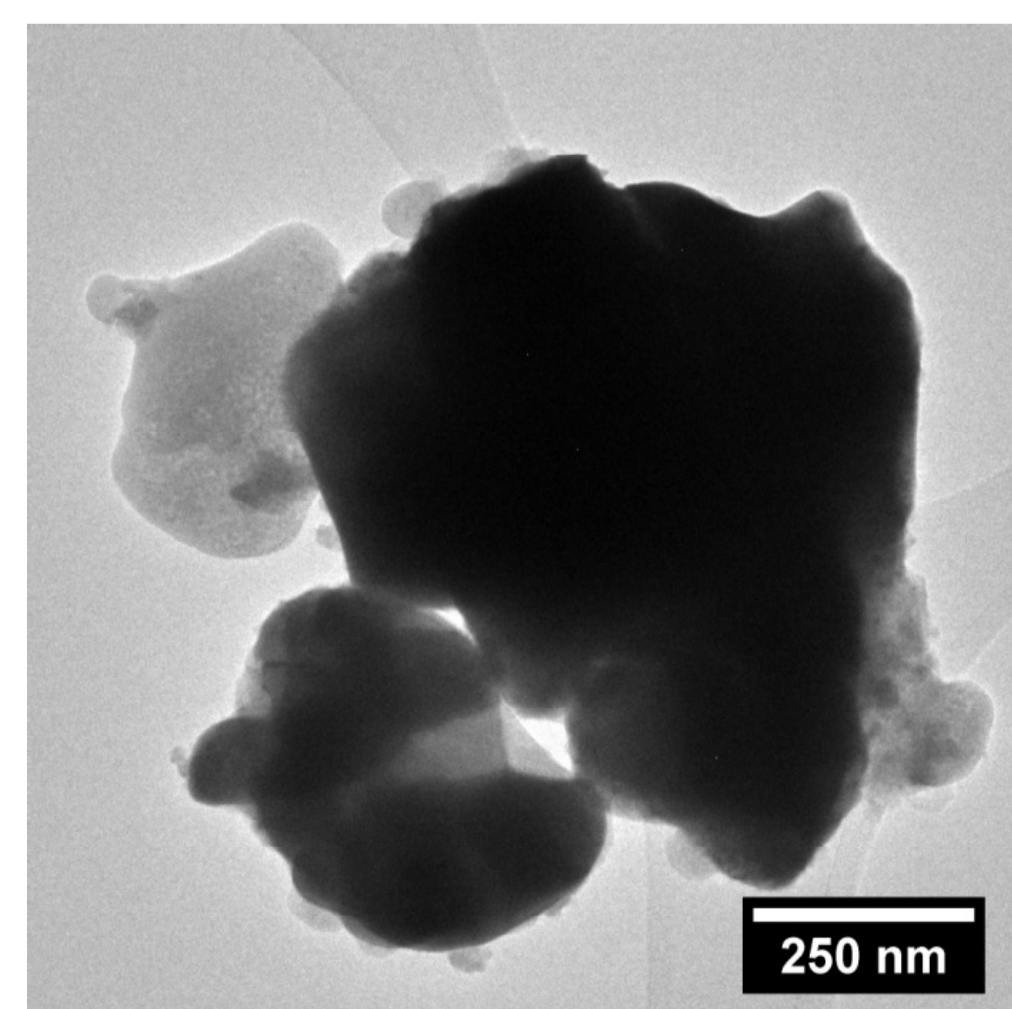
CuSn/Al<sub>2</sub>O<sub>3</sub> was predicted to be promising by DFT calculations. However, it shows strong degradation during catalytic testing. ETEM experiments were carried out in an environment (gas, pressure and temperature) that was chosen to approach realistic catalytic testing conditions.



High vacuum, RT

H<sub>2</sub> (1.2 mbar), 600K

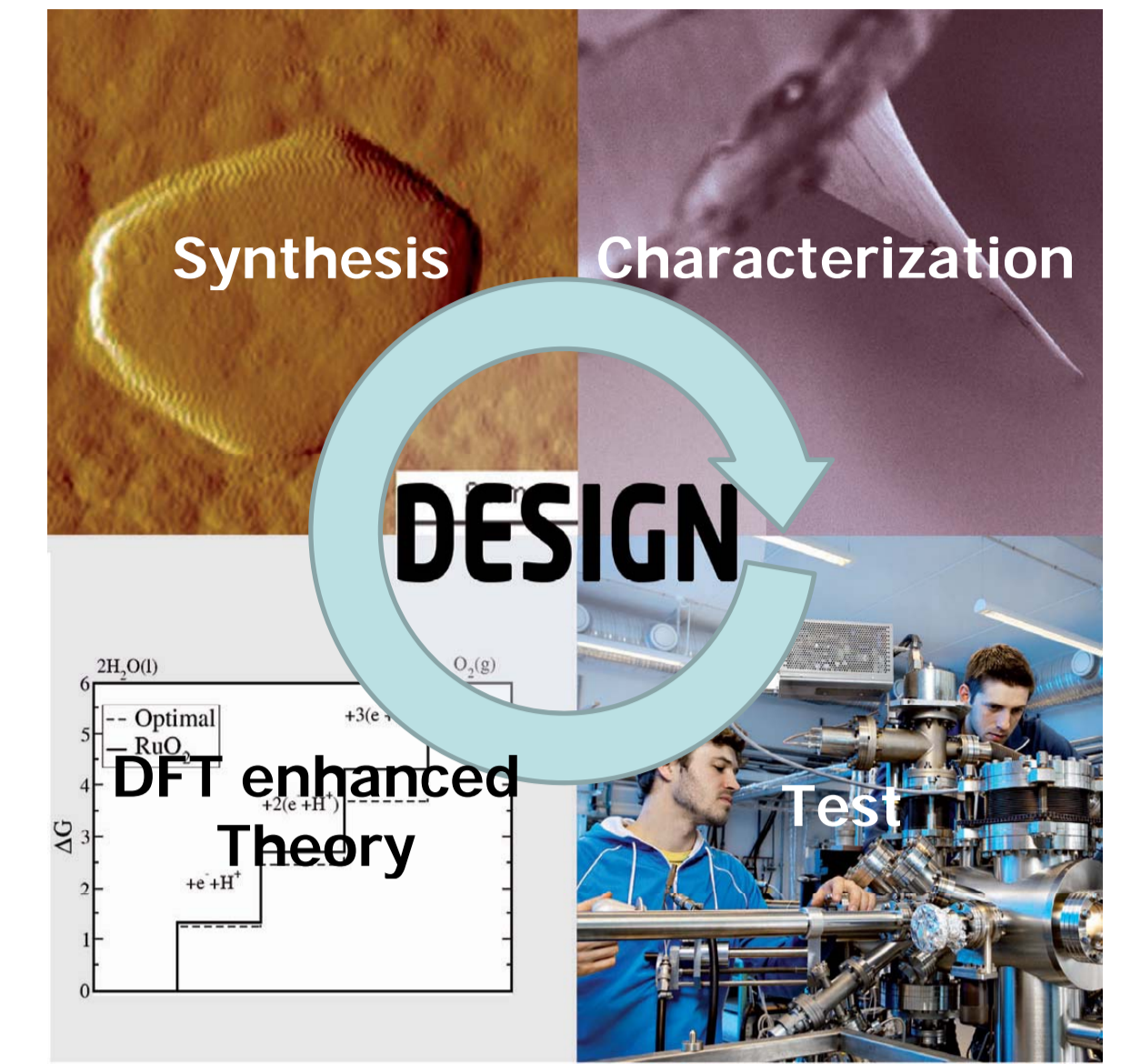
- The sample initially comprised 200 nm to 2 μm agglomerates.
- 30–300 nm crystals formed during *in situ* reduction.
- EELS investigation of these crystals shows both Cu and Sn.
- A catalyst examined after *ex situ* testing in a chemical reactor shows similar behaviour.
- However, the longer reaction time and higher gas pressure then leads to the formation of larger crystals.
- Sintering is proposed as the primary reason for catalytic deactivation.



Catalyst after usage in a chemical reactor

## Approach

This project is part of the CAtalysis for Sustainable Energy (CASE) initiative and involves the design and characterization of catalysts. When combined with DFT calculations, synthesis and chemical testing, a feedback loop for systematic design is obtained.

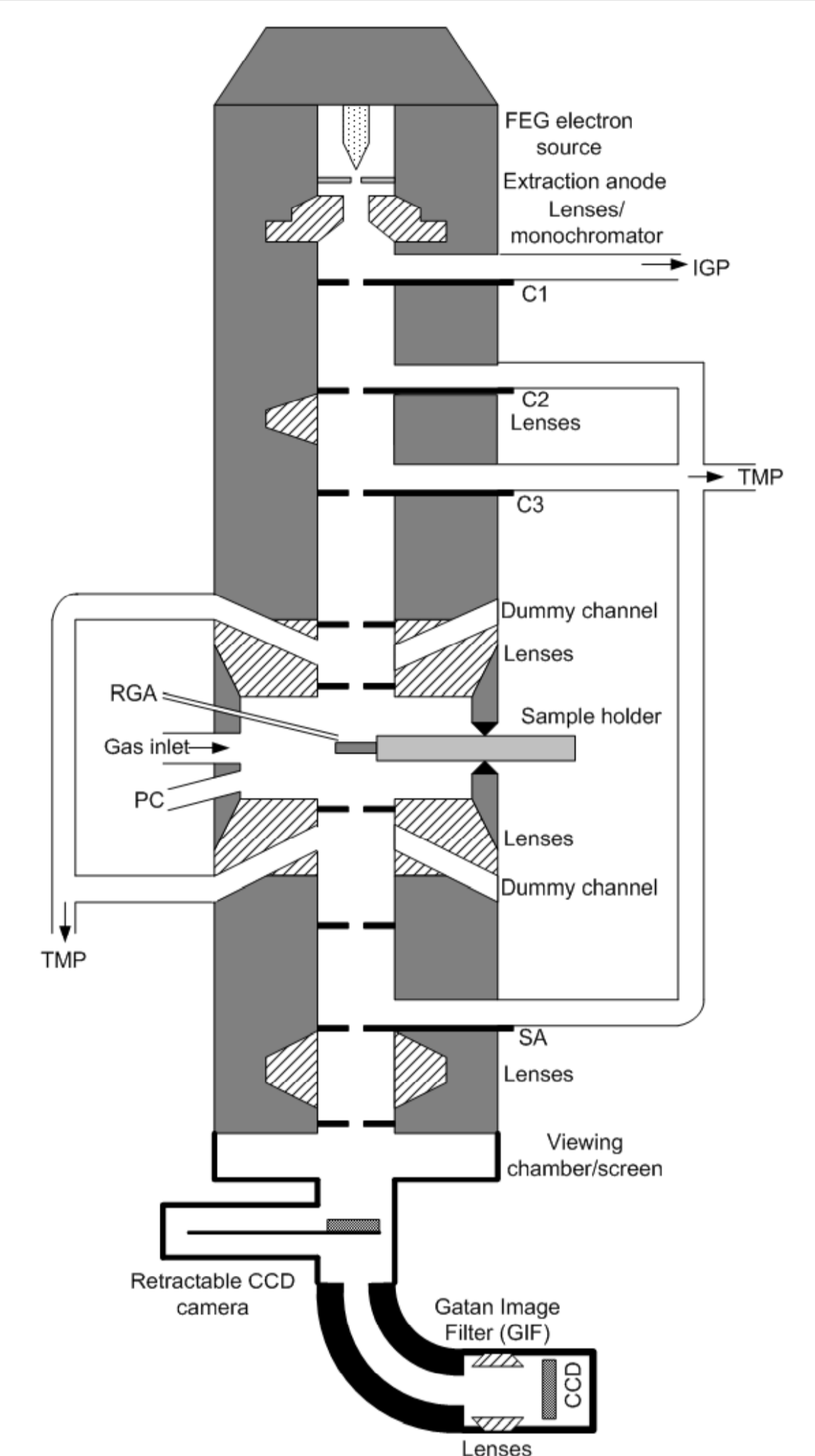


## Experimental details

FEI Titan environmental transmission electron microscope (ETEM) equipped with a differential pumping system, an image aberration corrector, an EDX spectrometer and a Gatan imaging filter.

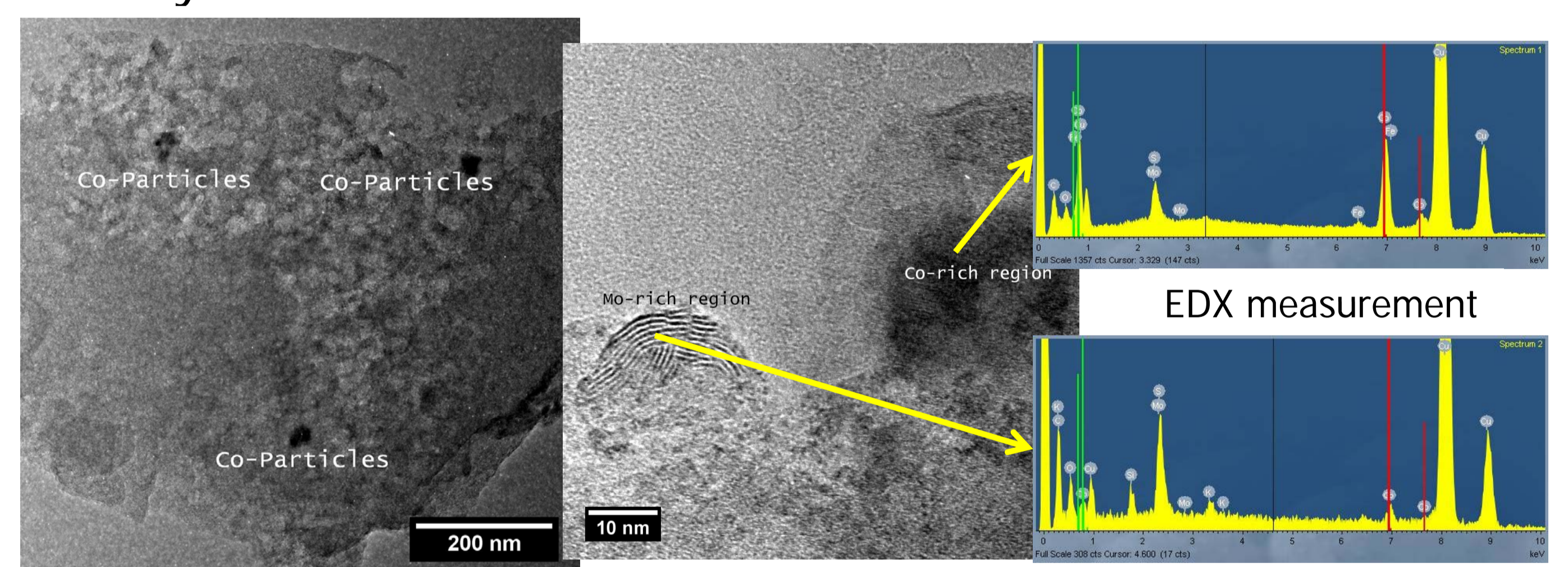
This microscope allows for experiments in different gases at elevated temperature without compromising the TEM performance significantly.

In this way, catalysts can be studied in the TEM in an environment that approaches realistic conditions [2, 3].

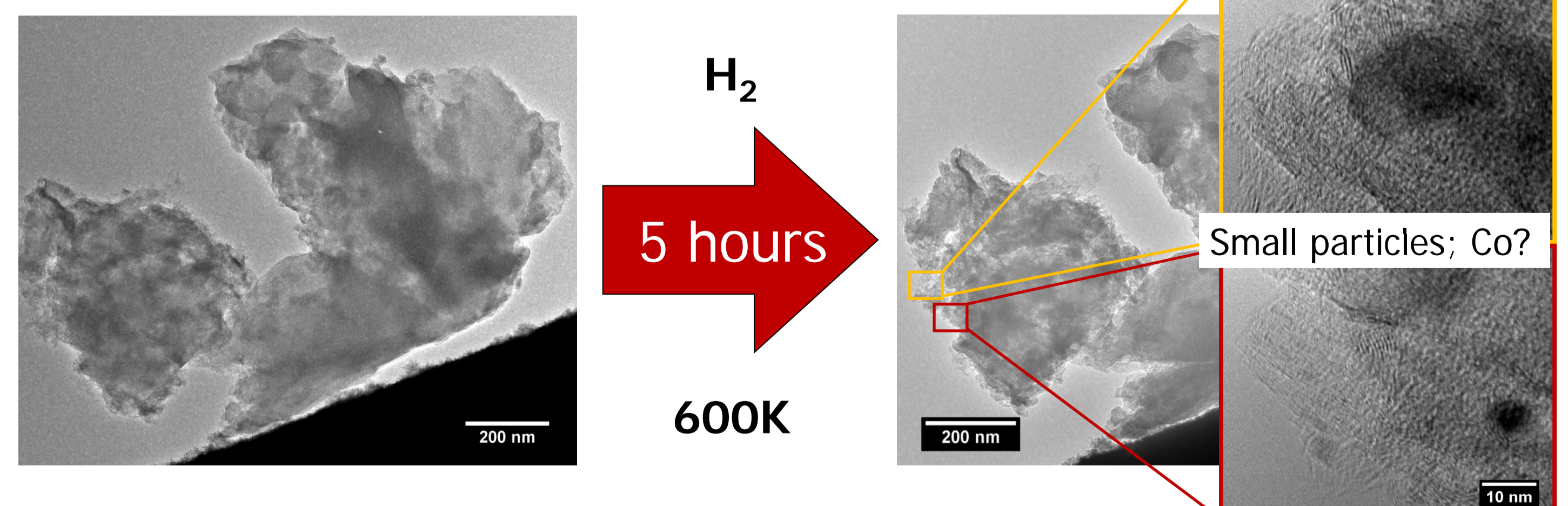


## K promoted Co–MoS<sub>2</sub>

After catalytic testing, K promoted Co–MoS<sub>2</sub> was investigated to identify structural changes. EXAFS measurements (not shown) indicated increased Co-Co coordination. TEM images confirmed Co crystal formation.



An ETEM reduction experiment on an as-cast catalyst of the same material also suggests the formation of Co crystals.



High vacuum, RT

H<sub>2</sub> (1.5 mbar), 600K

## References

- [1] J. J. Spivey, A. Egbebi, *Chemical Society Reviews*, **2007**, vol. 36, pp. 1514 - 1528.
- [2] A. K. Datye, *Journal of Catalysis*, **2003**, Vol. 216, pp. 144 - 154.
- [3] T. W. Hansen et al., *Science*, **2001**, Vol. 294 (5546), pp. 1508 - 1510.

## Acknowledgment:

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