Benefit of Research Infrastructures to industrial trade and wealth creation

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RESEARCH | TECHNOLOGY | CATALYSTS

Haldor Topsøe A/S
Catalysing your business

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What type of RI does industry need?

- Type of industrial use of RI's:
  mainly strategic R&D
  (not basic research, not quality control)
- Different ways:
  - fully involved in experiments
  - collaboration with academic world
  - analytical service, automatization (full analytical lab)
- Balance between techniques new for synchrotron (fs methods, high brilliance nano-beams) and established techniques (high flux)

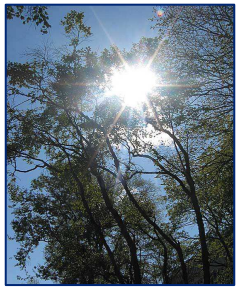
Goals in Catalysis

- Understand and predict relation between material properties and reactivity
- Towards a rational design of catalysts:
  Improve design and production of existing processes and catalysts and develop new ones
- Tailoring of:
  - selectivity (100%), activity, deactivation, shape, size, mechanical strength, thermal stability, material costs, purity of raw materials
- Driving forces: often (environmental) regulations

Global Challenges

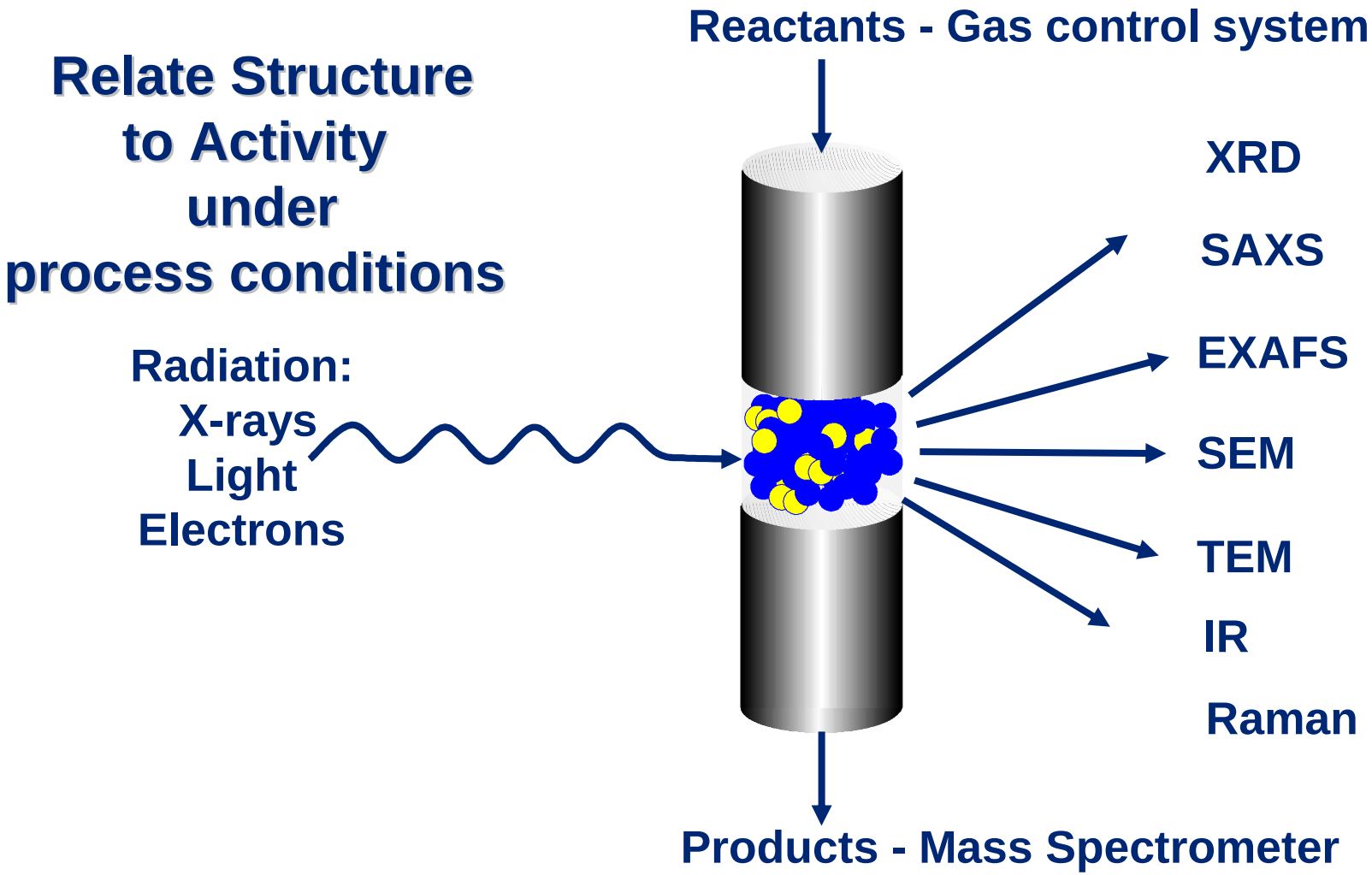
- World population
  - Food shortage

- Resources
  - Efficient use of resources



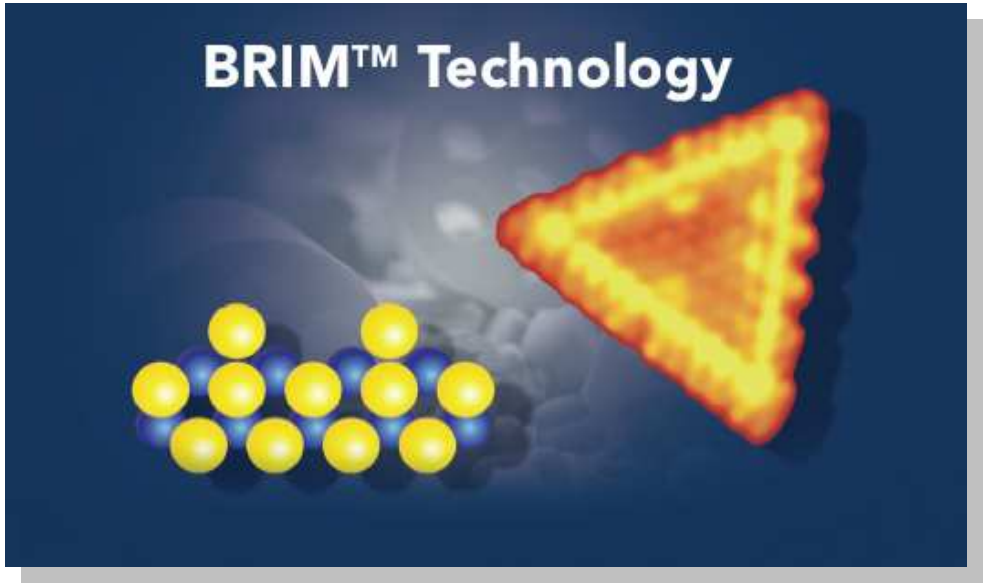
- Pollution
  - Reduction of smog, acid rain

In situ techniques in catalysis



Recent industrial refinery catalysts

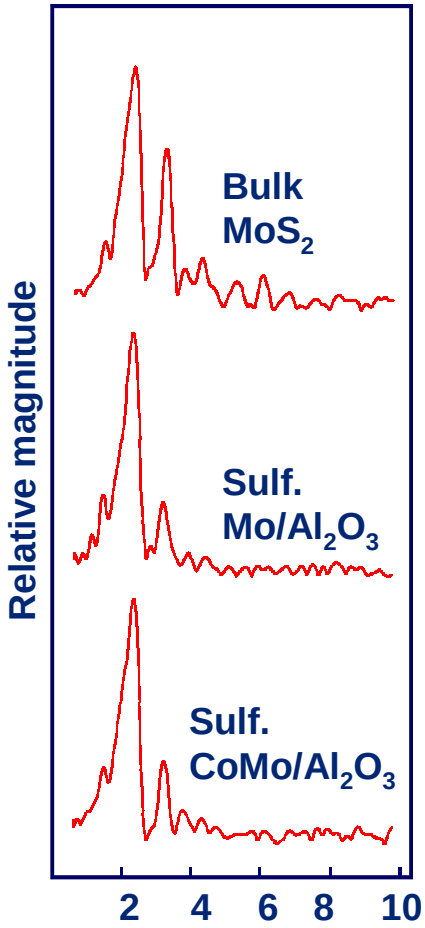
- Haldor Topsøe has introduced new generations of hydrotreating catalysts
- Aided by the input from molecular-scale research
- Named BRIM™ Technology



TK-558 BRIM™ (CoMo) and
TK-559 BRIM™ (NiMo) for FCC P/T
TK-576 BRIM™ (CoMo) for ULSD

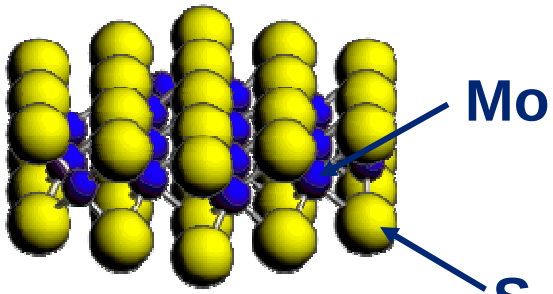
New *In Situ* Techniques Provided Insight

First in situ EXAFS Studies of $CoMo/Al_2O_3$ Catalysts



Clausen, Topsøe et. al (1981)

Mo present as MoS_2 nanoclusters



In situ FTIR: Monolayers (single MoS_2 slabs)

N.Topsøe (1980)

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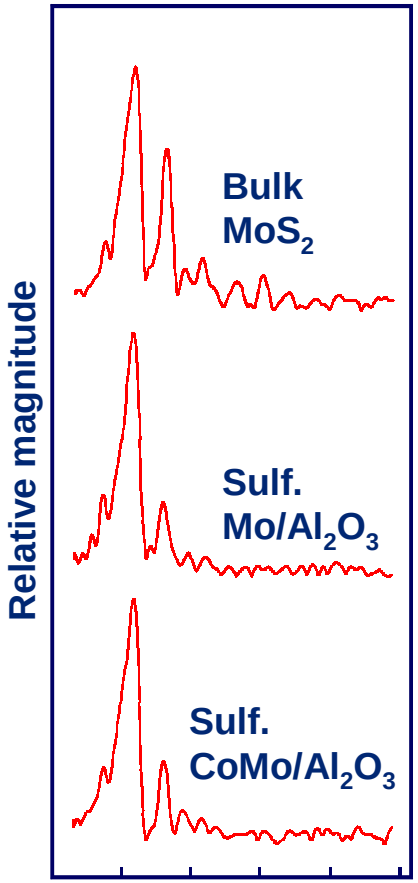

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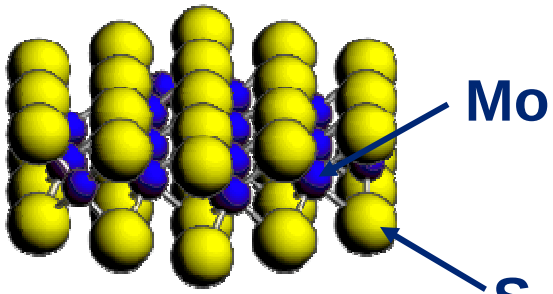
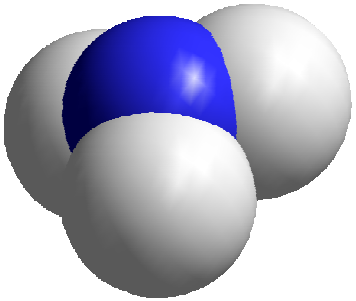


EXAFS: Does not provide a unique 3D structure

Interpretation still controversial!

(Clausen; Prins; Breysse;…)

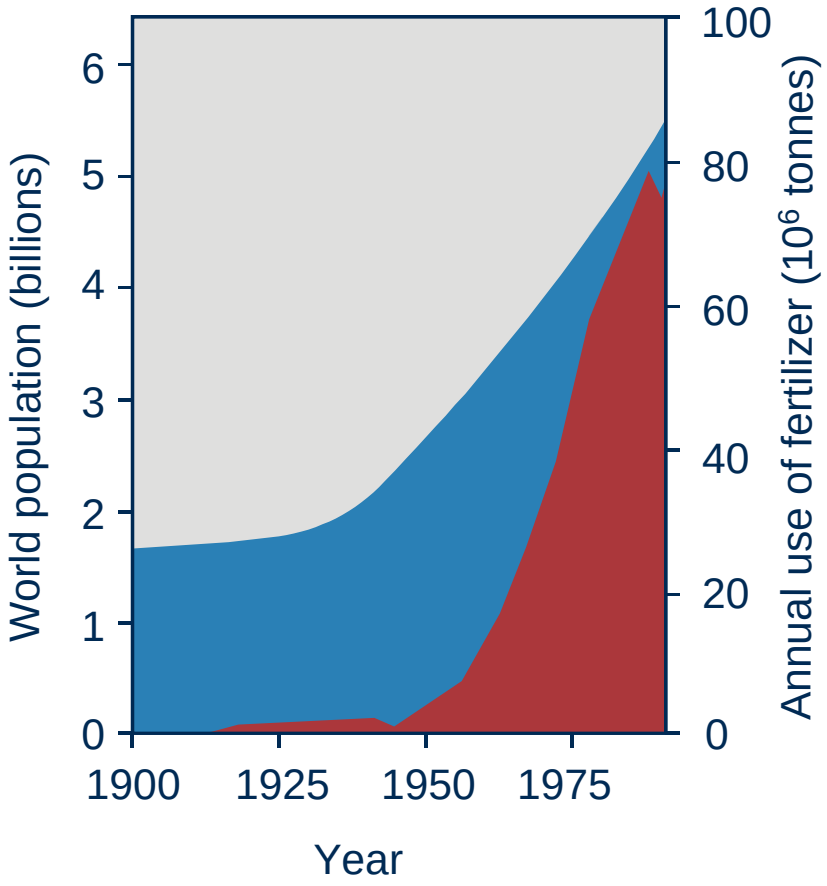
Ammonia Synthesis Catalyst

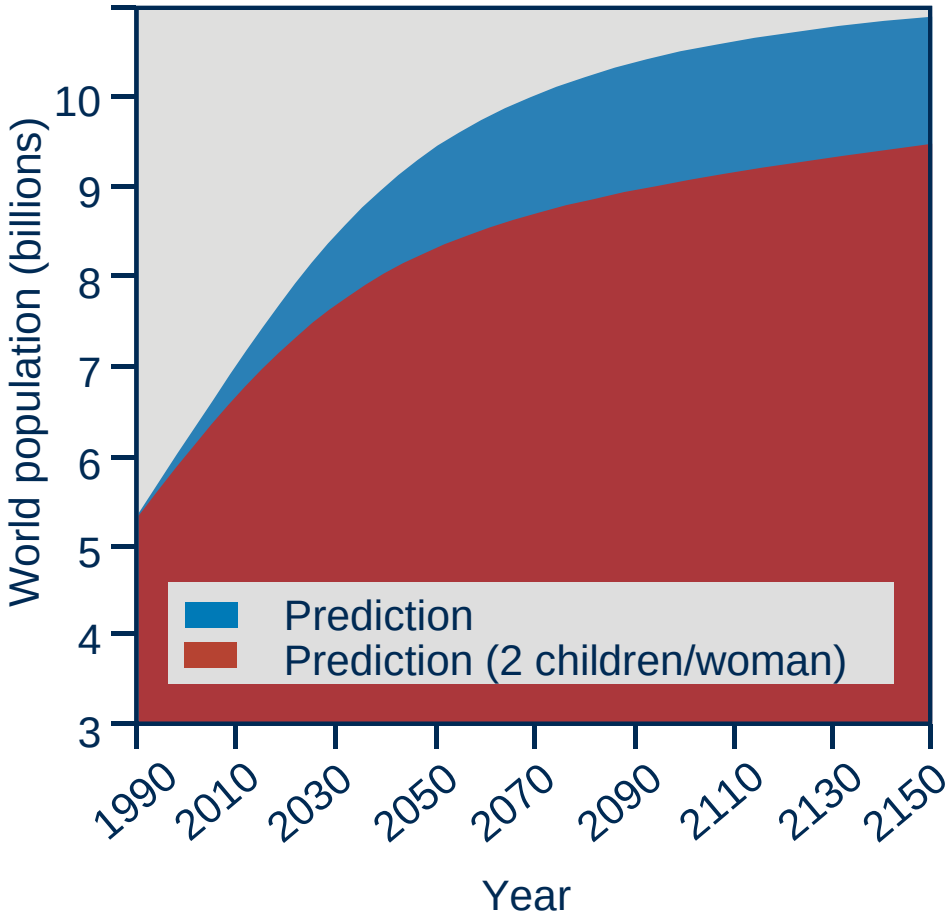


$$N_2(g) + 3\ H_2(g) \rightarrow 2\ NH_3(g)$$

- Fe/FeO-catalyst
- 1 kg of catalyst → 25 tonnes of NH_3
- Annual production: $150*10^6$ tonnes No catalyst? <150 tonnes

World Population





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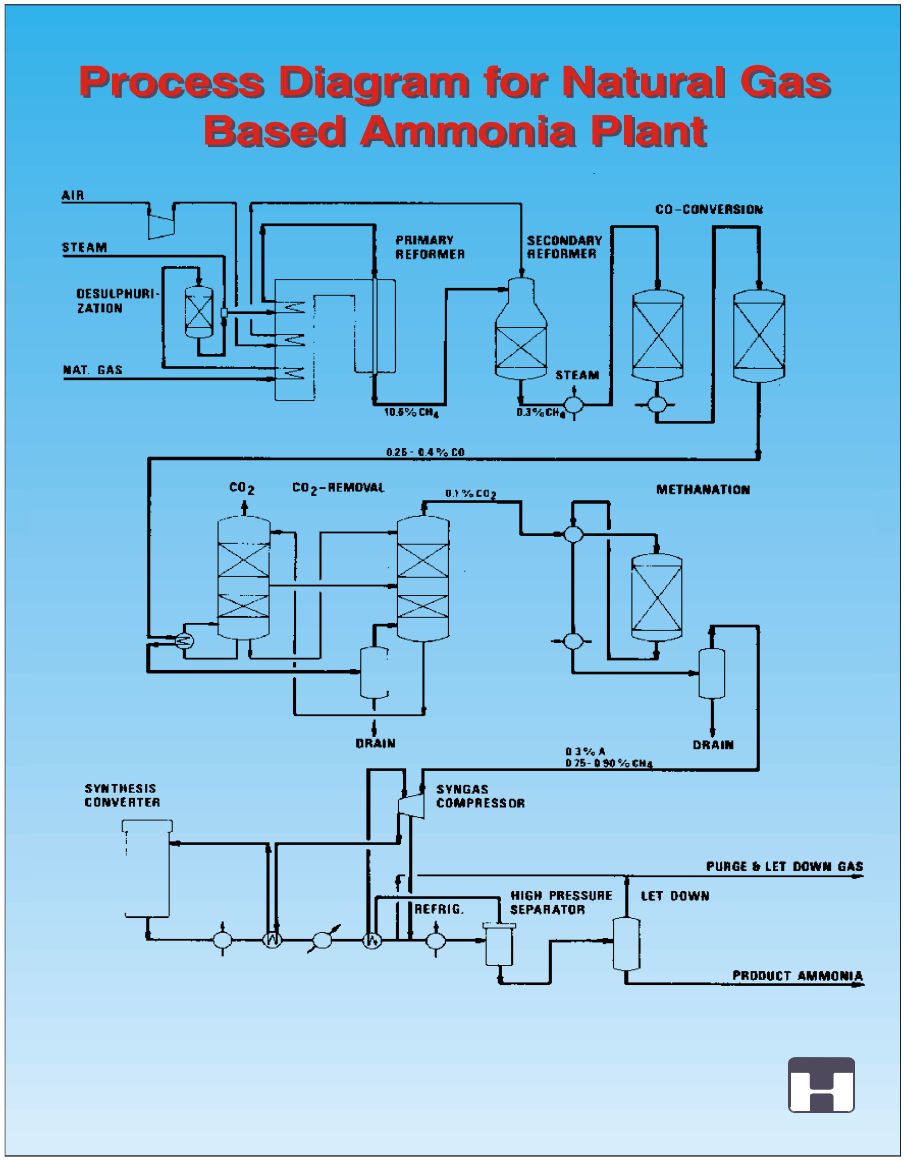
Process Plants are Highly Integrated



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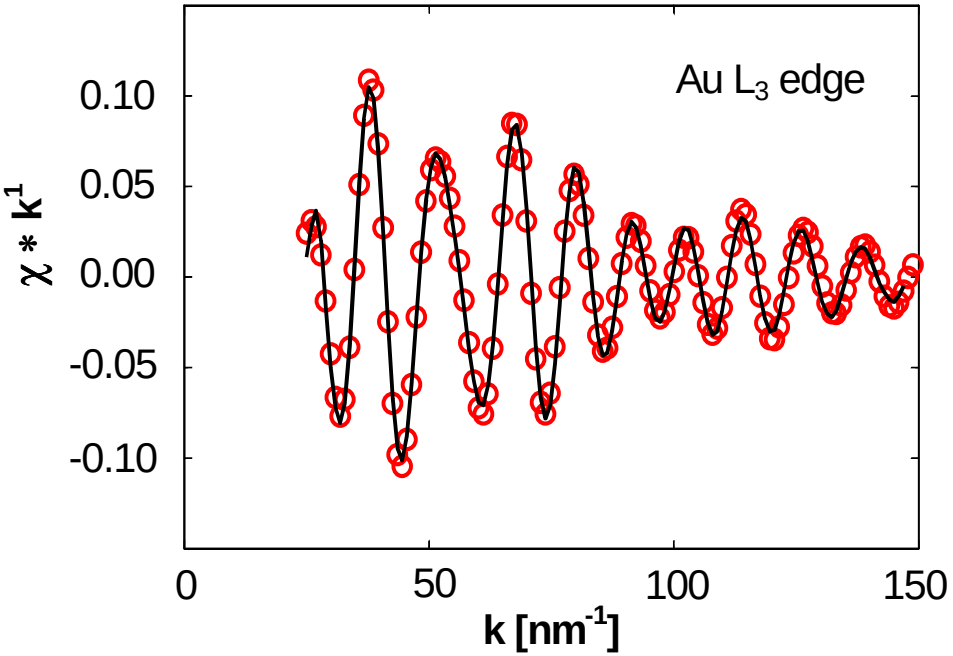
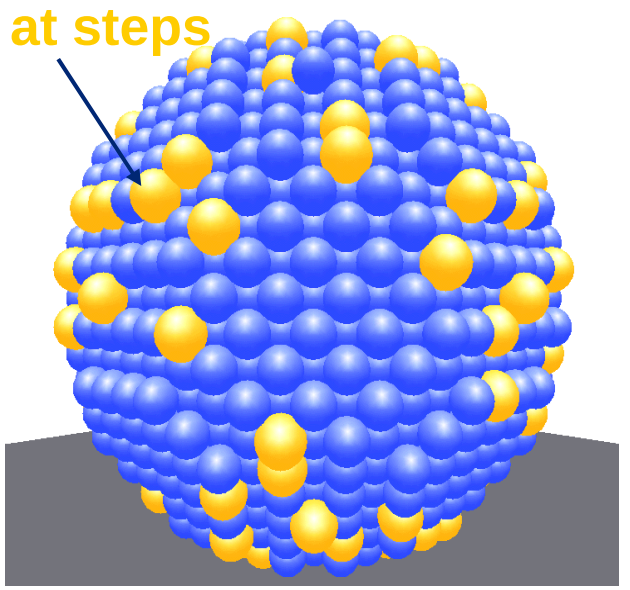
Reduction of deactivation by surface alloy formation of Au at Ni surface

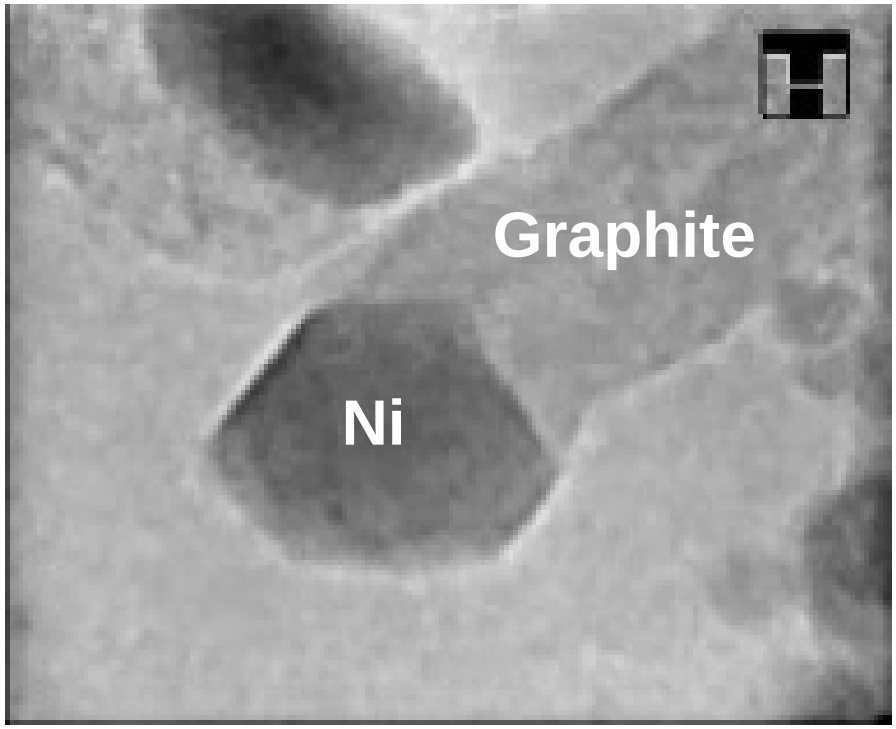
XAFS at Au L_3-edge:
Au present as surface alloy



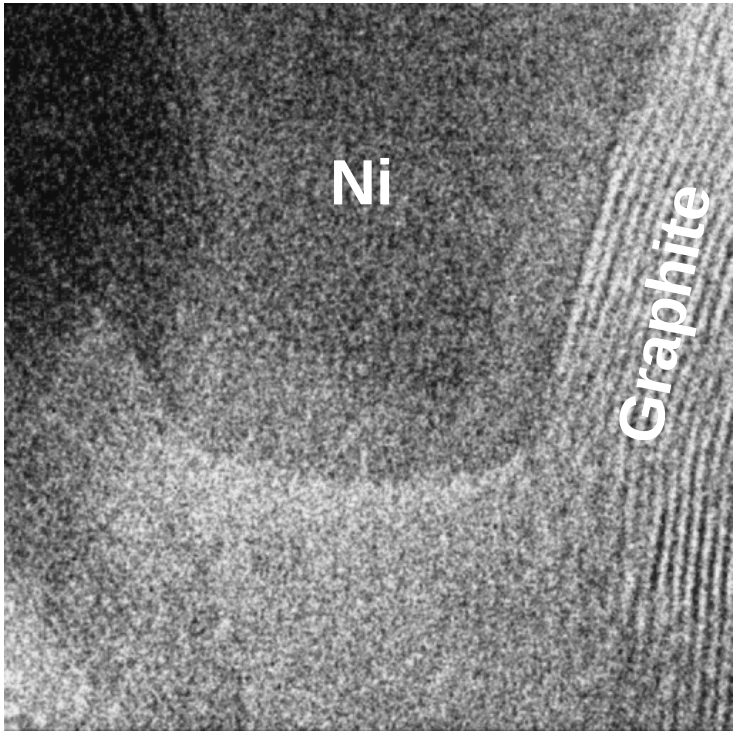


F. Besenbacher *et al.*, *Science* 279 (1998) 1913
A.M. Molenbroek *et al.*, *J. Phys. Chem. B* 105 (2001) 5450

From *in situ* HRTEM to Nano-tomography



5 nm



1 nm

Whisker carbon formation at steps: ~ 3 layers/sec
CH_4/H_2=1, P=5mbar , T=720℃

S. Helveg *et al*, Nature 427 (2004) 426

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Do we get what we need?

YES, but …

Key barriers for industrial use of RI's - 1

- Large expenses, large distance
  - travel, hotel, beamtime, equipment
- Many experienced researchers needed to perform an experiment
  - Complex experiments; unique results?; data analysis;
- Peer review system for beamtime applications:
  - Based on scientific quality, not on industrial relevance
- Confidentiality, IPR, secrecy agreements
- Beamline staff:
  - Lack of beamline staff: experiments run 24 hours/day but support staff not always available
  - Lack of experienced beamline staff (short-term contracts)
- Full remote control of experiments
- Chemistry lab close to beamline

Key barriers for industrial use of RI's - 2

- Differences with academic use of large-scale facilities:
  - Samples: larger amount; model vs. real catalysts
  - Faster results are demanded:
    robust methods + on-line analysis + fast access to facilities
  - Trend: reduced time from R&D to market
- Industry: product and process oriented

  Facilities: interest in methods and fundamental understanding
- Lack of quality control of beamlines:
  no standard protocols, no standardization of data formats
- Lack of automated on-line data analysis and reduction software
- Lack of standardized interfaces between beamline and sample environment

Increase of industrial use of SR

- Short access time to beamlines (2-4 weeks), preferably at short distance from home laboratories
- Professional and reliable operation of beamlines and synchrotron
- State-of-the-art beamline equipment, laboratories (also for sample preparation) and data analysis
- Building and operation of beamlines is responsibility of SR sources – industry is willing to pay for beamtime
- Beamline staff on long term contracts to improve competent service; basic understanding of catalytical processes performed present at beamline
- Coordination of industrial beamtime applications by an ”industrial user office” to ensure use of the proper beamlines + scientific support

Partly from:
Final declaration at *”Industrieforum In Situ Charakterisierung Katalytischer Prozesse”*, Nov. 2003, Hasylab (Bessy, Anka)

Future possibilities for industrial use of RI's

- Imaging:
  - Nano-tomography including element mapping as complementary method to transmission electron tomography: X-rays have larger penetration depth: in situ studies on < 10 nm length scale
- Design of new facilities:
  - workhorses vs. high brilliance
- Improved instrumentation (detectors):
  - Follow dynamic changes on short (<1ms) time scale e.g. during crystallization and agglomeration of catalysts
- New methods and techniques:
  - Improve sensitivity for surface and active sites
- XFEL:
  - Femtochemistry: Movie of chemical reaction during adsorption and desorption of gasses at a catalyst surface

Thank you for your attention

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