Future Energy: Chemical Solutions

Nottingham, 14 September 2007

Compact and safe hydrogen storage in solid metal ammines

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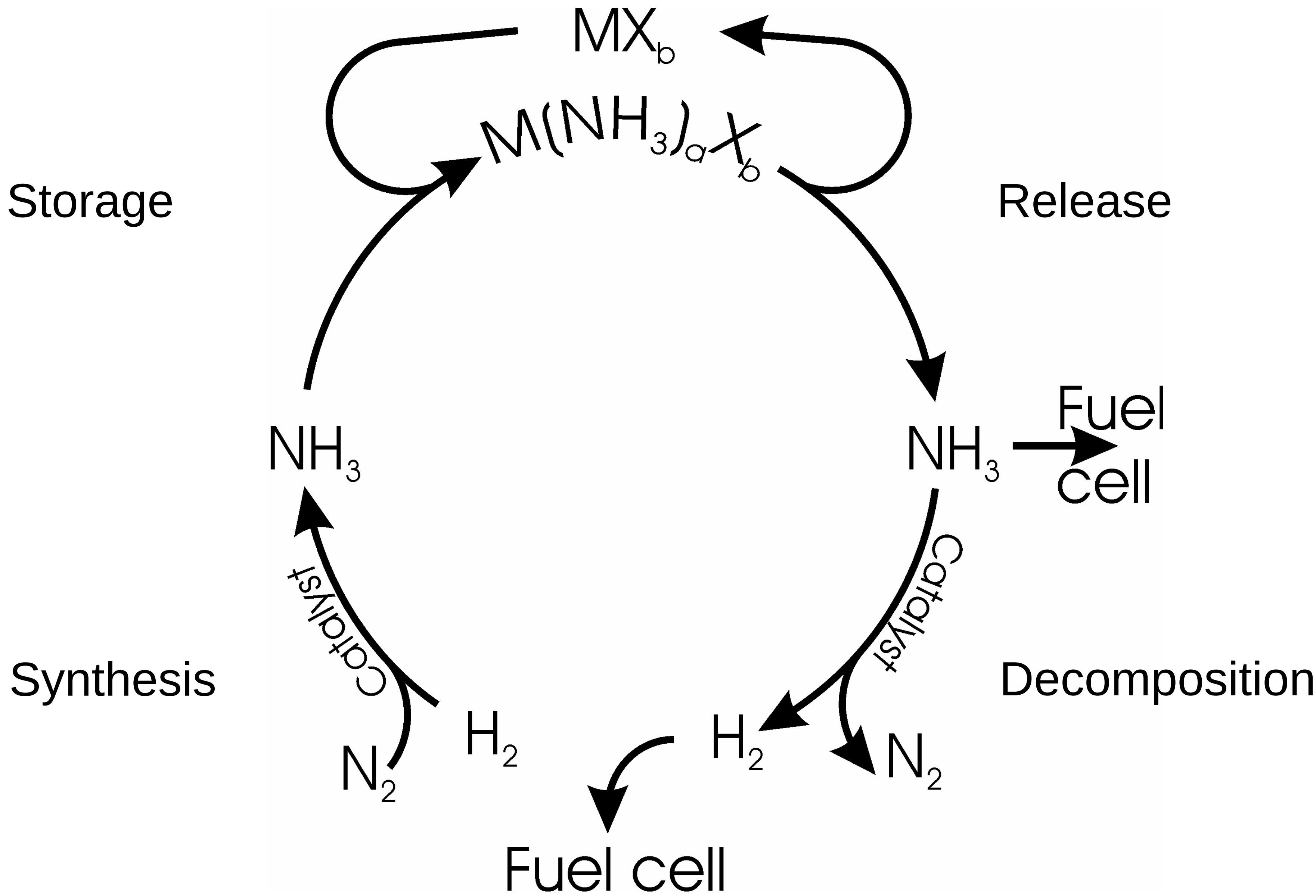
Danish National Research Foundation´s
Center for Sustainable and Green Chemistry

Technical University of Denmark DTU

Introduction



NH_3 for hydrogen storage

Table 1. Properties of ammonia

M_r	17.0312 g/mol
Liquid density	
(at –33.43 °C, 101.3 kPa)	0.682 g/cm^3

Ullmann's Encyclopedia of Industrial Chemistry

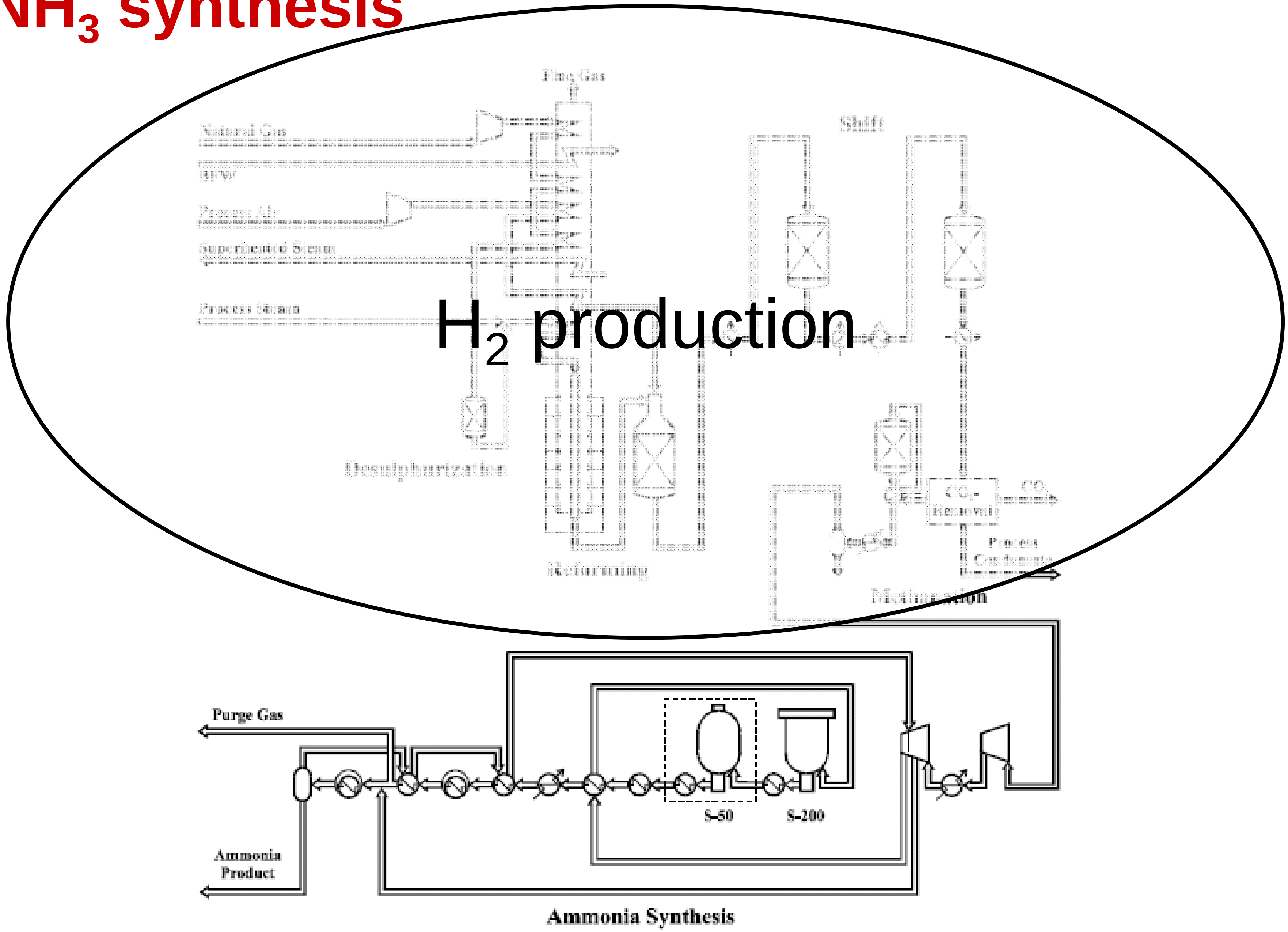
Hydrogen storage in liquid ammonia?

17.6 % w/w H

120.3 kg H / m^3

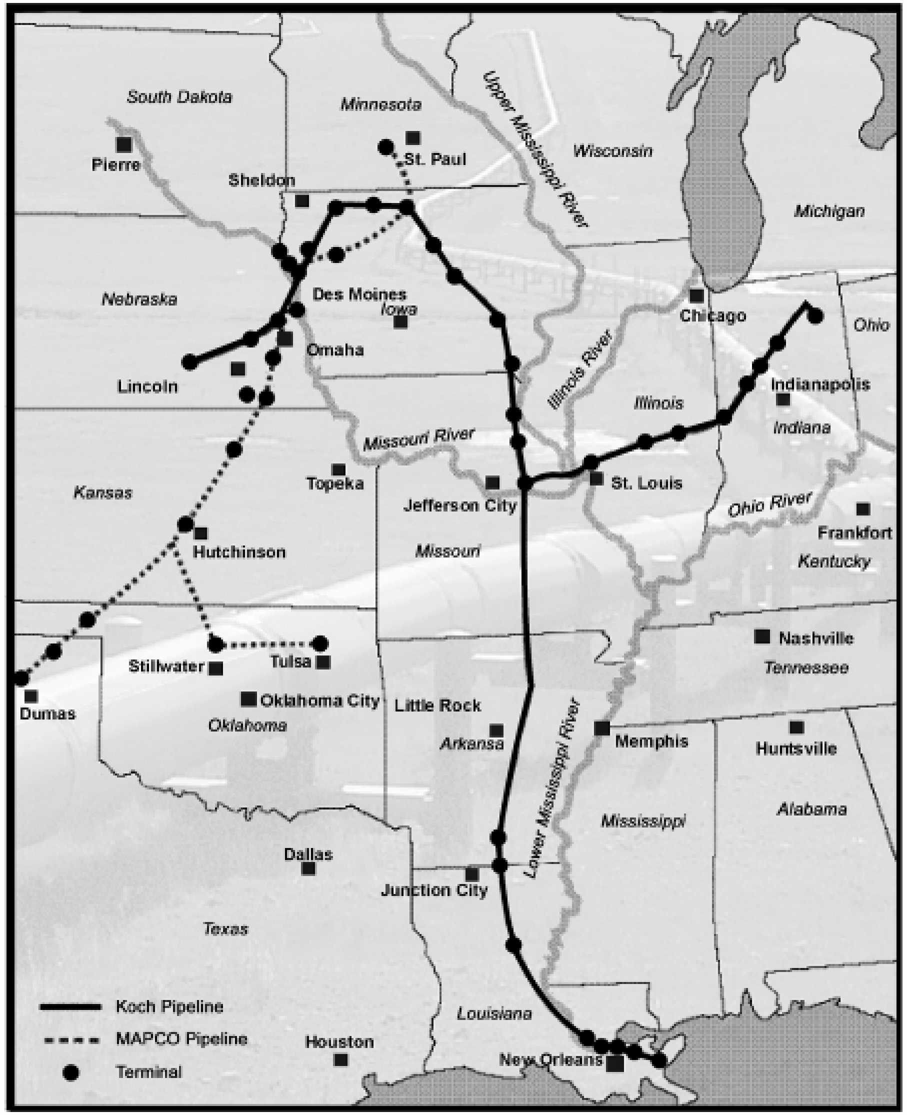
liquid at RT and 8 bar

NH_3 synthesis



Haldor Topsøe A/S

NH_3 infrastructure



- Ammonia pipelines
- Further transportation by rail or truck
- Storage tanks at -33°C with capacities up to 50.000 tonne

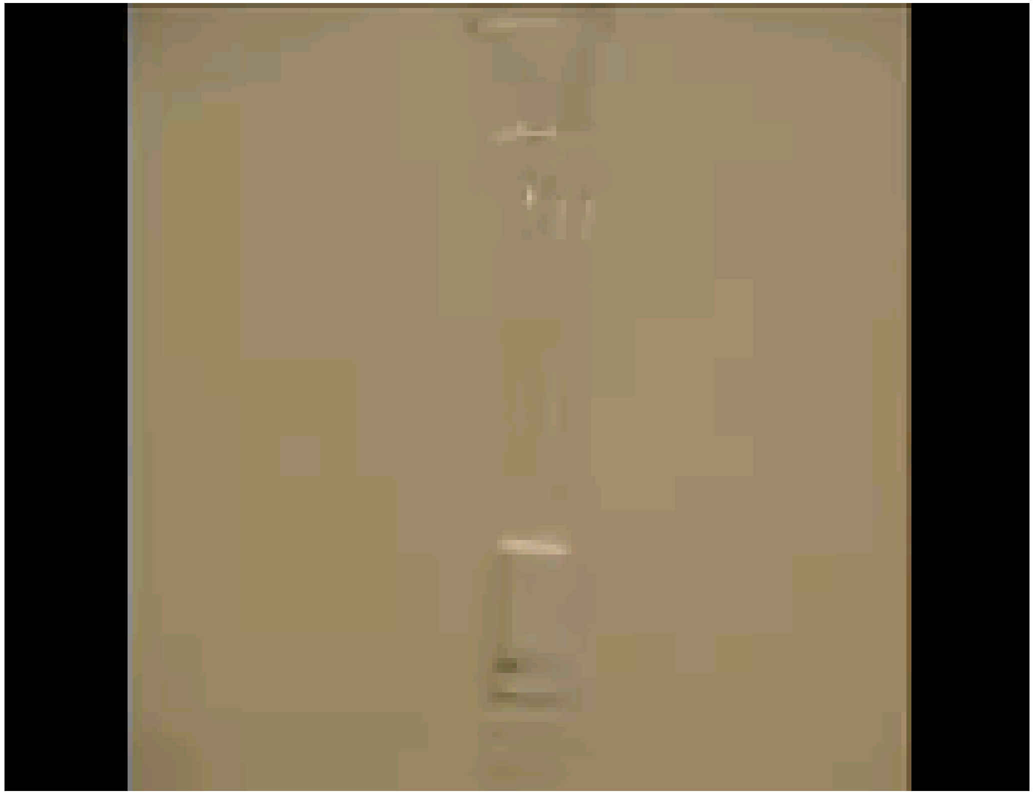
NH_3 safety

	Vapor pressure, p_i @ 293K (bar)	IDLH (NIOSH)* ppm	Relative volatility $p_{i,293K}$ /IDLH
Methanol	0.13	6000	21.6
Liquid ammonia	~ 8	300	~ $2.7 \cdot 10^4$
Window cleaning fluid (pH≈11)	$2 \cdot 10^{-3}$	300	6.0
Ammonia ($Mg(NH_3)_6Cl_2$)	$1.4 \cdot 10^{-3}$	300	4.65
Ammonia ($Ni(NH_3)_6Cl_2$)	$2 \cdot 10^{-4}$	300	0.68

*NIOSH Immediately Dangerous To Life or Health Concentration

Improving safety

$$CuSO_4 + xNH_3 \longrightarrow Cu(NH_3)_xSO_4$$



NH_3

Compression

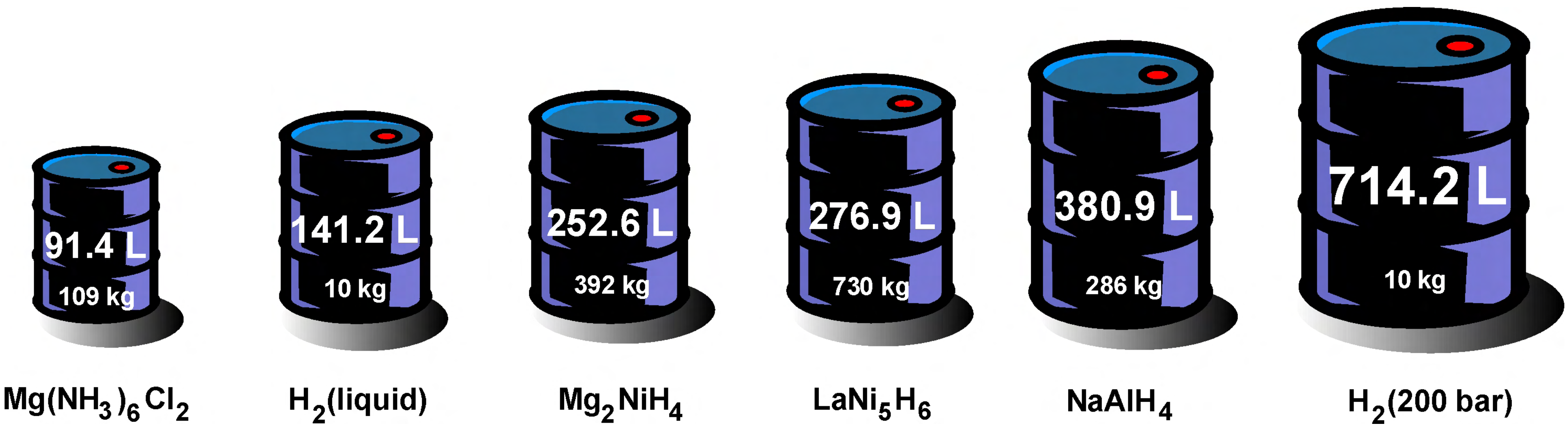
$Ca(NH_3)_8Cl_2$
1.18 g/cm^3
9.78 wt % H
0.12 kgH/L

$Mn(NH_3)_6Cl_2$
1.34 g/cm^3
7.96 wt%H
0.11 kgH/L

$Ni(NH_3)_6Cl_2$
1.41 g/cm^3
7.83 wt% H
0.11 kgH/L

Hydrogen density of safe storage

Storage of 10 kg H by 6 methods:



•Mosher, D. et al. *High Density Hydrogen Storage System Demonstration Using NaAlH4 Based Complex Hydrides*, FY 2006 Annual Progress Report, DoE Hydrogen Program, **2006**, 281-284

•Liu, X. et al. *Int. J. Hydrogen Energy*, **2007**, article in press, DOI: 10.1016/j.ijhydene.2006.09.037

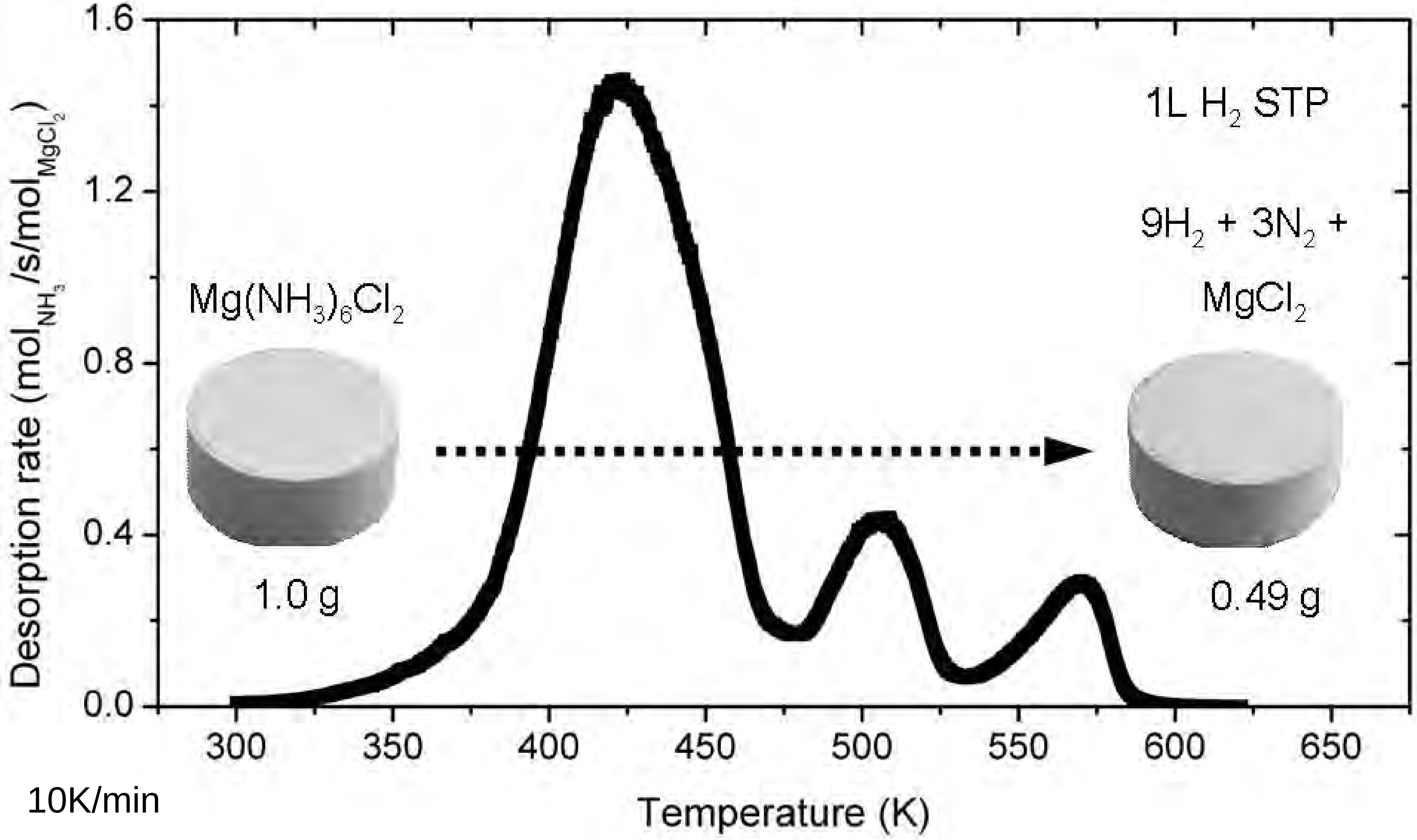
•Nomura, K. et al. *Journal of the Less-Common Metals*, **1991**, *169*, 9-17.

•Suissa, E. et al. *Journal of the Less-Common Metals*, **1984**, *104*, 287-295

•El-Osairy, M. A. et al. *Int. J. Hydrogen Energy*, **1993**, *18* (6), 517-524.

•Laidler, K. J.; Meiser, J. H. Physical Chemistry, third edition, New York, Houghton Mifflin Company, **1999**.

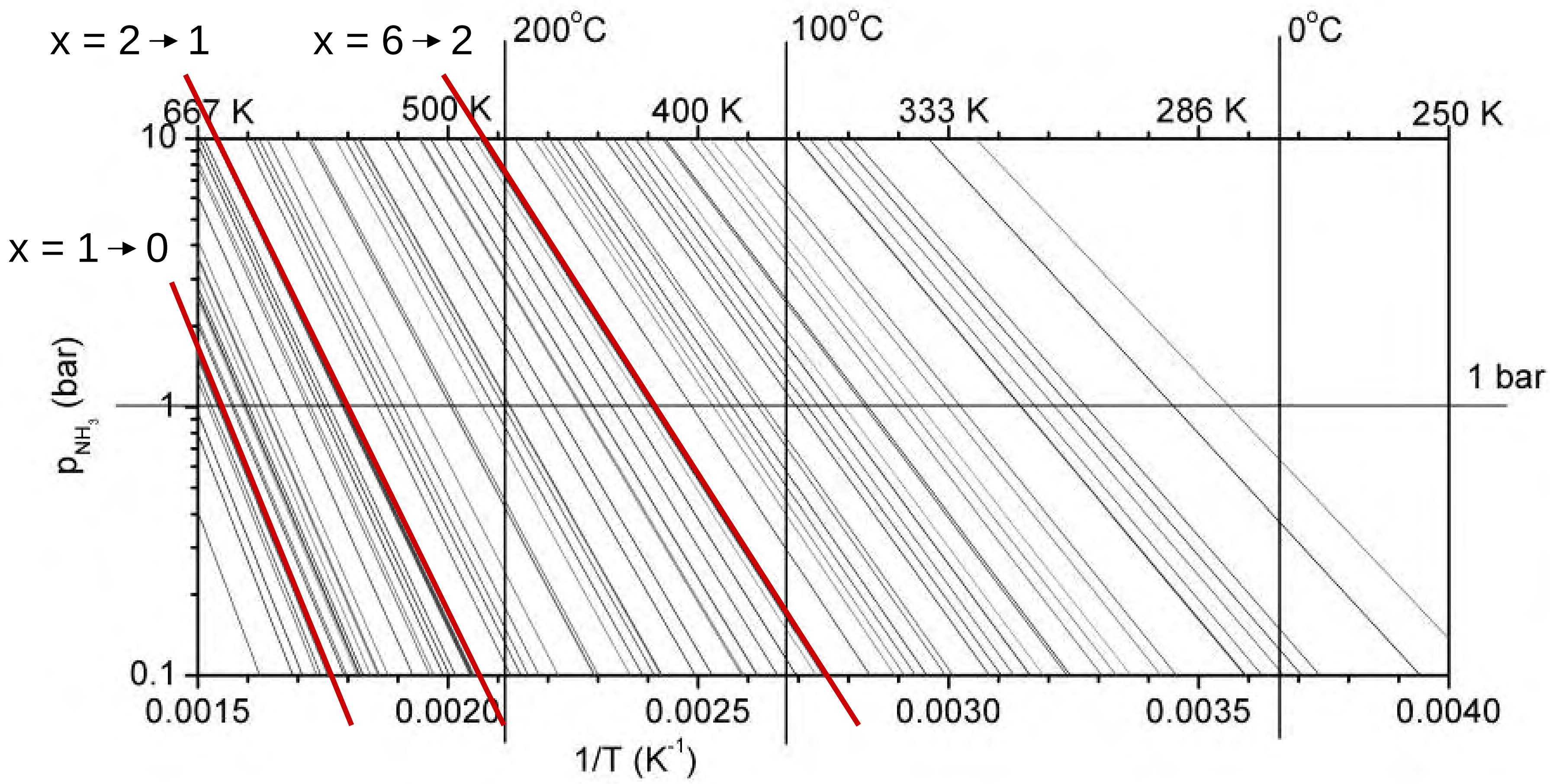
Desorption of NH_3



10K/min

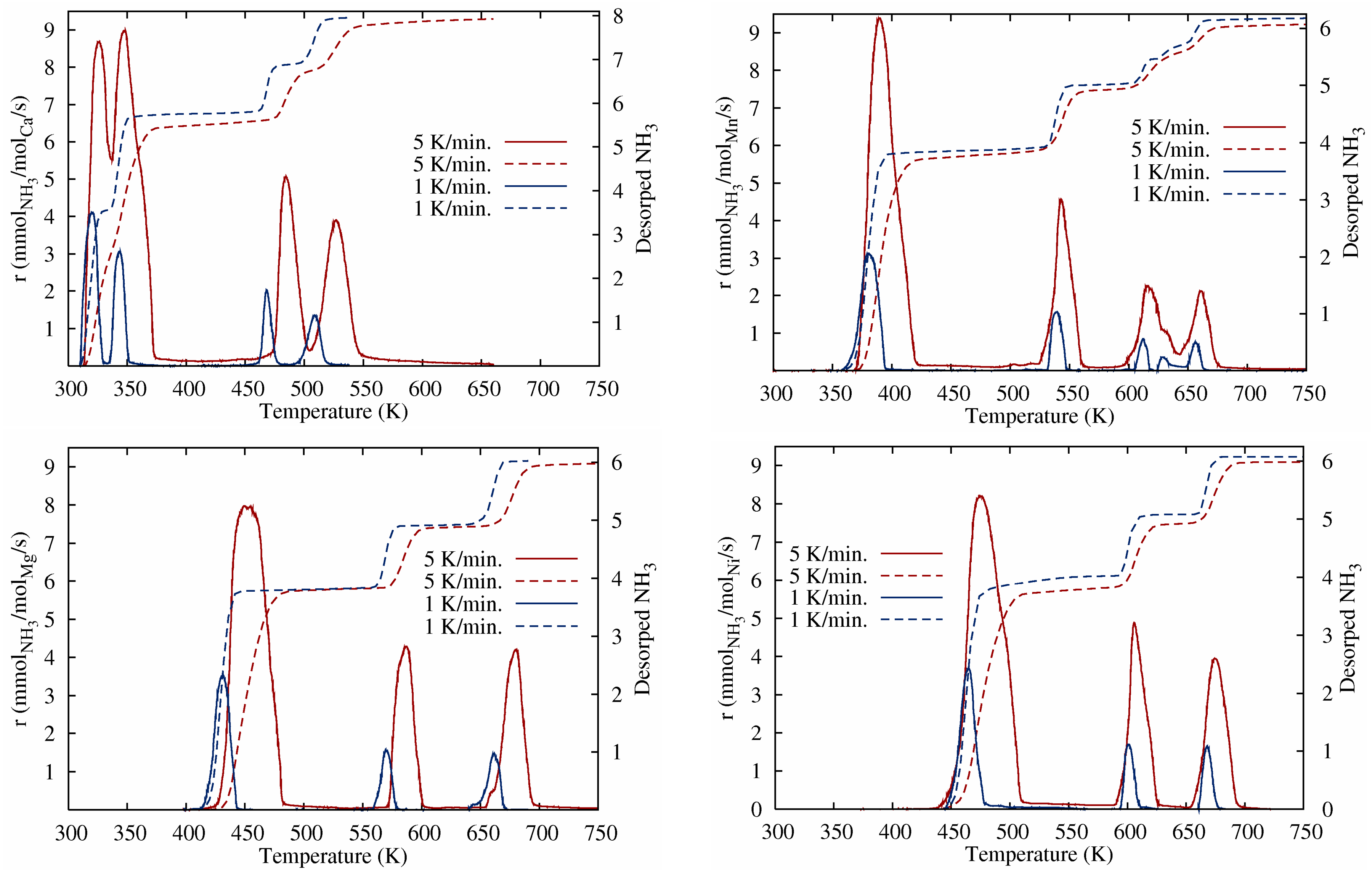
Variations in ΔH

- $Mg(NH_3)_xCl_2$

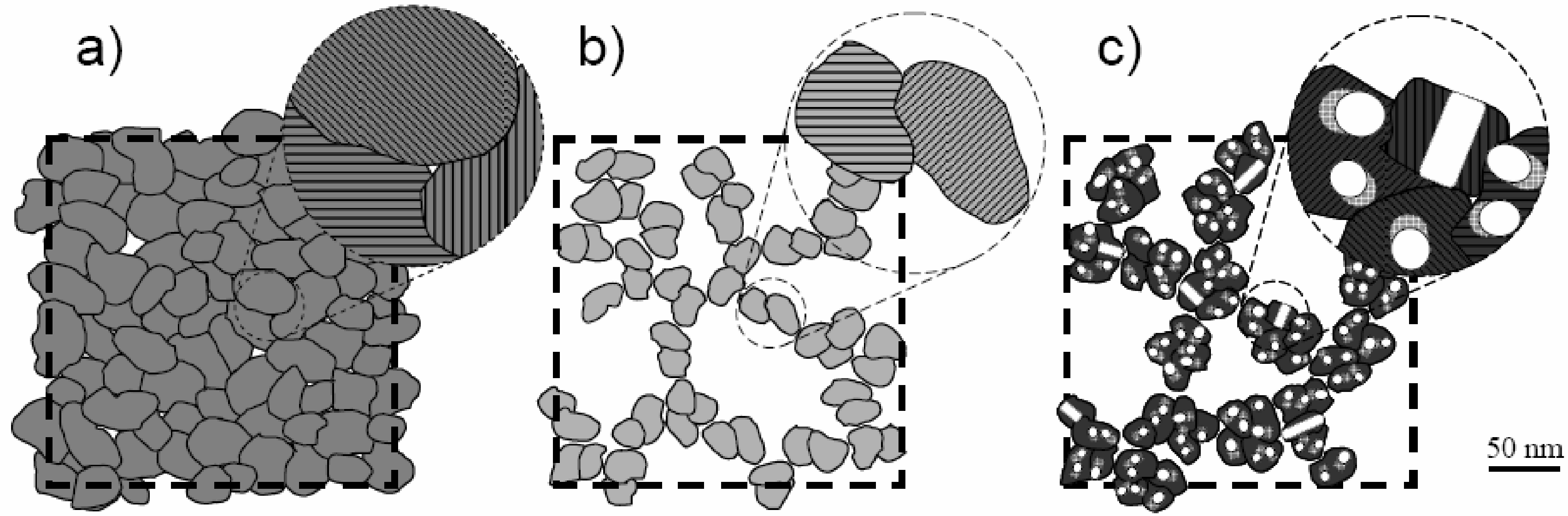


Biltz, W. *Z. Anorg. Allgem. Chem.*, **1923**, *130*, 93-139.
Lepinasse, E.; Spinner, B. *Rev. Int. Froid.*, **1994**, *17*, 309-321.

Examples

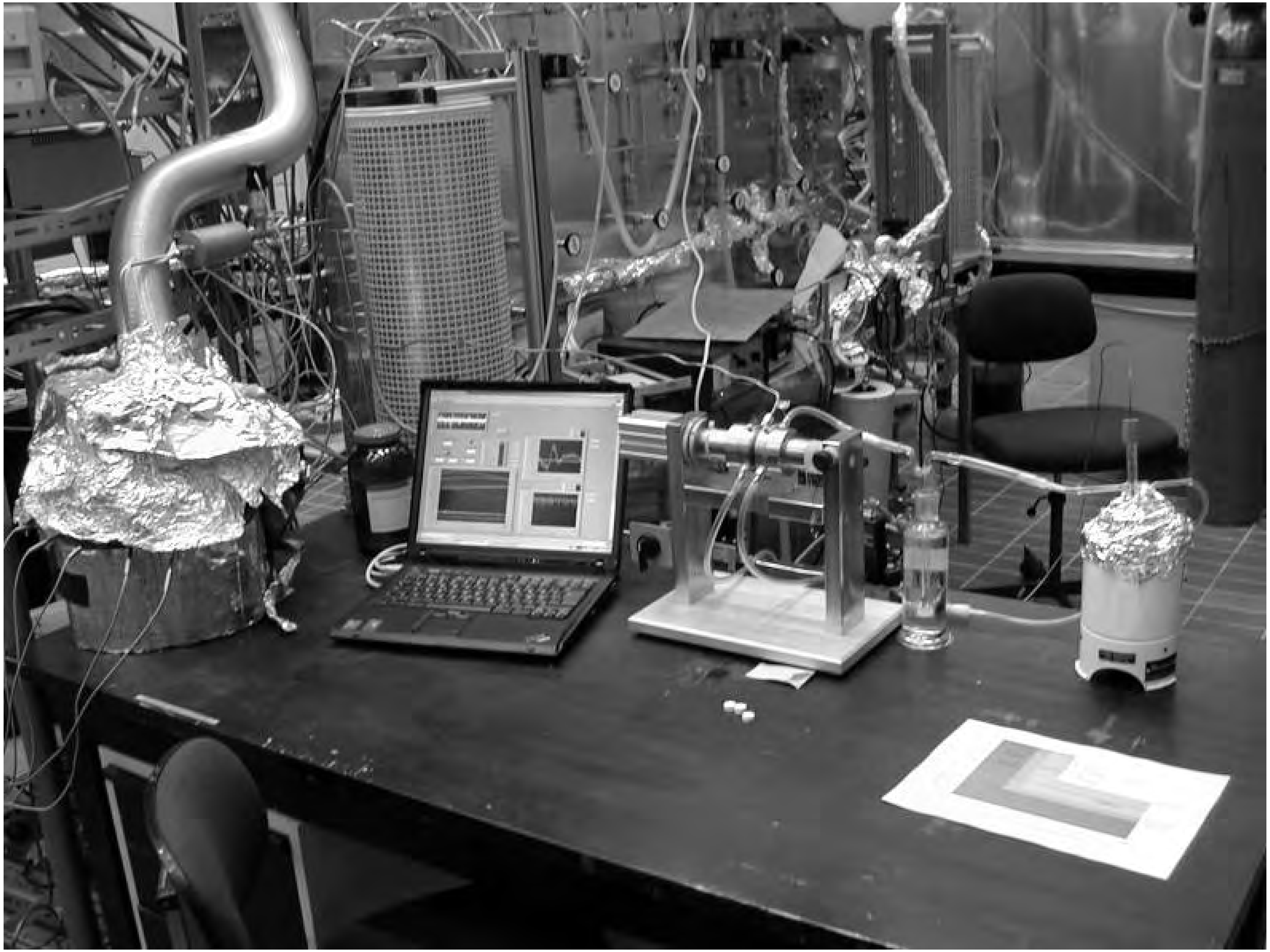

r (mmol$_{NH_3}$/mol$_{Ca}$/s)
Desorped NH_3
Temperature (K)
5 K/min.
5 K/min.
1 K/min.
1 K/min.
r (mmol$_{NH_3}$/mol$_{Mn}$/s)
Desorped NH_3
Temperature (K)
r (mmol$_{NH_3}$/mol$_{Mg}$/s)
Desorped NH_3
Temperature (K)
r (mmol$_{NH_3}$/mol$_{Ni}$/s)
Desorped NH_3
Temperature (K)

Pore size development



The porous system which arise from the lattice contraction in the polycrystalline $Mg(NH_3)_XCl_2$ salt.
a) x=6, b) x=2, c) x=0

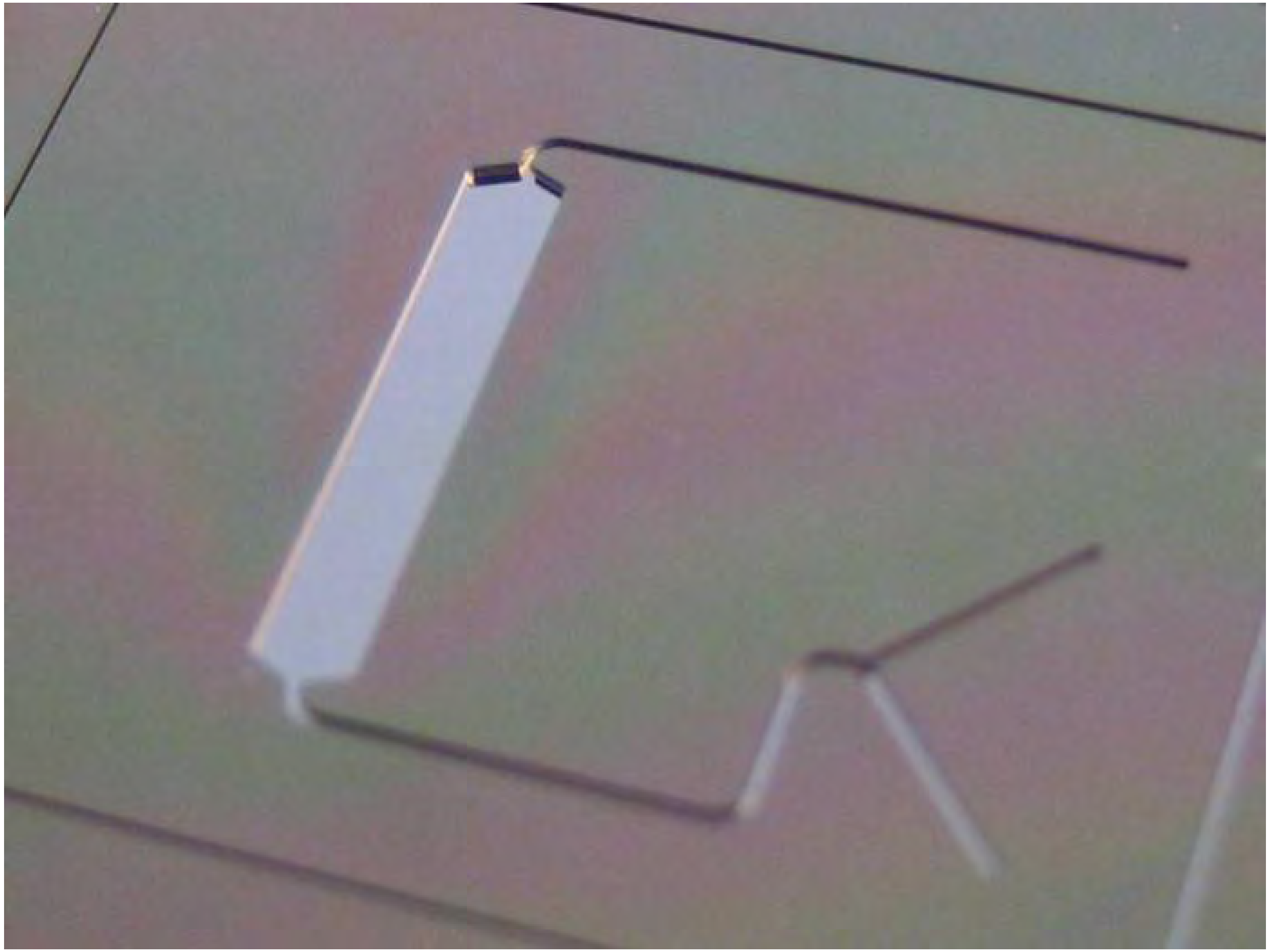
Energy from PEM



Practical dosing

Micro-reactors

8.0mm x 1.5mm x 0.3mm (3.6μL reactor volume)



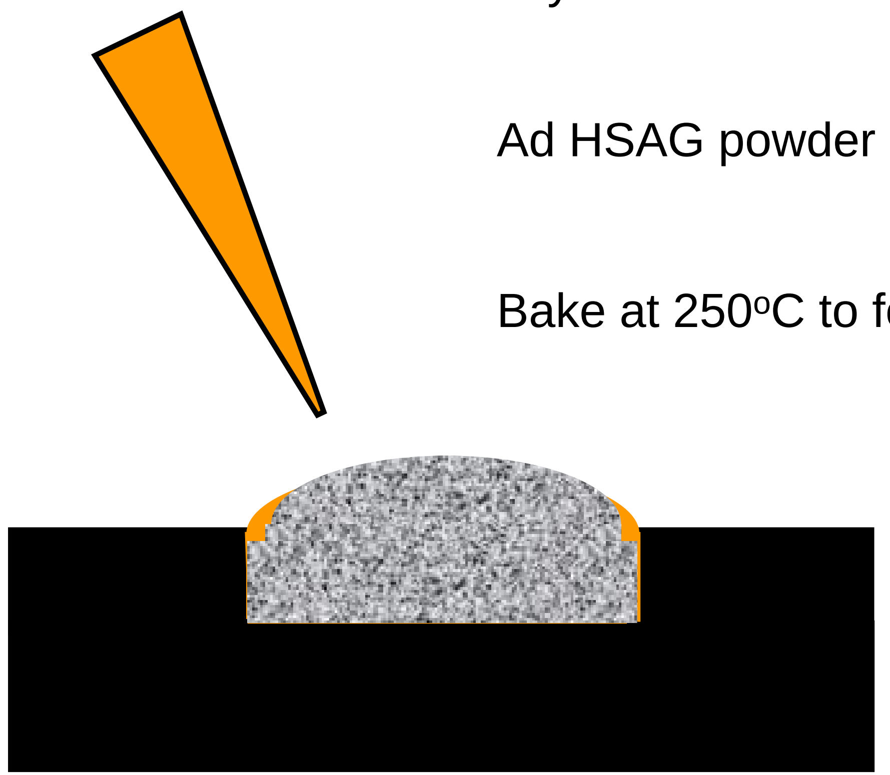
Deposition method for HSAG

Fill with sucrose solution (140g/l)

Dry at 80^{o}C

Ad HSAG powder

Bake at 250^{o}C to form final support

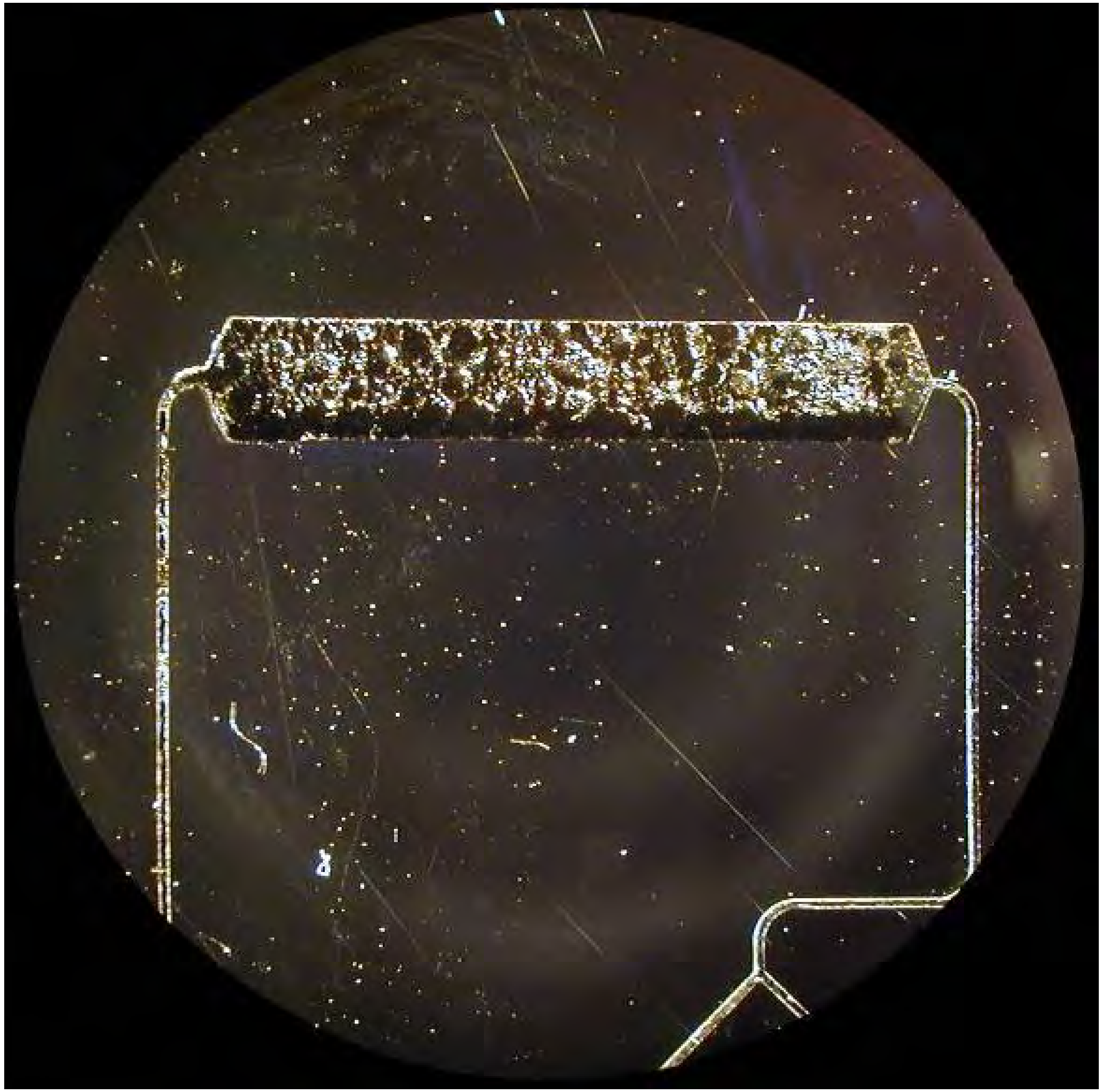


Porosity of the graphite support

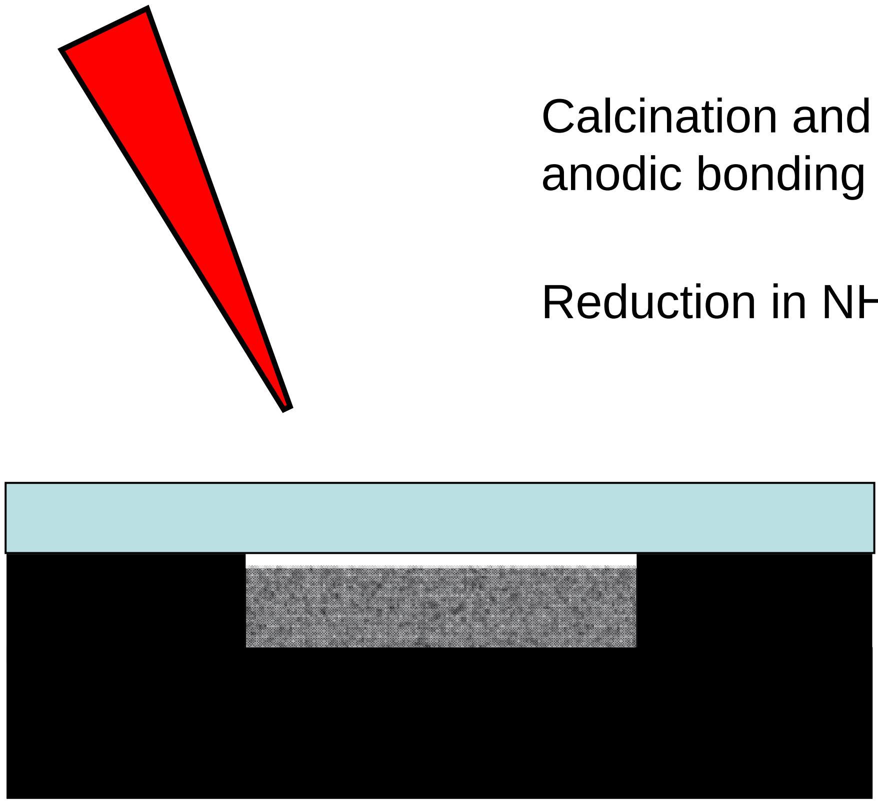
Timrex HSAG:
322m^2/g

Final support:
167m^2/g

Mass fraction sucrose:
~40%



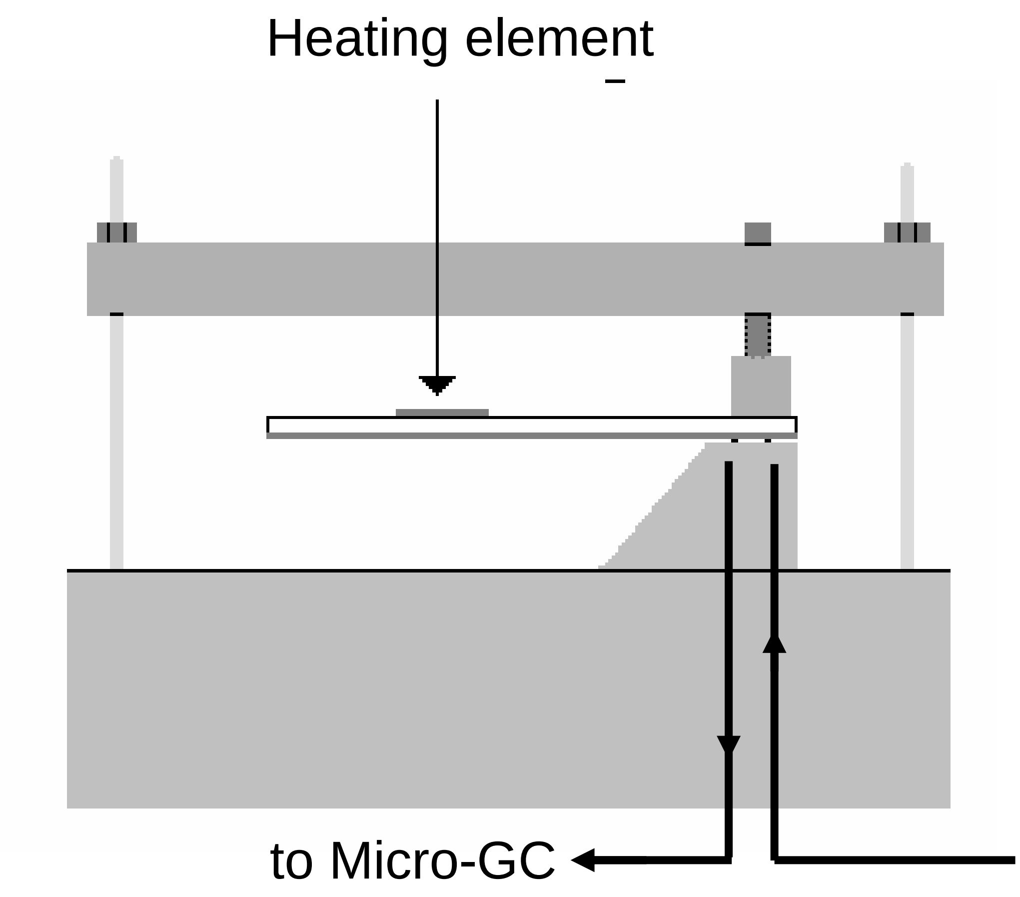
Impregnation and bonding

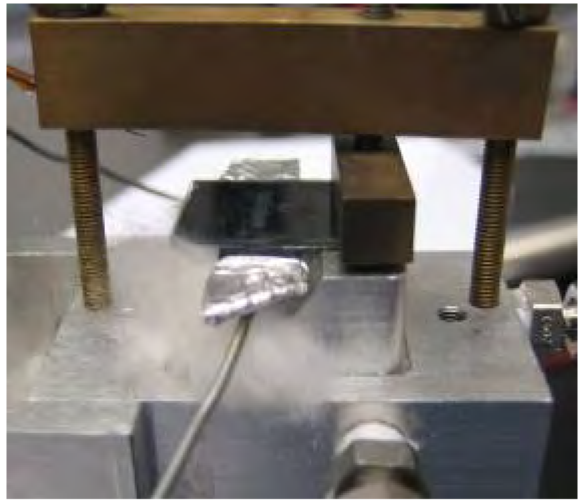


Calcination and sealing by anodic bonding at 320°C

Reduction in NH_3 flow

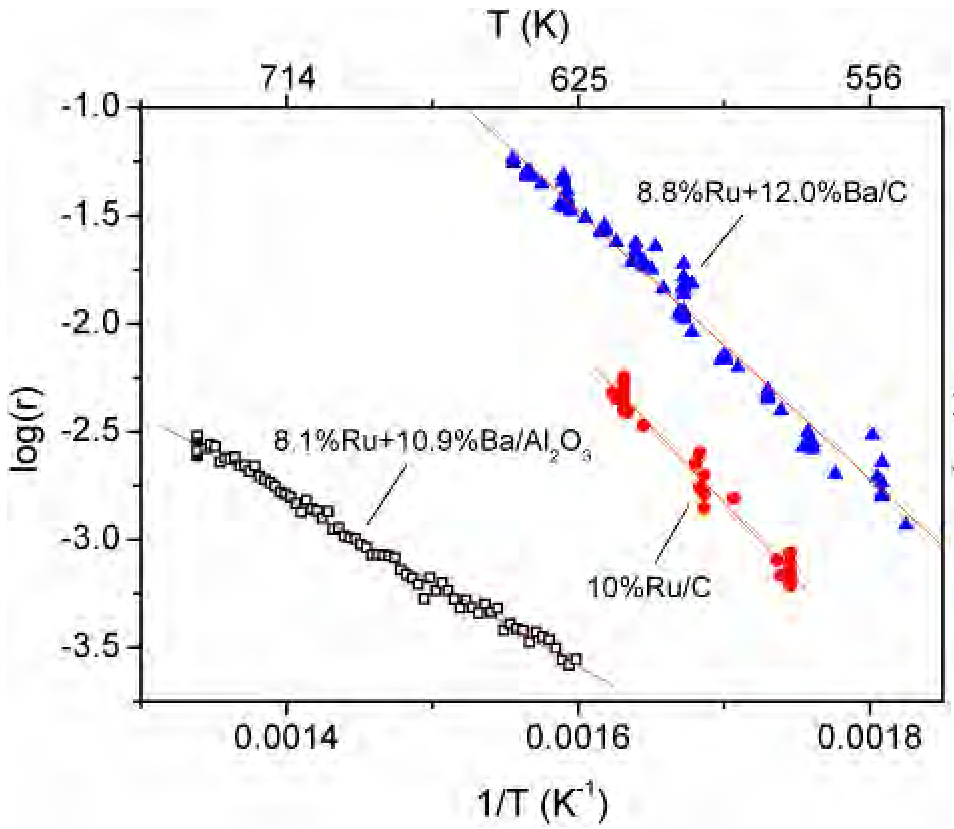
Catalyst testing

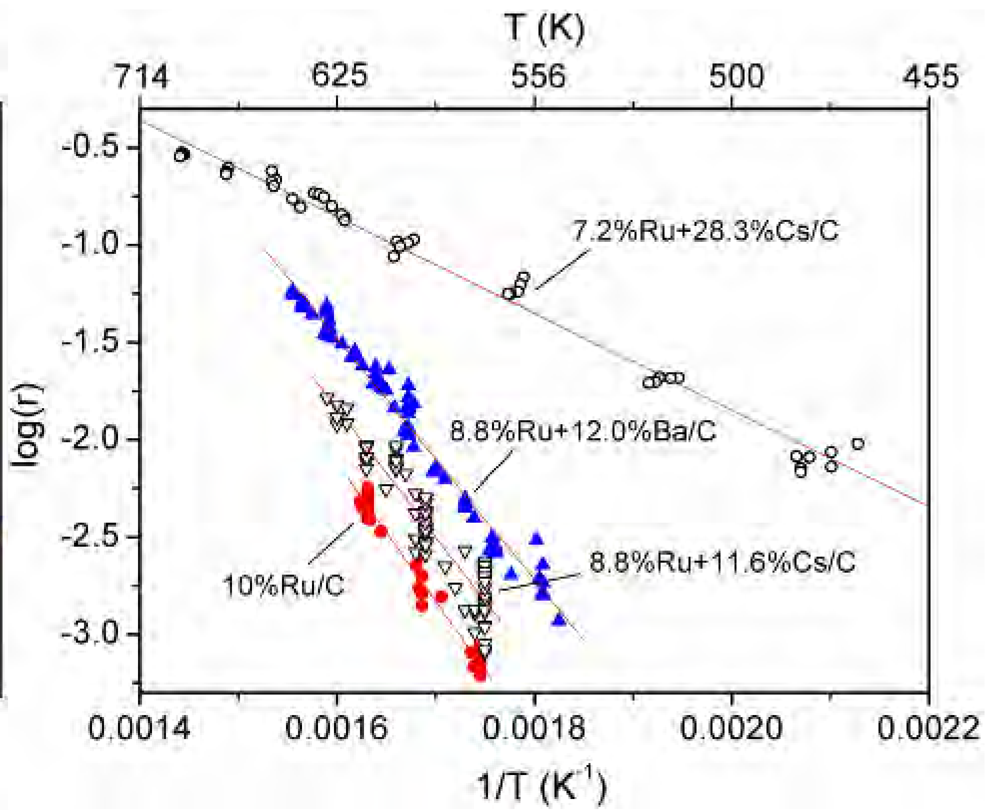




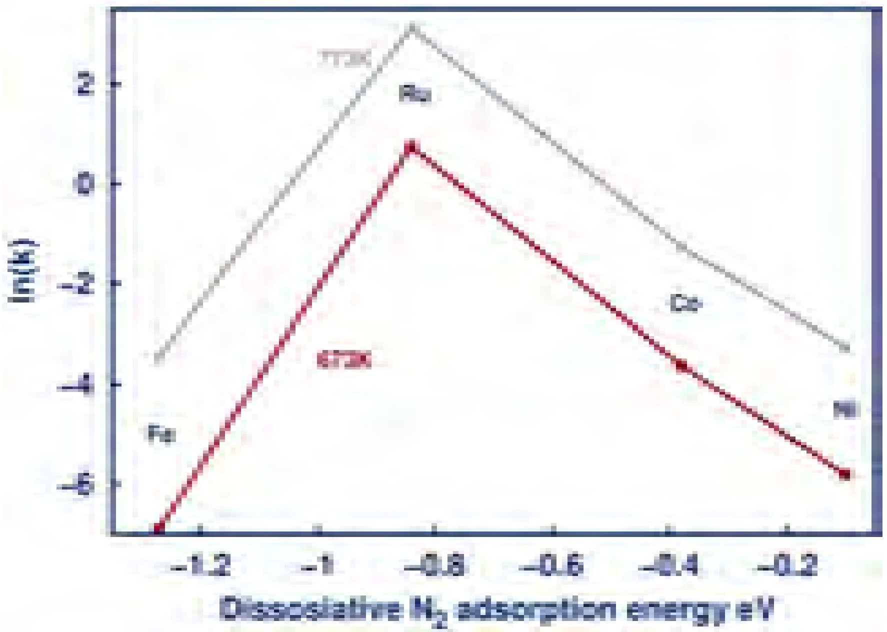
Each catalyst tested at 2mL/min

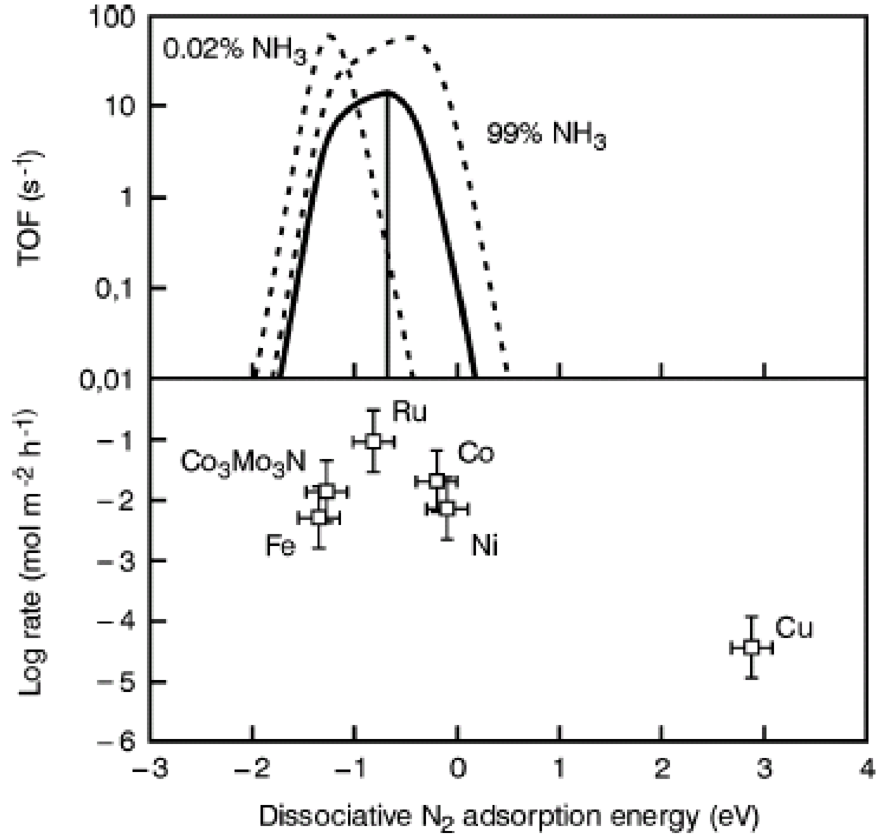
High activity catalysts





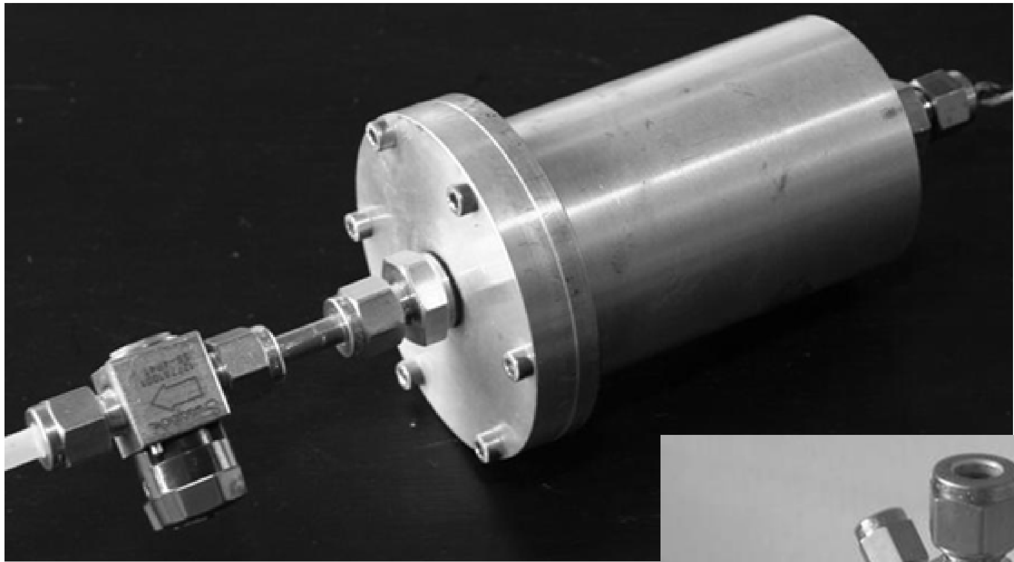
Activity trend reproduced



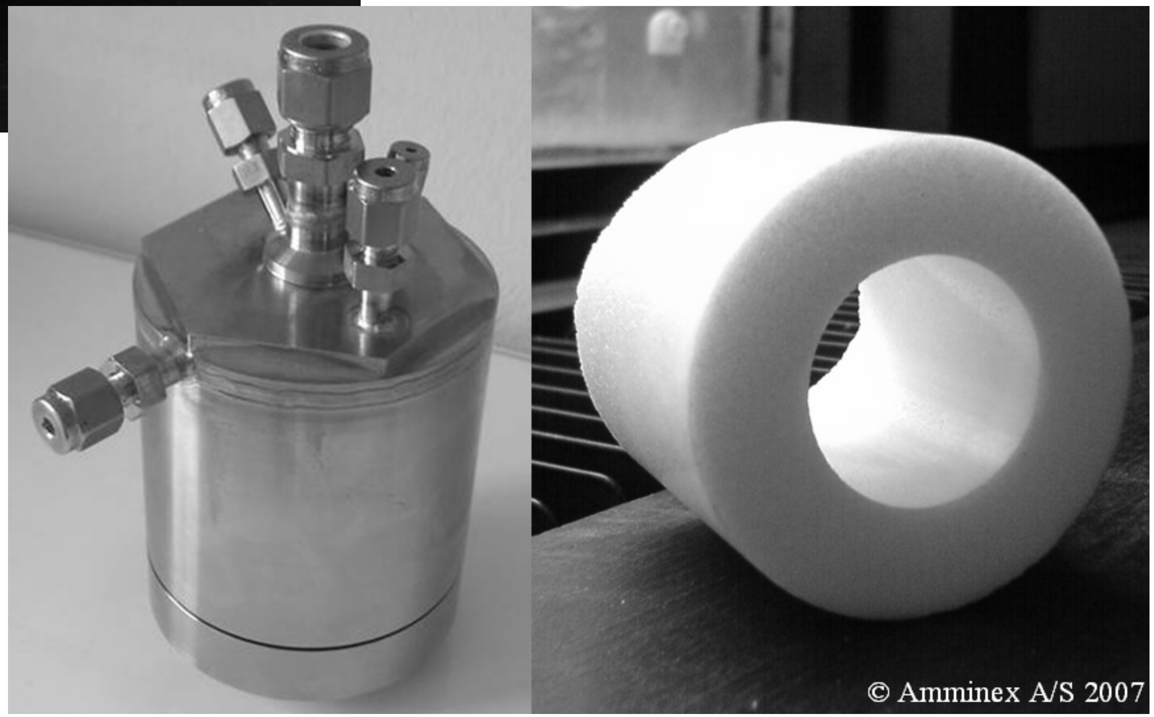


A. Boisen *et al.* J. Catal. 2005, 230, 309-312

Application of metal ammines

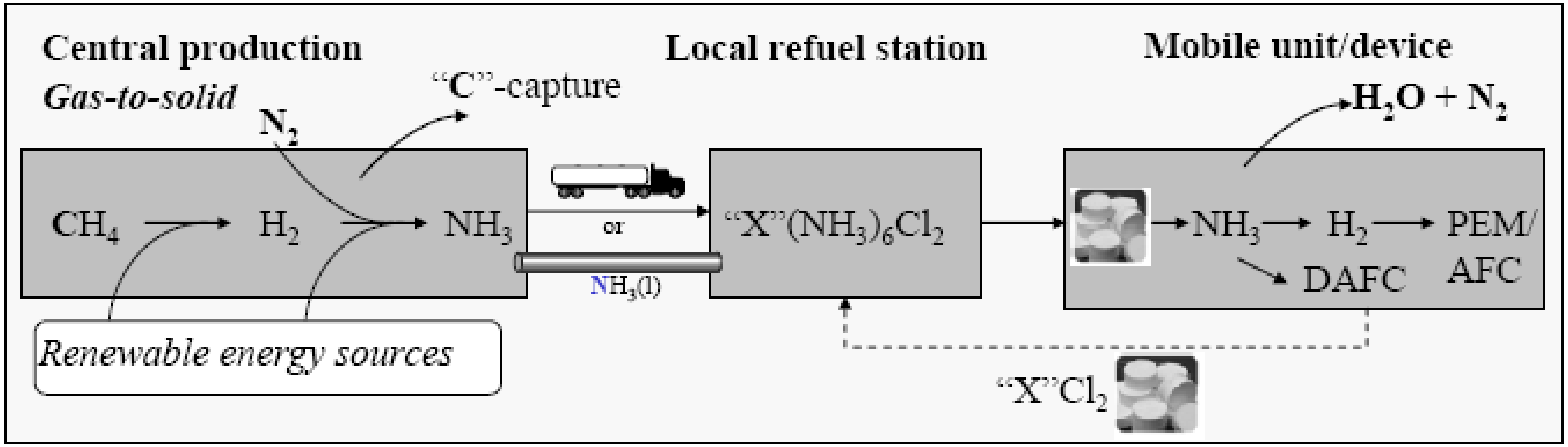


Plug and play ammonia gas generator for production of 1-1.2 L/min., power consumption 40-45W.

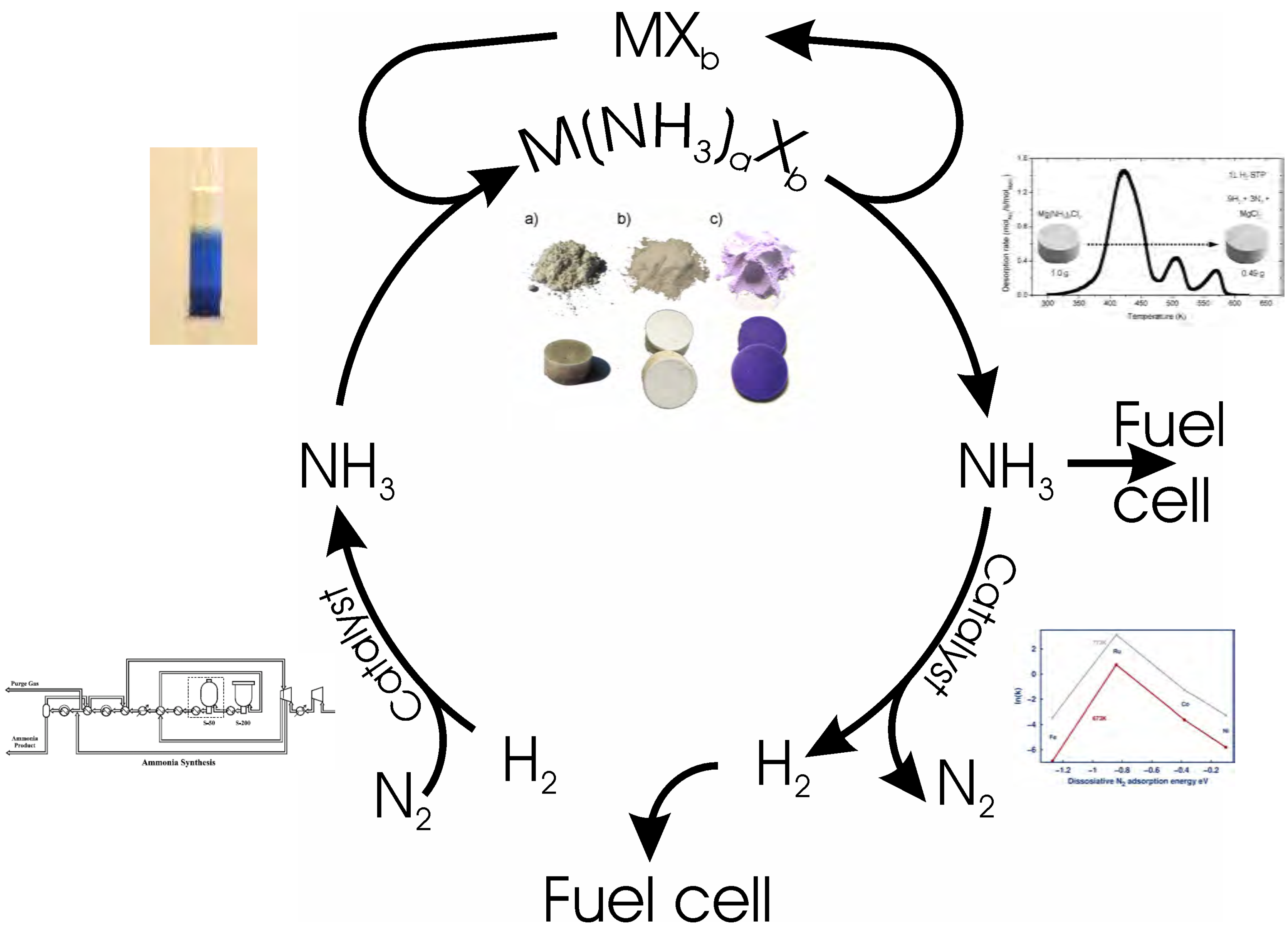


Integrated hydrogen generator based on metal ammine, integrated with a ammonia cracker

Ammonia distribution path



Conclusions



Acknowledgements

Danish National Research Foundation´s
Center for Sustainable and Green Chemistry

Claus Hviid Christensen

AMMINEX

Tue Johannessen
Henning Schmidt

Center for Individual Nanoparticle Functionality

Ulrich Quaade

Desorption data

Salt	Highest wt%H	T for 1 bar (°C)		ΔH at coordination nr.:							
	< 1bar at 0ºC	Initial	Final	9	8	6	5	4	3	2	1
$CaCl_2$	9.8	32	242		41.0			42.3		63.2	69.1
LiCl	9.7	61	114						44.8	48.1	51.9
$MgCl_2$	9.2	142	375			55.7				74.9	87.0
$SrCl_2$	8.2	35	85		41.4						48.1
$MnCl_2$	8.0	80	354			47.4				71.0	84.2
$FeCl_2$	7.9	109	374			51.3				76.2	86.9
$NiCl_2$	7.8	168	396			59.2				79.5	89.8
$CoCl_2$	7.8	129	384			54.0				78.1	88.3
LiBr	7.8	45	151					42.7	46.4	49.0	56.9
$CaBr_2$	7.2	32	306		41.0	49.0				71.5	77.8
$BaCl_2$	7.0	8	8		37.7						
$MnBr_2$	6.4	122	351			53.1				77.1	83.8
$MgBr_2$	6.3	201	403			63.6				84.1	90.8
$SrBr_2$	6.3	67	251		45.6					63.6	70.3
LiI	6.0	88	225					48.5	51.0	57.7	66.9
$FeBr_2$	5.7	143	374			55.8				83.1	86.9
$NiBr_2$	5.7	205	374			64.2				85.4	86.9
$CoBr_2$	5.6	163	381			58.6				84.4	87.9
$BaBr_2$	5.6	38	95		41.8			42.7		44.4	49.4
SrI_2	5.1	70	297		46.0	52.7				64.9	76.6
BaI_2	5.0	61	145	41.8	44.8	46.4		47.3		56.1	
MgI_2	4.8	265	431			72.2				94.5	

Future Energy - Chemical Solutions
Date:12-14 September 2007

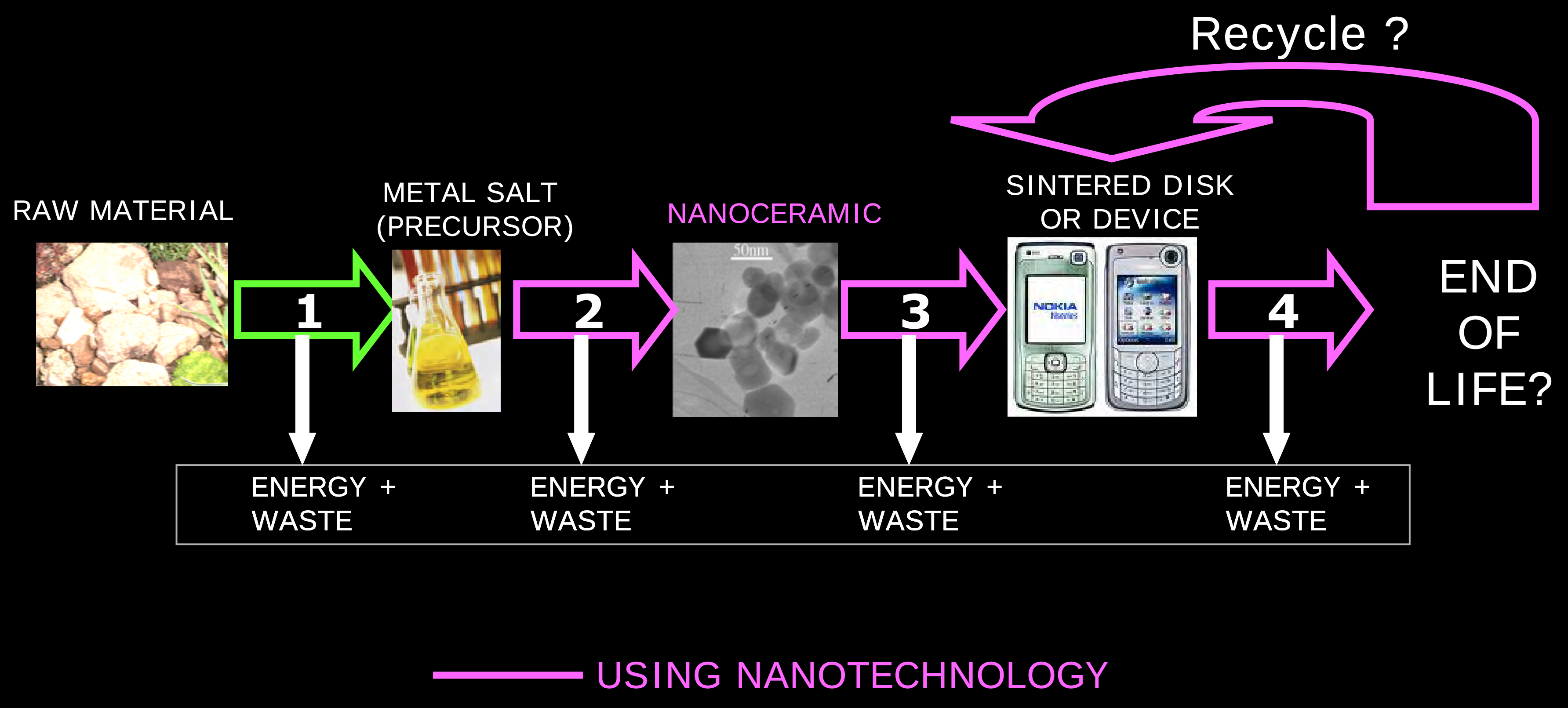
High Throughput Nanoceramics Discovery; Efficient Methods for Making Energy Materials

Jawwad A. Darr
Clean Materials Technology Group
UCL chemsitry

Contents

- Intro: life cycle assessment
- Hypothesis: “bottom up” = efficient ?
- Current: efficient materials syntheses
- Future: materials discovery (photocats)

Life cycle analysis



By building “bottom up”, can we mix things so well that we can....

- reduce total W/ E in the life cycle
- make products / materials faster ?
- reduce no. of manufacturing steps?
- more homogenous/pure phase prod?
- Improved or unique properties?

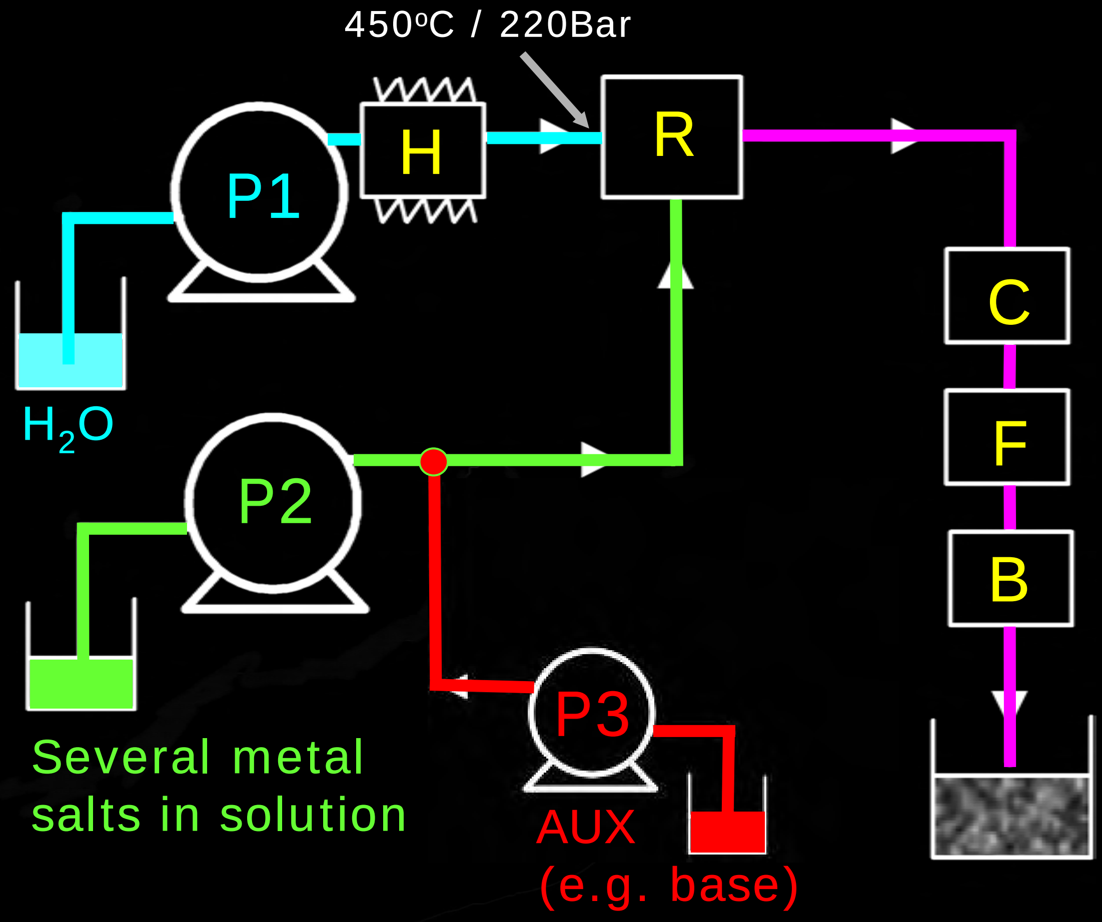
SPEED UP MATERIALS DISCOVERY

Conventional technologies

1. Ball milling
2. Co-precipitation
3. Sol-gel
4. Plasma spray (Qinetic)
5. Thermal spray
6. MBE methods
7. Emulsion methods
8. Hydrothermal batch

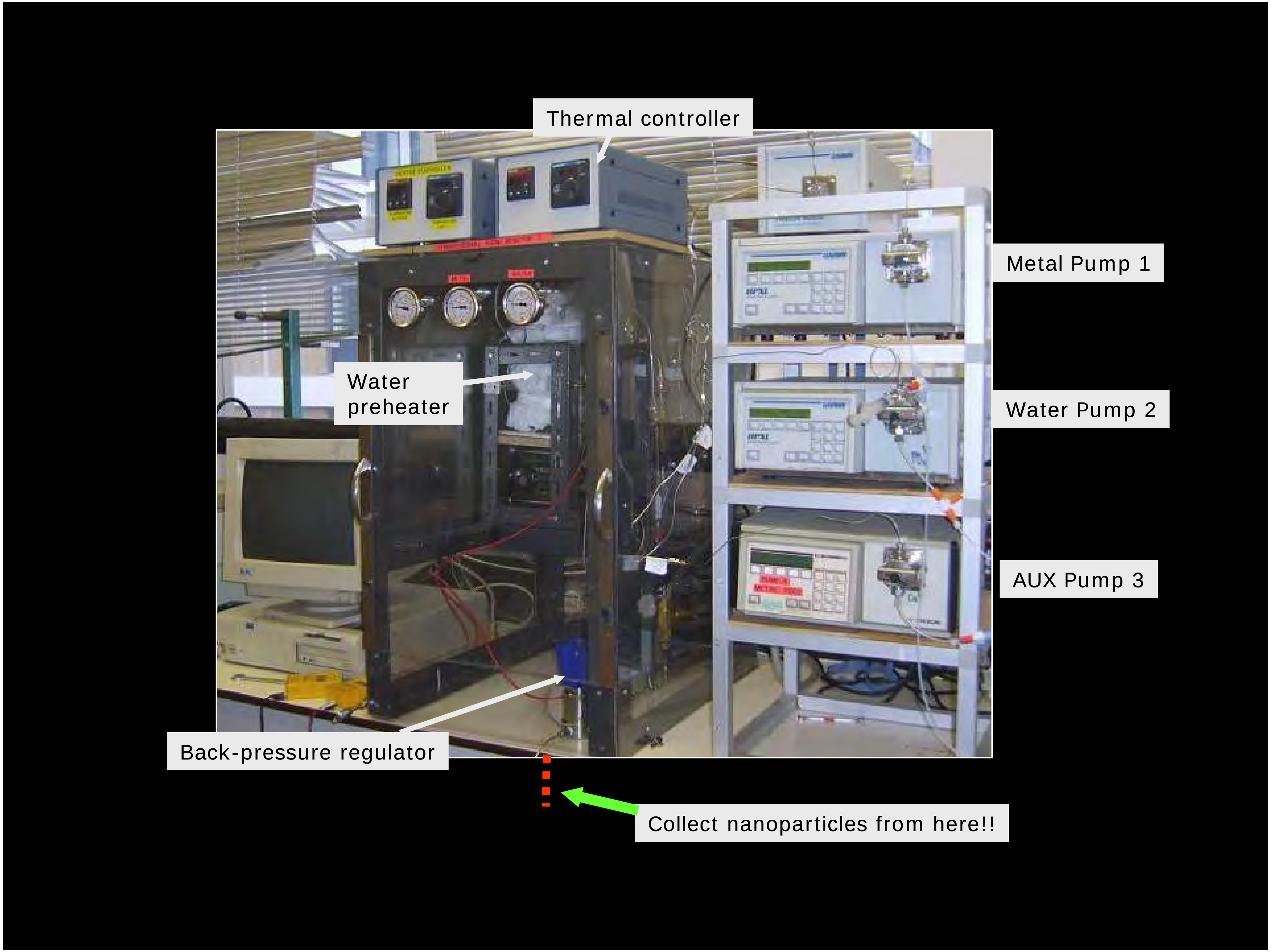
Hydrothermal flow systems for synthesis of nanoparticles

Continuous Hydrothermal Nanomaterials Syntheses



- Nanoparticles
- Rapid, direct
- Crystalline
- High SA
- Novel Materials

Key: P = HPLC pump; H = water pre-heater; R = Reactor; C = cooling; F = Filter; B = back pressure regulator



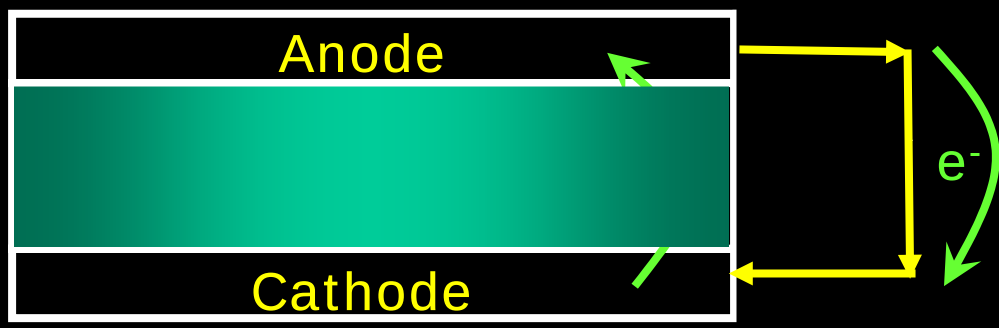
Thermal controller
Metal Pump 1
Water preheater
Water Pump 2
AUX Pump 3
Back-pressure regulator
Collect nanoparticles from here!!

Current Nano Research

- SOFC components
- Li battery materials
- Catalysts / Photocats
- Sunscreens, Bioceramics
- Dielectrics / semiconductors
- Magnetics, Metallics, alloys
- Ceramic inks

Solid Oxide Fuel Cells

$CO + H_2 + 2O^{2-}$ $CO_2 + H_2O + 4e^-$



$O_2 + 4e^-$ $2O^{2-}$

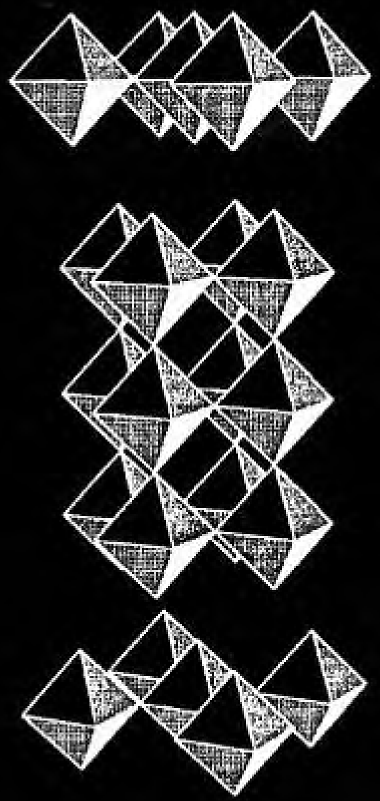
Nanomaterials for SOFCs

"Use of nanoparticles for electrolytes can give better packing and denser materials with no pores"

"Use of nanoparticles also lowers the required sintering temperature which enables the electrolyte layers to better withstand rapid temperature flucutations"

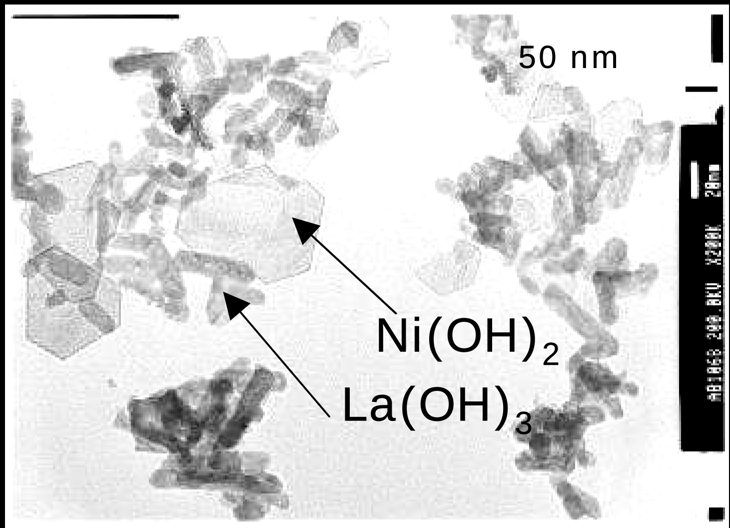
R.M. OMEROD, Chem Soc Rev 2003, 31, 17-28

$La_{n+1}Ni_nO_{3n+1}$ series



- Varying ion and e- conduction
- best balance is $La_4Ni_3O_{10}$ (n=3)
- SS reaction / E intensive
- > 14 days at 1075 °C
- multiple regrinding steps

Efficient Synthesis of $La_4Ni_3O_{10}$

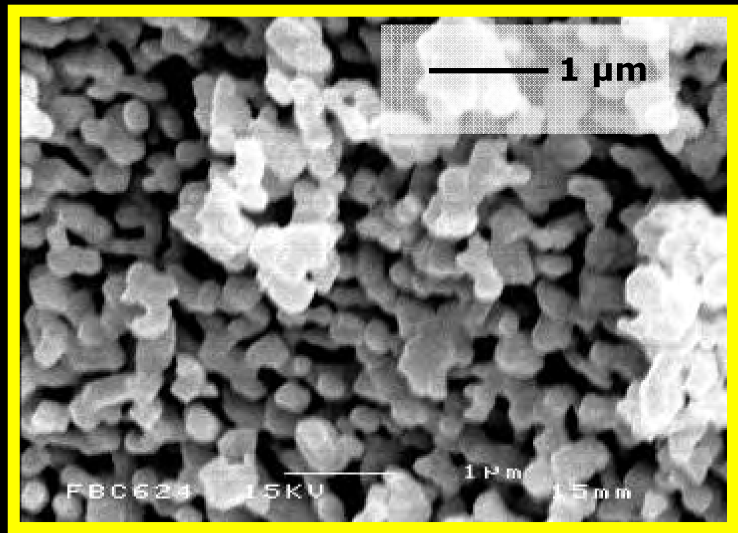


Co-precipitate from CHFS

1075 °C

12 hrs

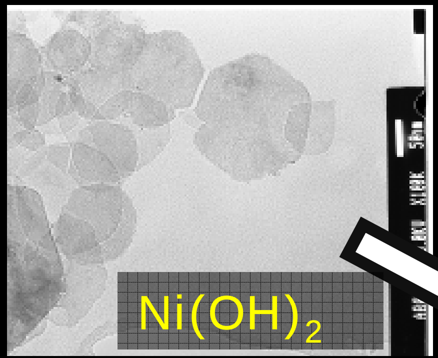
Single step

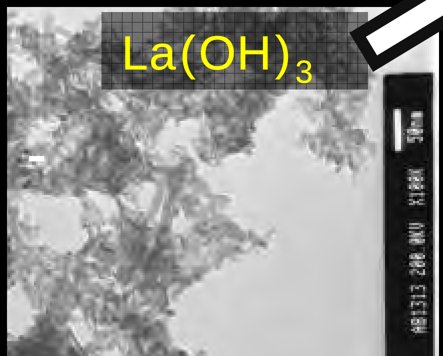


$La_4Ni_3O_{10}$ (F*mmm*)

"Direct Syntheses of Mixed Ion and Electronic Conductors La4Ni3O10 and La3Ni2O7 from Nanosized Co-precipitates", Weng, X.; Boldrin P.; Abrahams, I.; Skinner, S.J.; Darr, J.A. *Chem. Mater.*, **2007,** *19*(18) 4382 – 4384. DOI: 10.1021/cm070134c

Ultrasonic Mixing of Nanoparticles

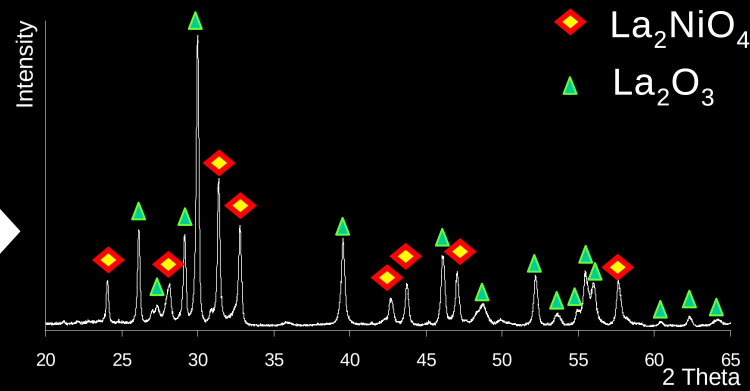




Ultrasonic mixing

1075 °C

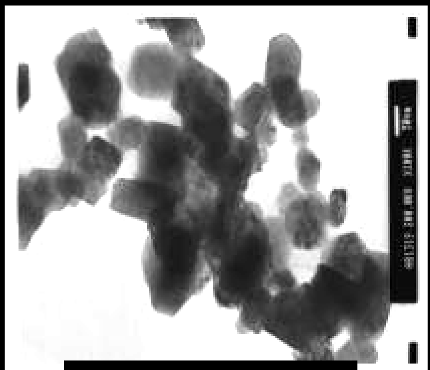
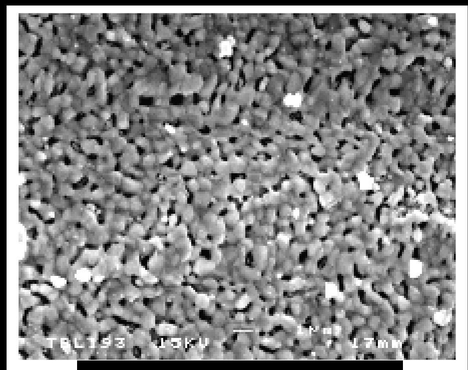
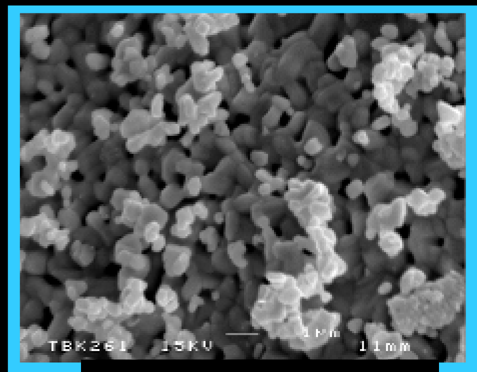
12h



NO $La_4Ni_3O_{10}$ phase observed !

$La_{n+1}Ni_nO_{3n+1}$ series

Co-precipitates from Hydrothermal flow system in the correct La:Ni ratio

750°C 6h	1200ºC 6h	1150ºC 12h	1075ºC 12h
n = ∞	n = 1	n=2	n=3
			
$LaNiO_3$	La_2NiO_4	$La_3Ni_2O_7$	$La_4Ni_3O_{10}$

All conversions require only a single heat treatment

$La_{n+1}Ni_nO_{3n+1}$ Summary

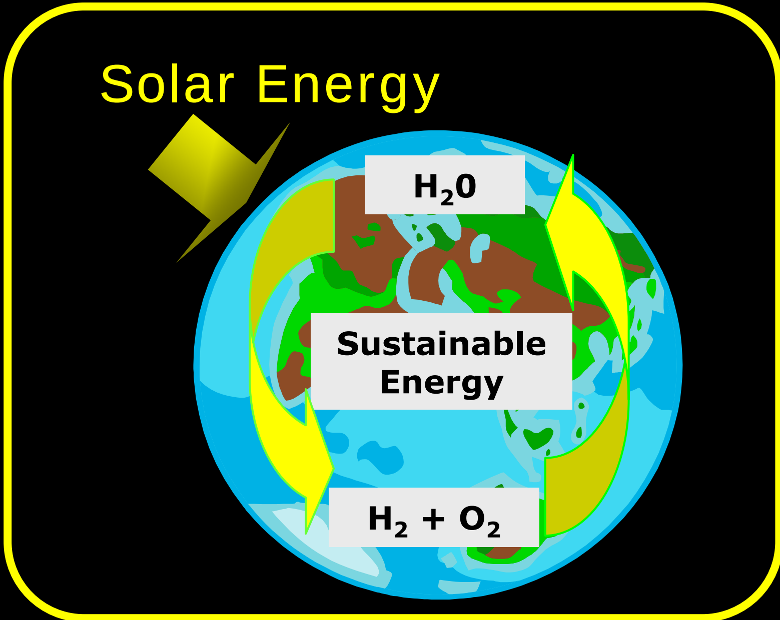
- Intimately mixed precursors
- Reduced steps / no grind
- 5-50x faster
- Use in materials discovery?
- Ion/e- conductivity underway

Future Hydrothermal Research

HT-Nanomaterials Discovery

"To find better photocatalysts to split water"

“High Throughput Nanomaterials Discovery”



EPSRC

Grant reference EP/D038499/1

Hydrogen Solar

SRI International

Malvern

Insight High throughput technologies

JM Johnson Matthey

London Technology Network Ideas into Action

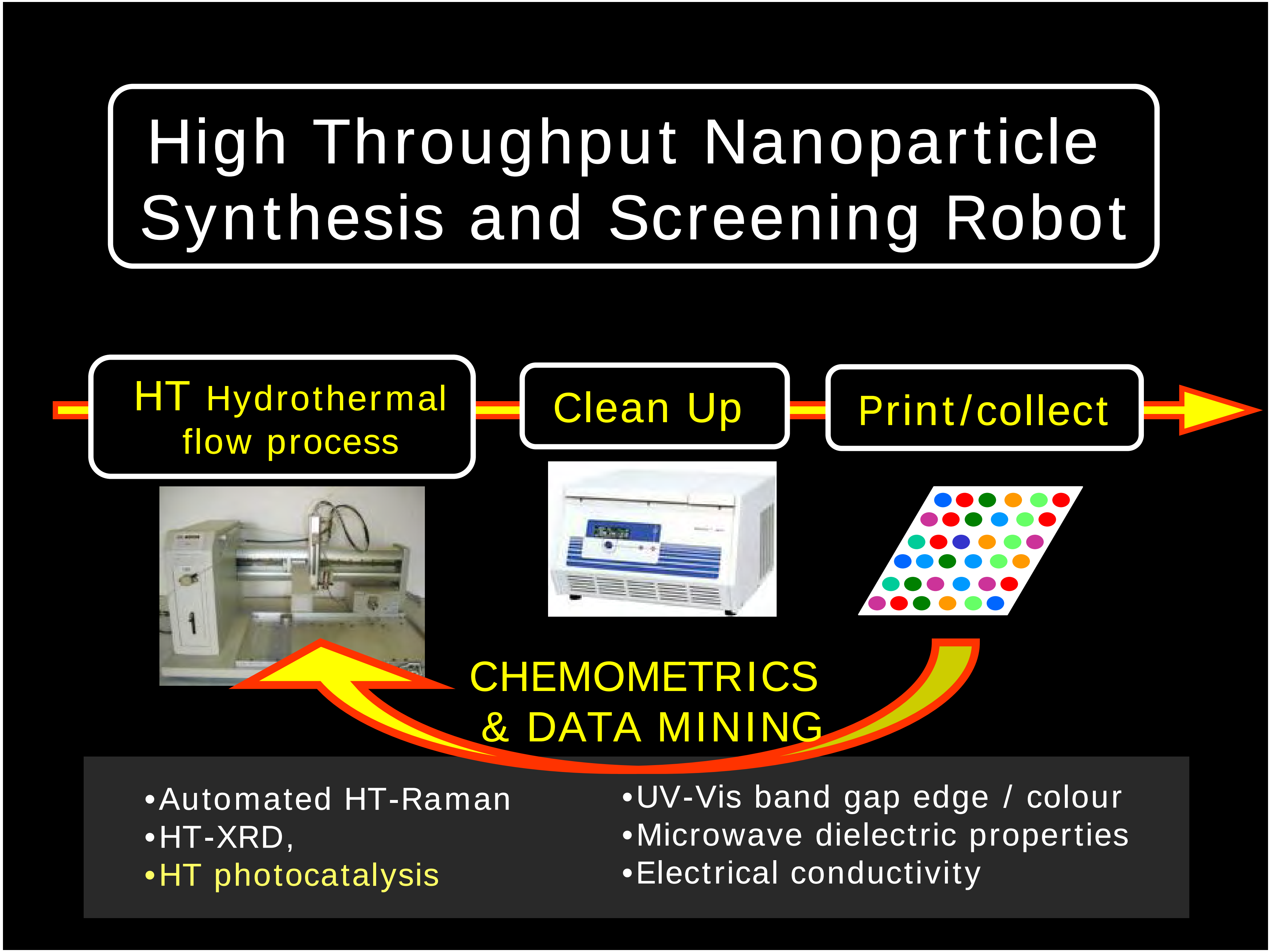
Labman Automation www.labman.co.uk

AMR TECHNOLOGIES INC.

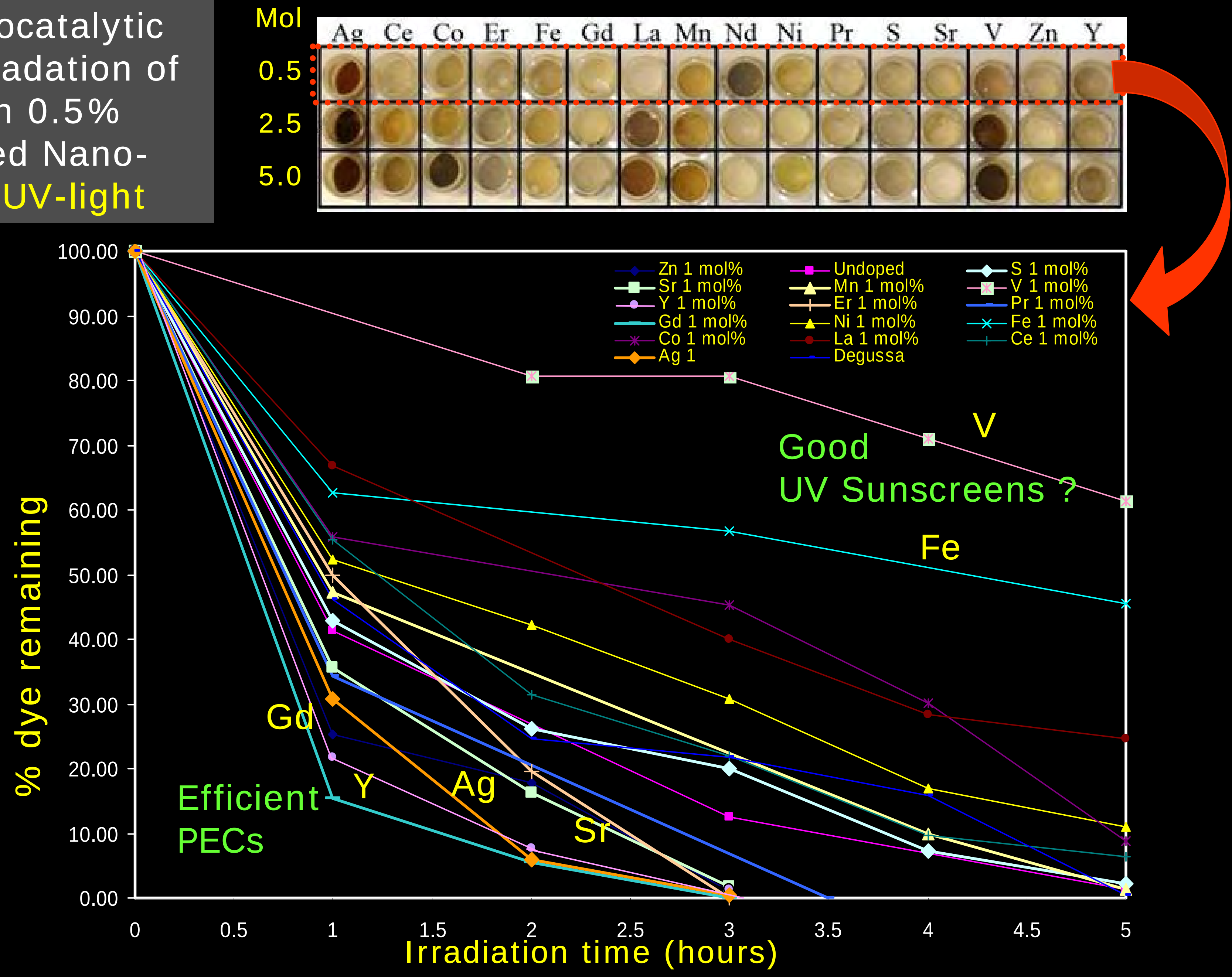
SunChemical

Thermo ELECTRON CORPORATION

Collaborators: Leeds University Prof Xue Wang

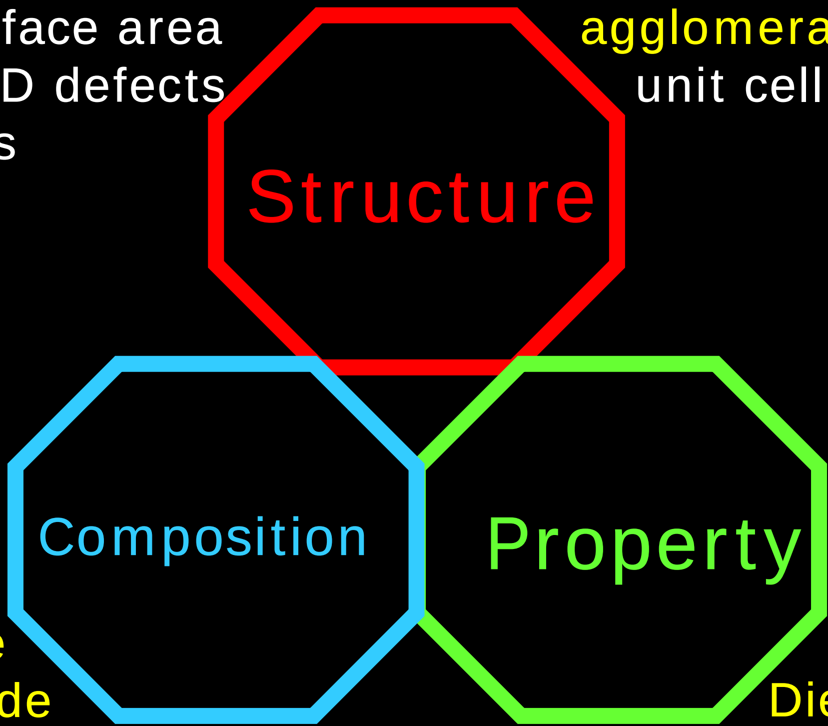

High Throughput Nanoparticle
Synthesis and Screening Robot
HT Hydrothermal
flow process
Clean Up
Print/collect
CHEMOMETRICS
& DATA MINING
•Automated HT-Raman
•HT-XRD,
•HT photocatalysis
•UV-Vis band gap edge / colour
•Microwave dielectric properties
•Electrical conductivity

Photocatalytic Degradation of MB in 0.5% Doped Nano-TiO_2 UV-light



Chemometrics / Data Mining

Particle size, micro/macro porosity, bonding
Surface area
agglomeration,
1D, 2D, 3D defects
unit cell, lattice
distortions

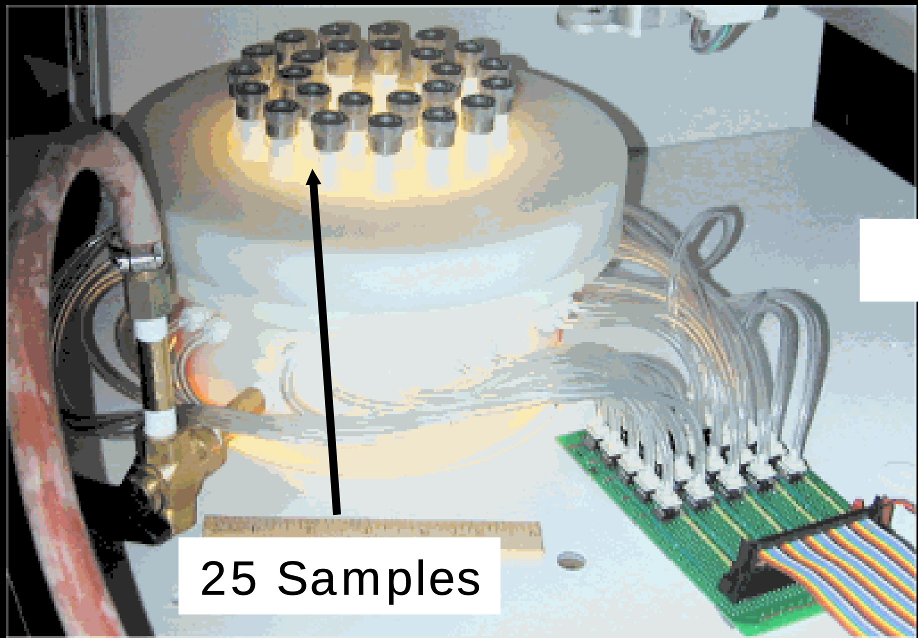


Dopants
Metal oxide
Metal nitride
Metal sulphide
M = doped Ti, Nb, W

Catalyst activity
Band gap
UV scattering
Dielectric const
Conductivity....

Solar simulator; H_2 sensing array

2nd Generation (pressure sensor)



courtesy B. Macqueen, SRI international

3rd Generation (2007+)

- 49 Sample array
- H_2 electronic sensor
- Faster equilibration

J. Darr , A. Mills and K. Thompson 2007

Smaller more E efficient devices

Low T Co-fired Dielectric Resonators at MW Freq.

Doped TiO_2

Low T Co-fired Dielectrics

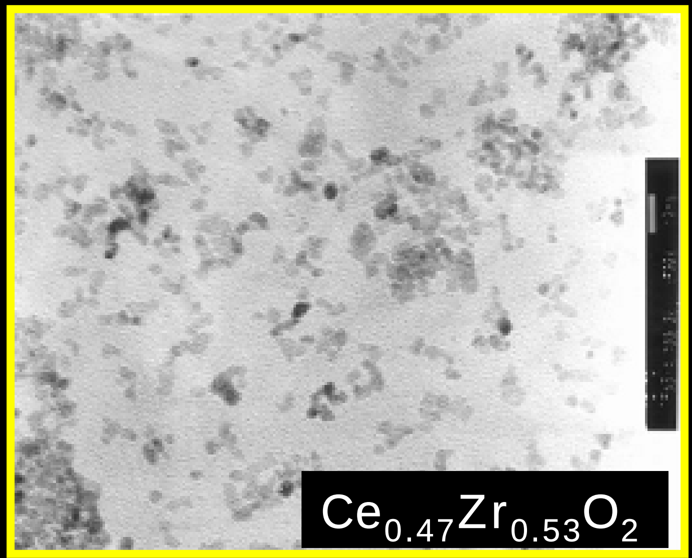
- 3D co-firing saves cost
- Allows miniaturisation
- “Nano” should sinter at Low T
- Want to sinter < 900 ^{O}C
- Want a high Qf (>10k)

Low T Co-fired Dielectrics

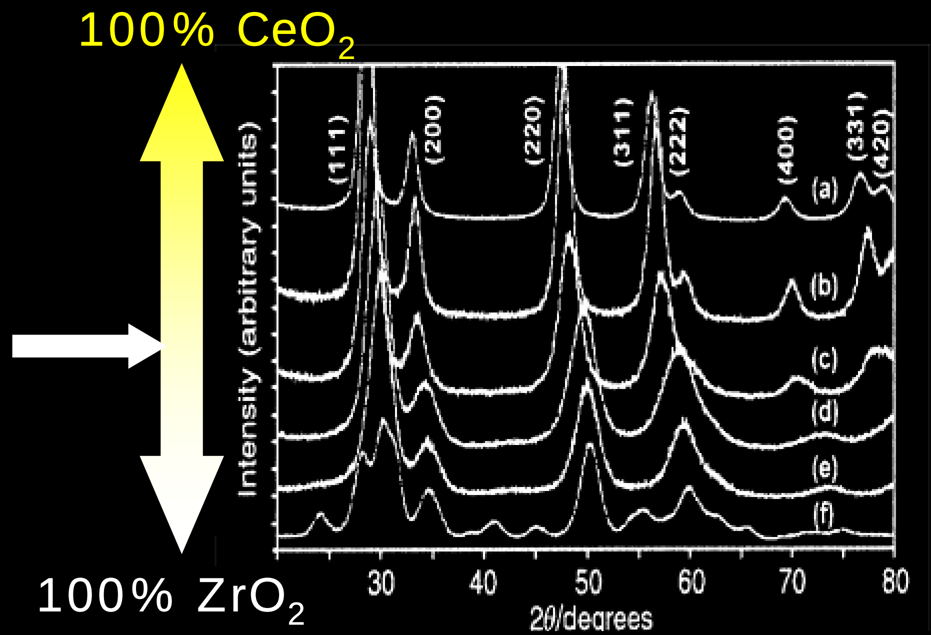
- Density low, but > micro
- Q_f follows density?
- Can sinter at 900 ^{o}C !
- Q_f values are high (>20k)
- High density via particle eng?

Nanoparticles for autocats

$Ce_{1-x}Zr_xO_{2-y}$ solid solutions



5 nm particles



A CONTINUUM OF COMPOSITIONS ARE POSSIBLE

Summary

- Multidisciplinary
- Improve Understanding
- Green / Novel Chemistry
- Energy efficient

Acknowledgements


- Sun chemical
- Johnson Matthey
- AMR Europe Ltd.

- Hydrogen Solar
- Rolls Royce FCS
- Thermo Electron
- Malvern instruments
- Industrial Chemicals
- Insight Faraday

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- Rob Pullar (IC)
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- Dr J. Anwar (Kings)
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- J. Elliot,
- I. Rehman,
- ZX Guo

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Development of Novel Nanostructured Catalysts For Cleaner Energy Production

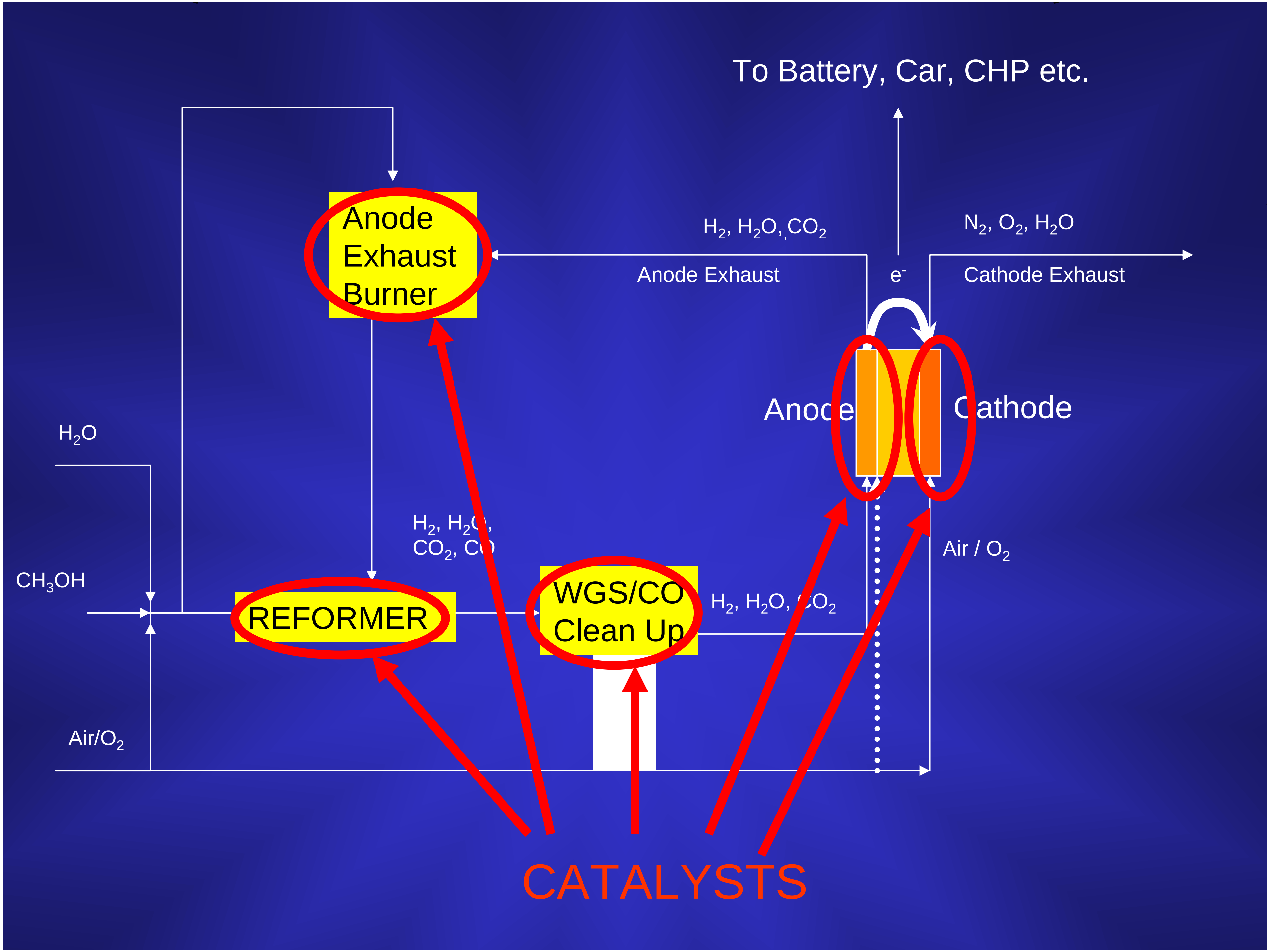
By

S. C. Edman Tsang

Wolfson Catalysis Centre
Inorganic Chemistry Labratory
University of Oxford

Why are we interested in nanocatalyst synthesis ?

- (1) Motives
- (2) Difficulties encountered and solutions
- (3) Typical Preparative methods
- (4) Current Research


To Battery, Car, CHP etc.
Anode Exhaust Burner
H2, H2O, CO2
Anode Exhaust
e-
N2, O2, H2O
Cathode Exhaust
Anode
Cathode
H2O
CH3OH
Air/O2
REFORMER
H2, H2O, CO2, CO
WGS/CO Clean Up
H2, H2O, CO2
Air / O2
CATALYSTS

Reforming of Hydrocarbons (or Methanol) to H_2

(A) Steam reforming:

$C_xH_y + H_2O \rightarrow H_2 + CO + CO_2$

An endothermic process

(B) Partial Oxidation

xs $C_xH_y + O_2 \rightarrow CO + CO_2 + H_2$

An exothermic process

(C) Auto thermal reforming – a combination of both approaches which is self-sustaining.

Problems of metal particle sintering and carbon formation !

Generate catalysts that have high activity/selectivity of:

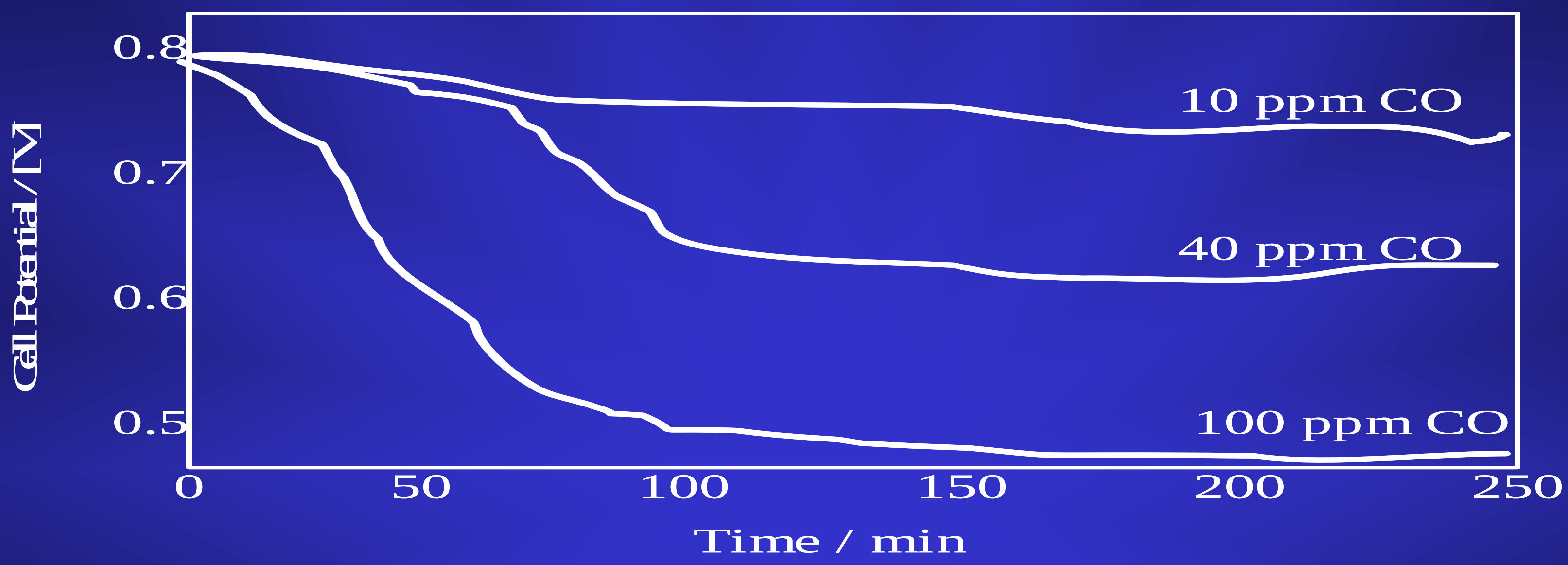
- $CO + H_2O \leftrightarrow CO_2 + H_2$ (Water-Gas-Shift)
- $CO + O_2 \rightarrow CO_2$ but not $2H_2 + O_2 \rightarrow 2H_2O$ (Selective Oxidation)

(Use of $Au/Fe_2O_3/TiO_2$)

Improve WGS and CO selective oxidation !

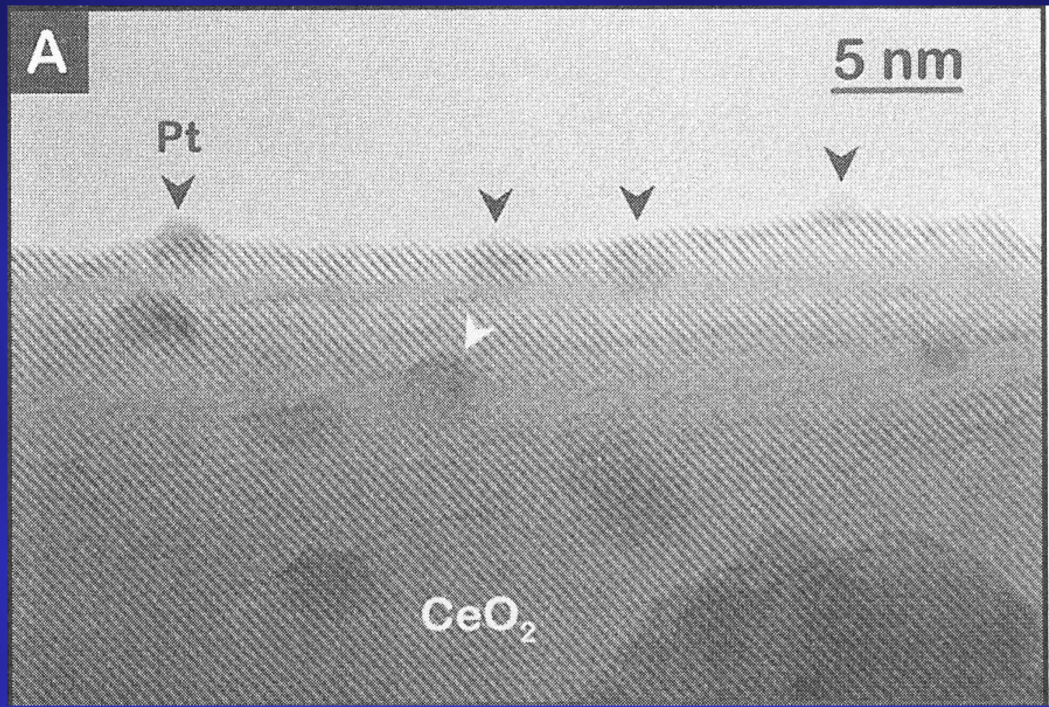
CO irreversibly adsorbs on the catalytic Pt Particles

$Pt + CO \rightarrow Pt\text{-}CO_{ads}$.



Problem of CO degrading the anode performance !

Conventional Catalysts

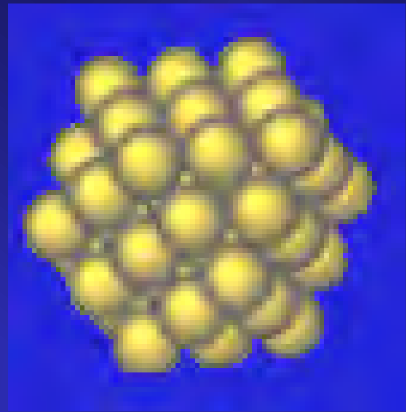


A typical HREM image corresponding to the Pt (4%)/CeO_2 prepared by a conventional impregnation technique

Metal size

Metal shape

Metal-Support Interactions in Catalysis



Shell	Atoms in a cluster	Surface atoms	Proportion of surface atoms (%)
1	13	12	92
2	55	42	76
3	147	92	63
4	309	162	52
5	561	252	45

Site Reactivity on Nanoparticle

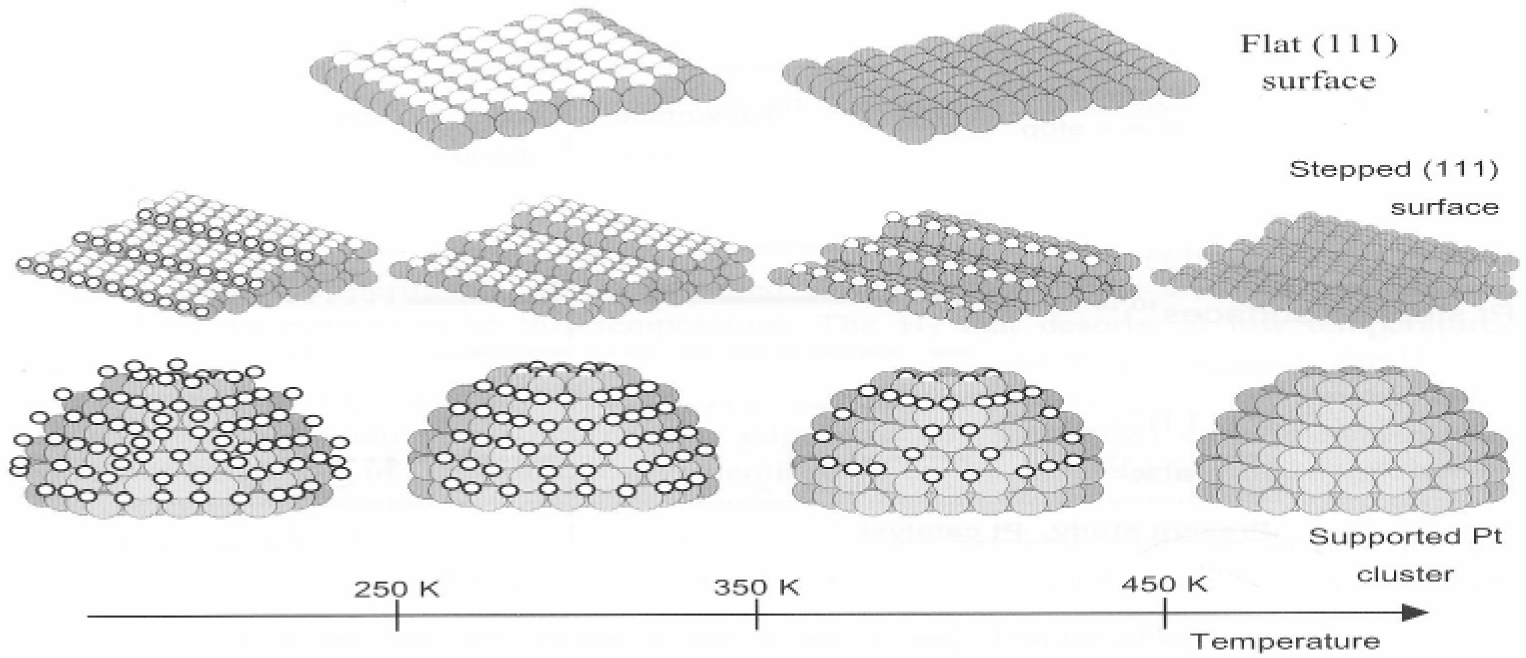
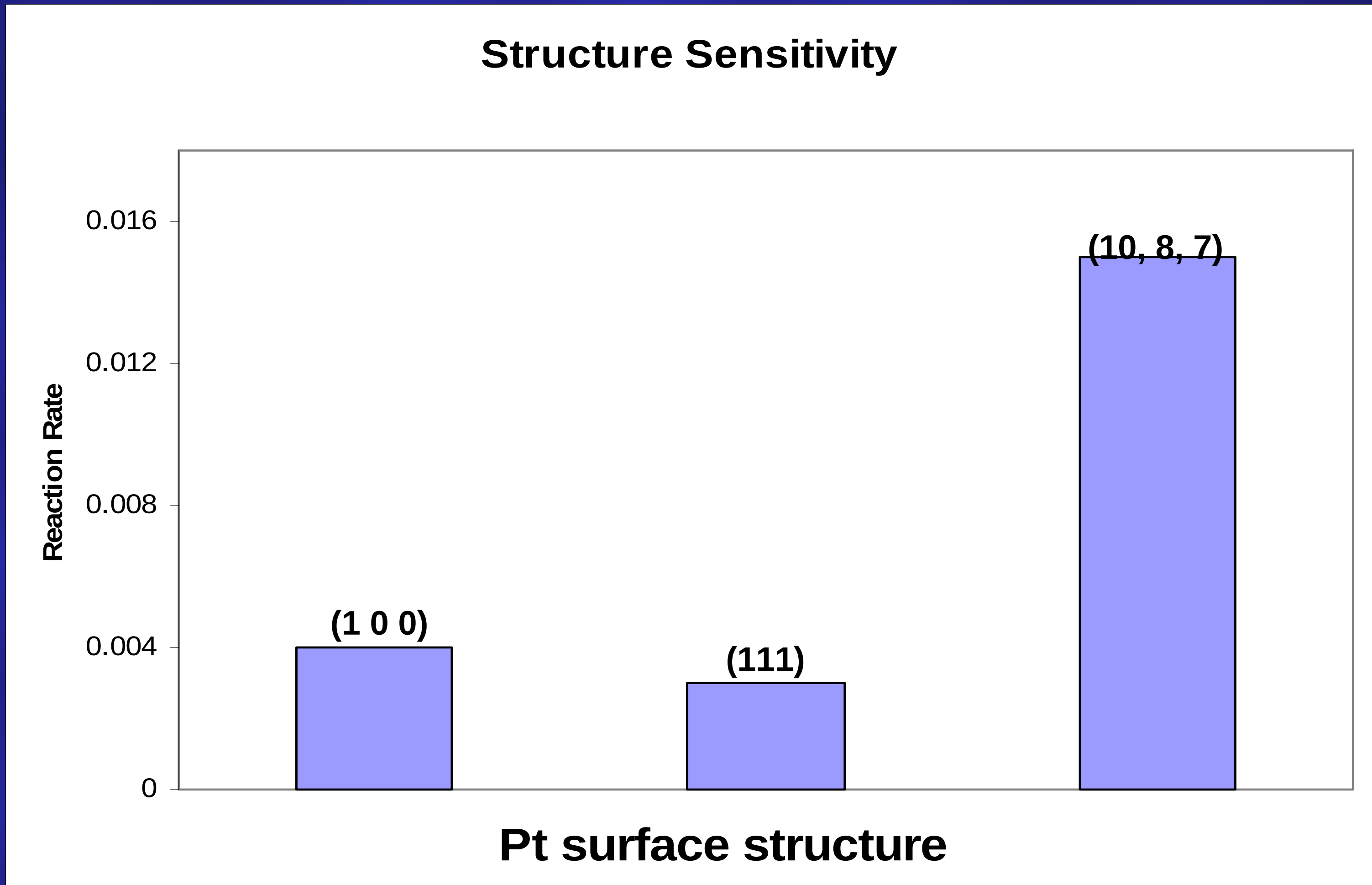
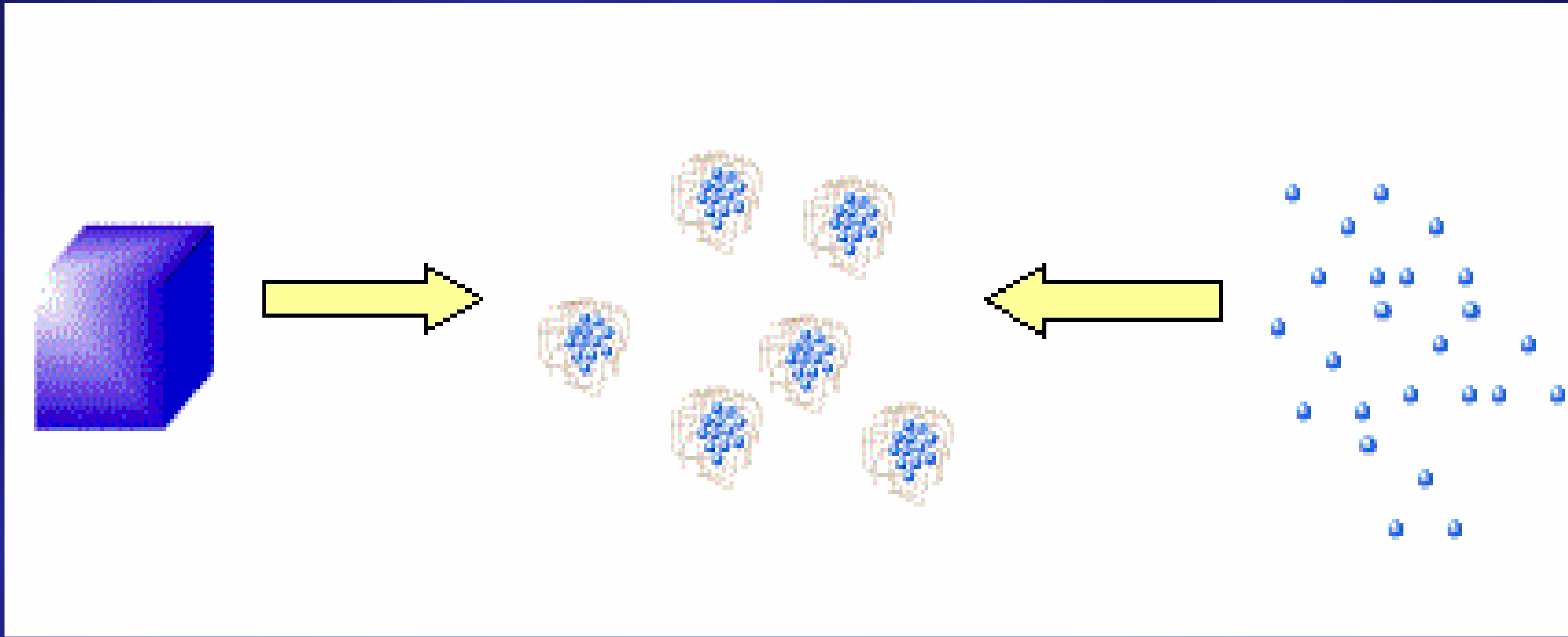


Figure 2: The coverage with H of a flat Pt (111) surface (top), a stepped surface (middle) and a supported Pt cluster (bottom) as a function of temperature. Dark spheres: platinum; white spheres: H.

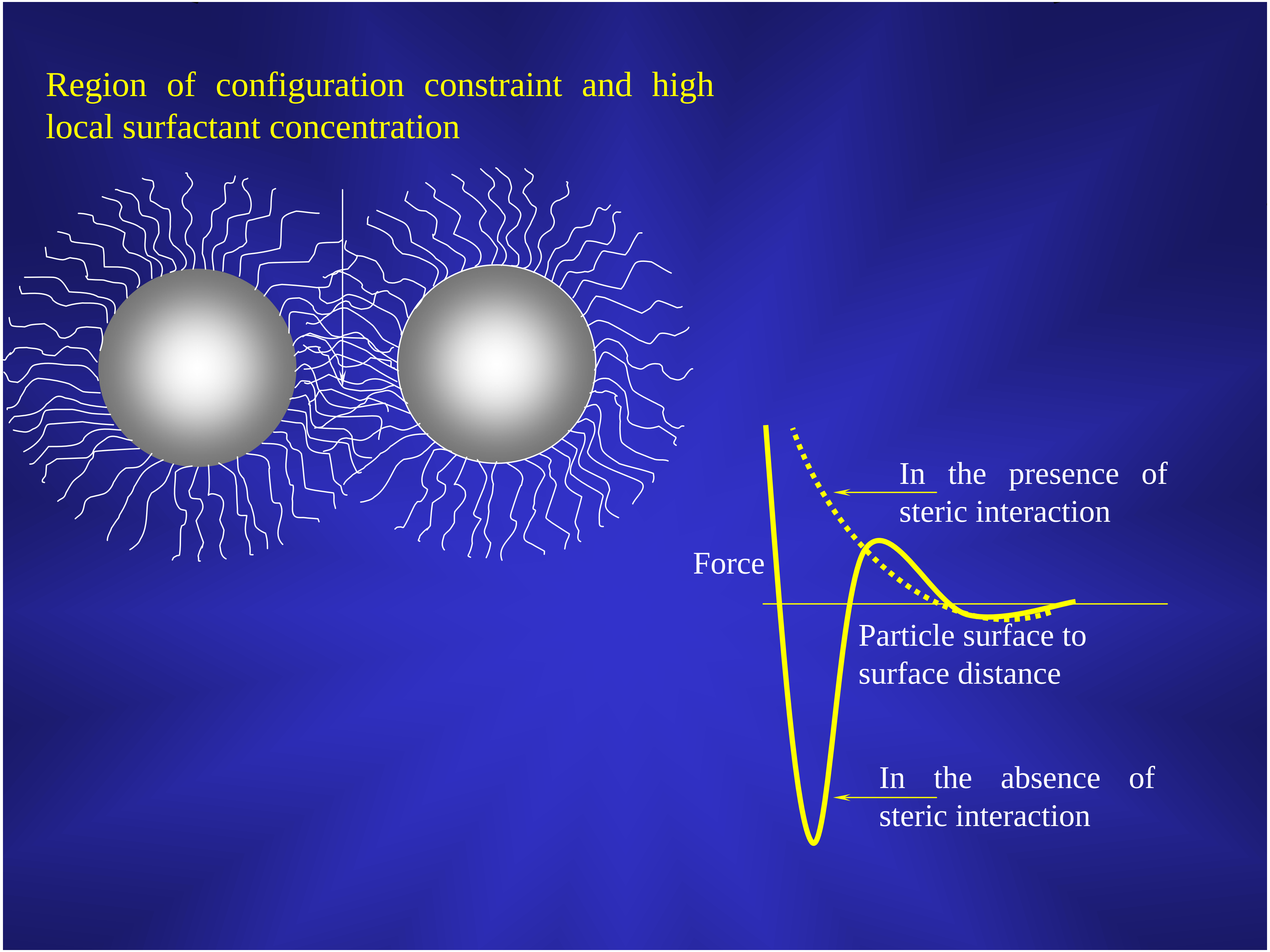
Site Reactivity on Nanoparticle

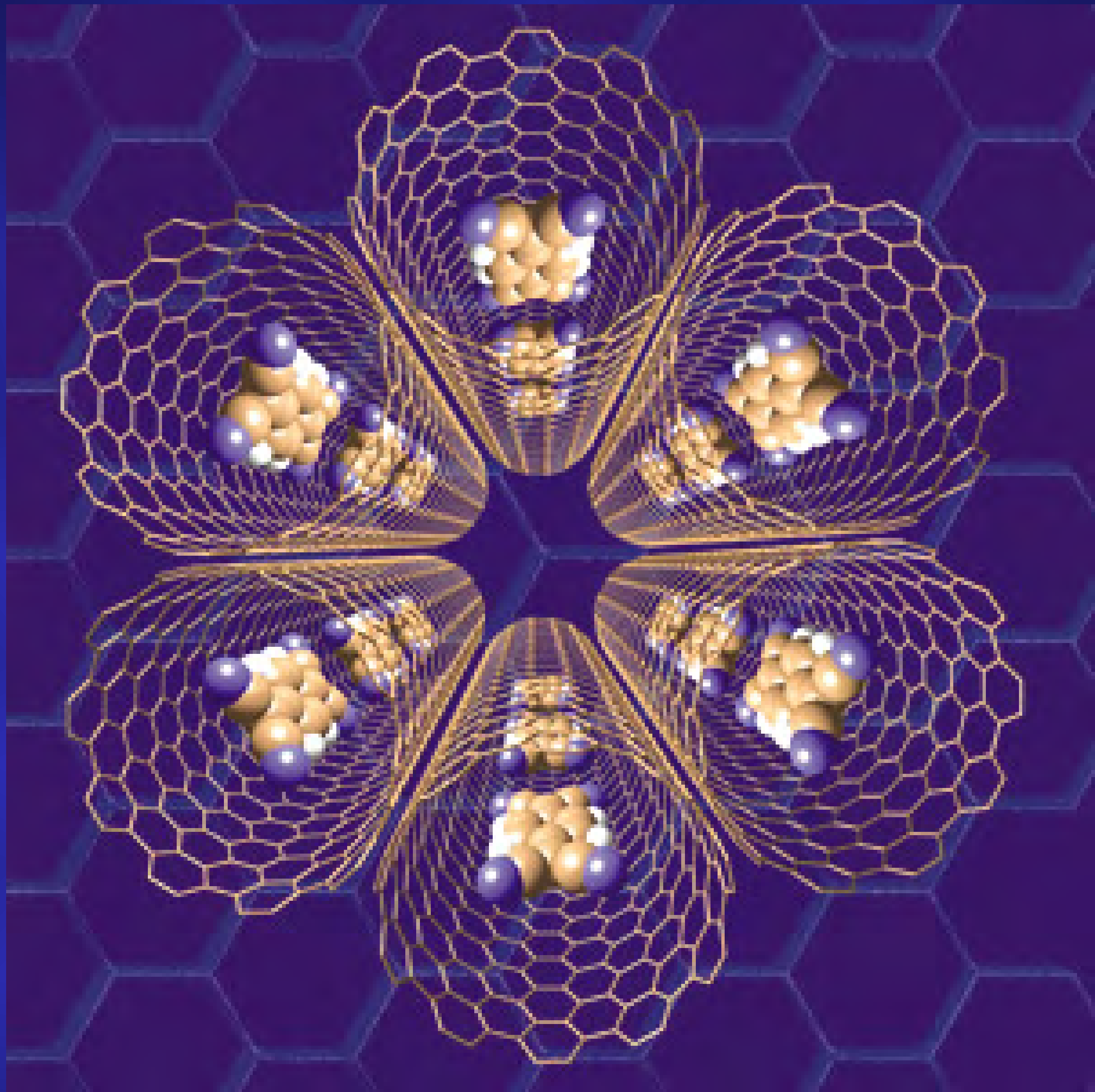


GA Somorjai, Introduction of Surface Chemistry and Catalysis, Wiley- Interscience Pub., 1994



Top down and bottom up approaches for the synthesis of nanoparticles


Region of configuration constraint and high local surfactant concentration
Force
In the presence of steric interaction
Particle surface to surface distance
In the absence of steric interaction



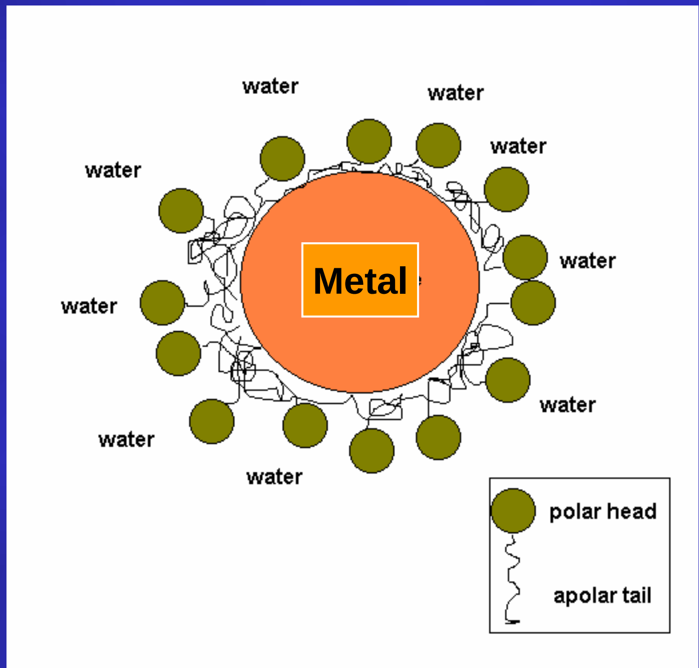
Caps, Paraskevas and Tsang, Chem Commun.2005, 1708

Hydrosol Method

Organo-ammonium sulfonate

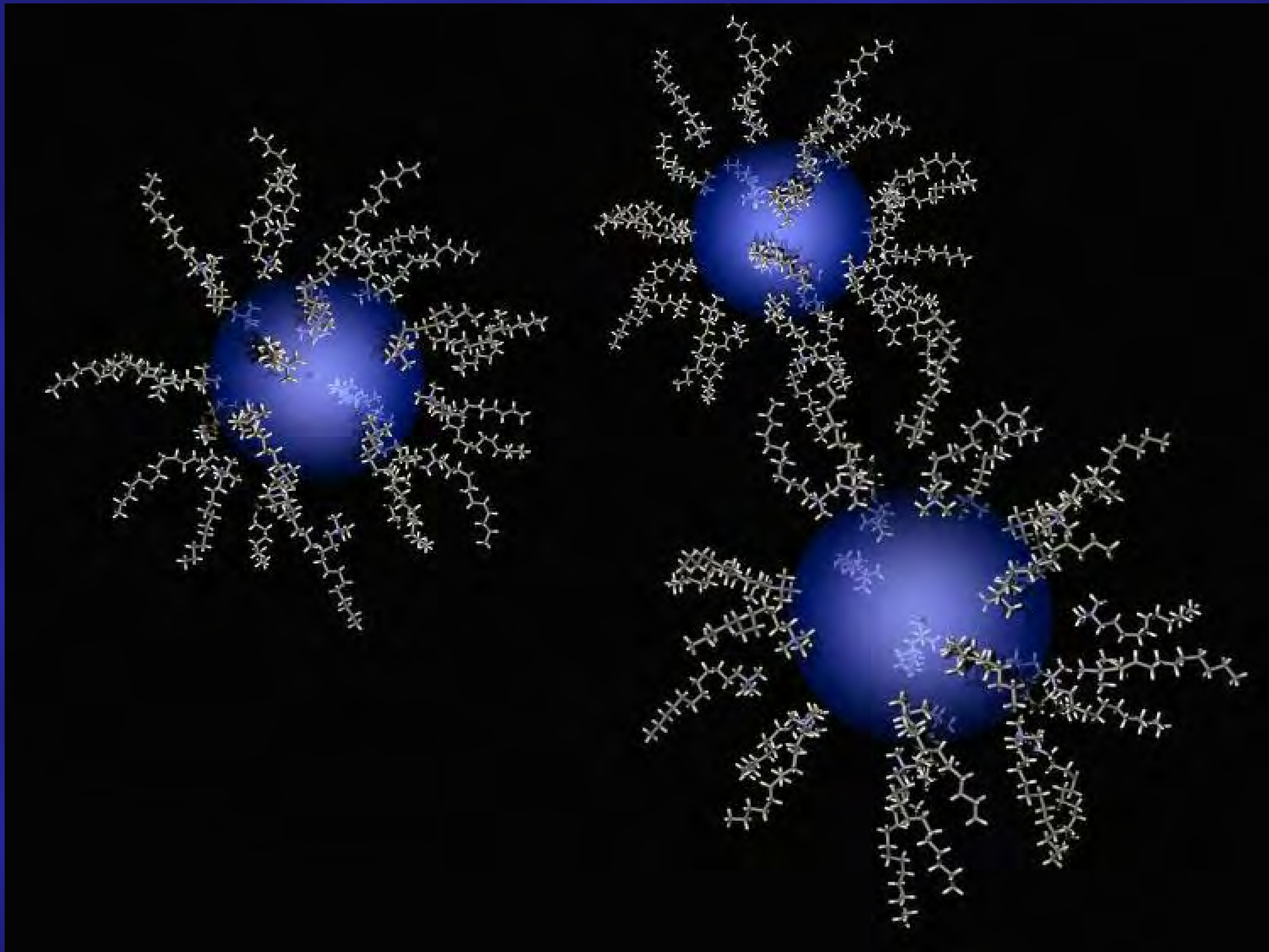
$$PtCl_2 + 4C_{12}H_{25}N^+(CH_3)_2(C_3H_6\text{-}SO_3^-) + Li_2CO_3 + H_2 \rightarrow$$

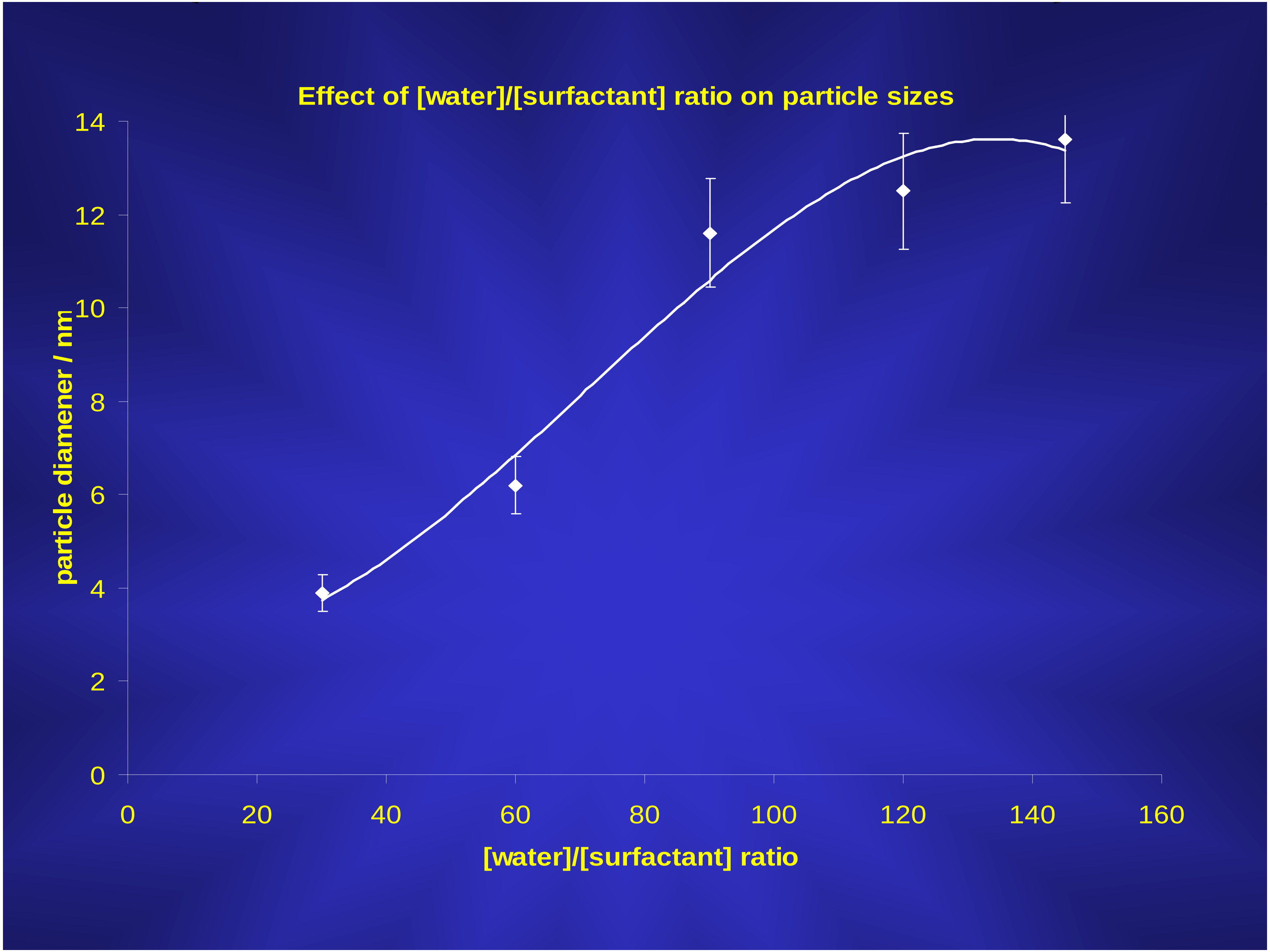
$$Pt + 4C_{12}H_{25}N(CH_3)_2(C_3H_6\text{-}SO_3) + H_2O + CO_2\uparrow$$



aqueous colloidal solutions of Au, Ag, Ir, Pt,Pd, Rh and Ru and alloys can be prepared

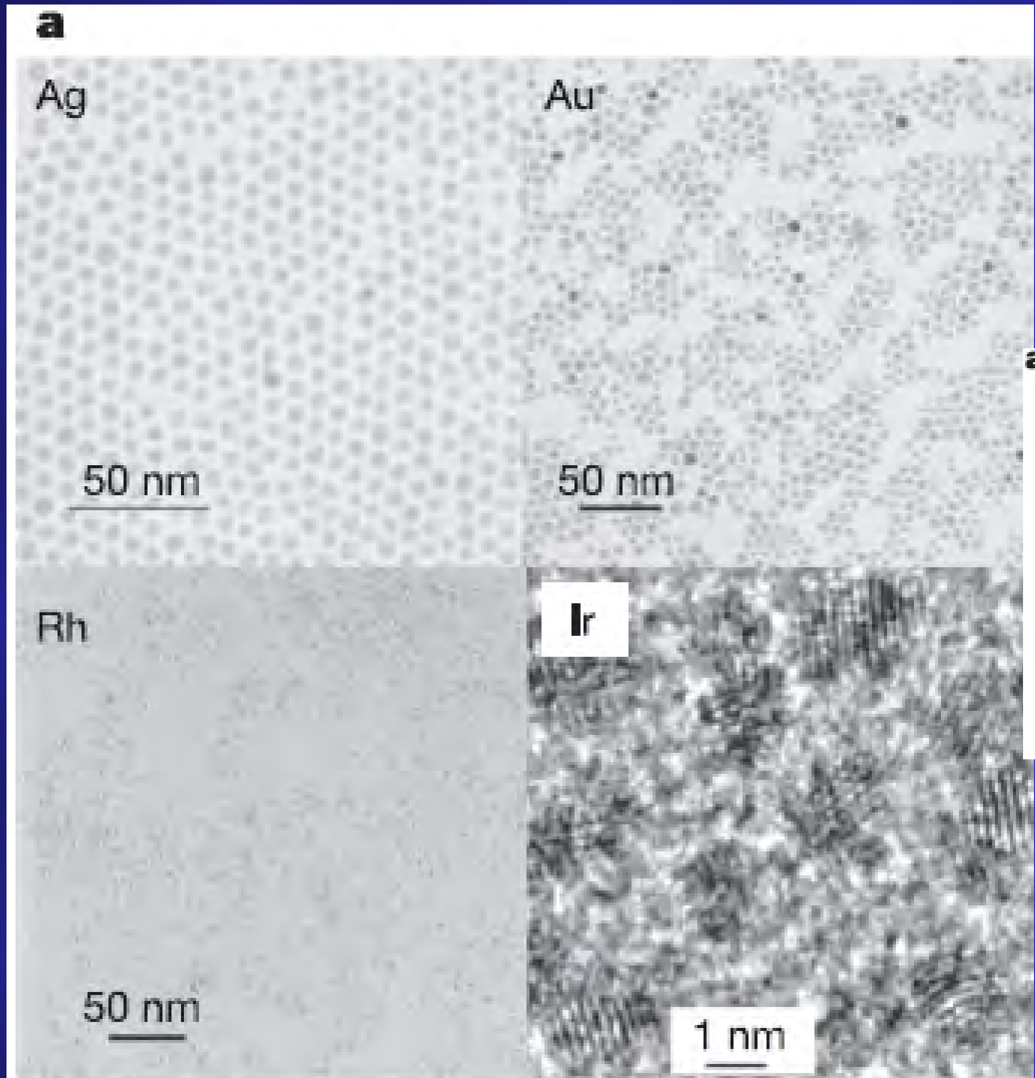
Micelles



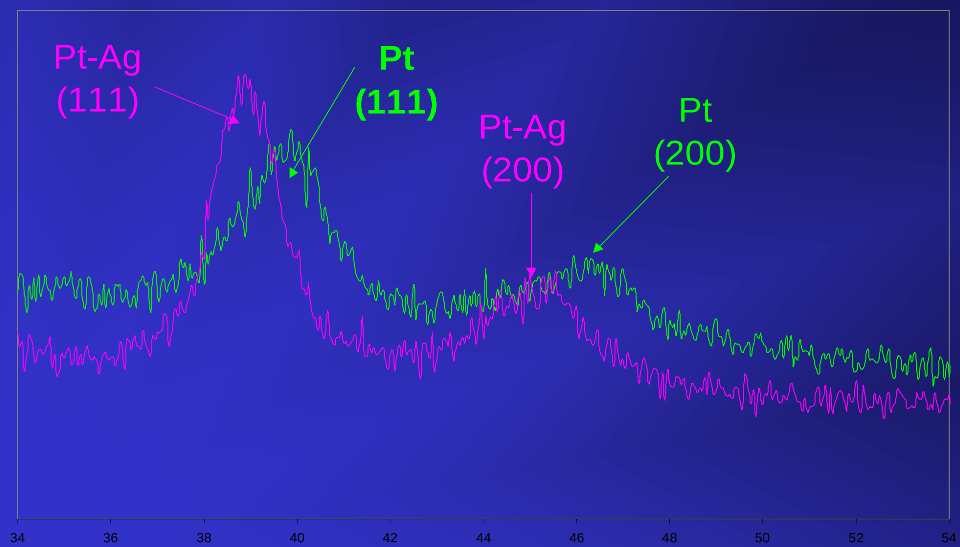


Effect of [water]/[surfactant] ratio on particle sizes
particle diamener / nm
14
12
10
8
6
4
2
0
0
20
40
60
80
100
120
140
160
[water]/[surfactant] ratio

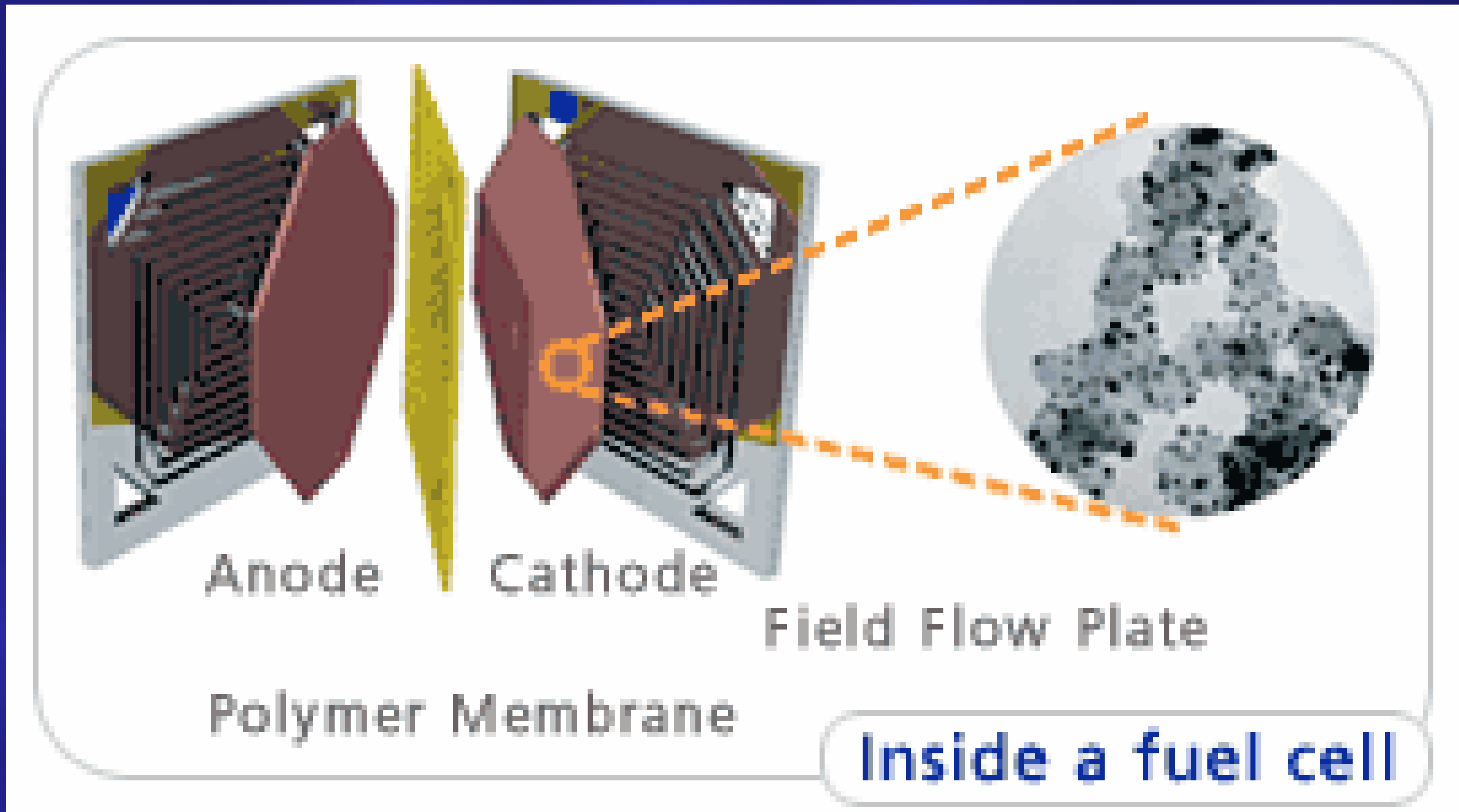
Preformed nanocrystals



Characterisation



A Pt-Ag metal core showing a Pt-Ag {111} spacing of 2.30 ± 0.02Å (scale bar of 23.0 Å where 10 lattice fringes can be clearly visible).


Anode
Cathode
Field Flow Plate
Polymer Membrane
Inside a fuel cell

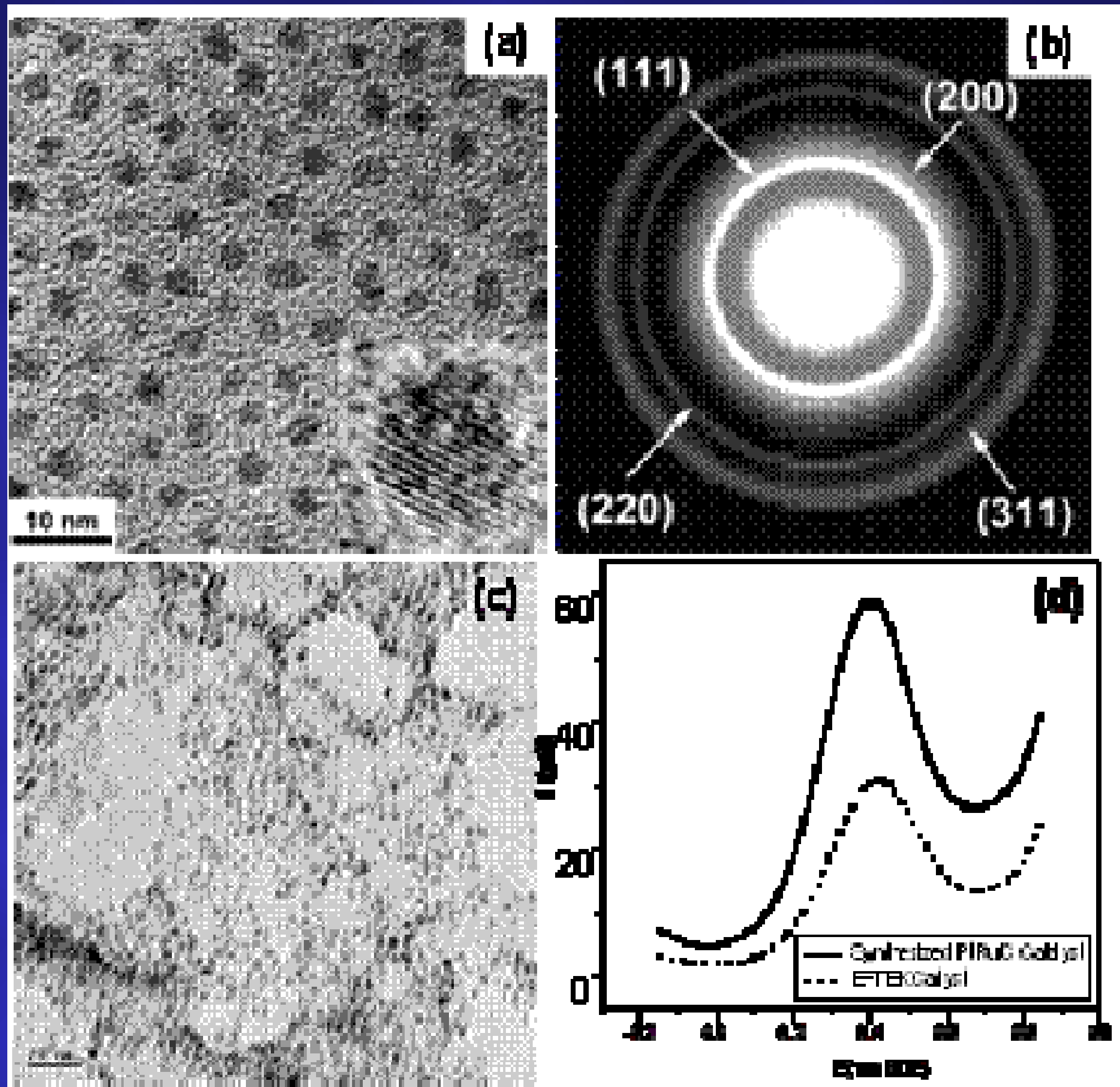
Pt-Ru nano-catalyst as fuel cell anode

It is believed CO adsorbs only onto Pt while Ru supplies active oxygen to Pt at lower potentials than is possible for Pt alone to liberate the Pt active site.

$$Pt\text{-}CO + Ru\text{-}OH \rightarrow Pt + Ru + CO_2 + H^+ + e^-$$

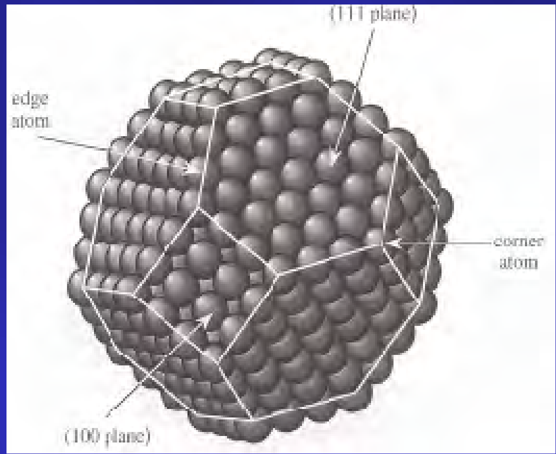
Pt-Co nano-catalyst as fuel cell cathode

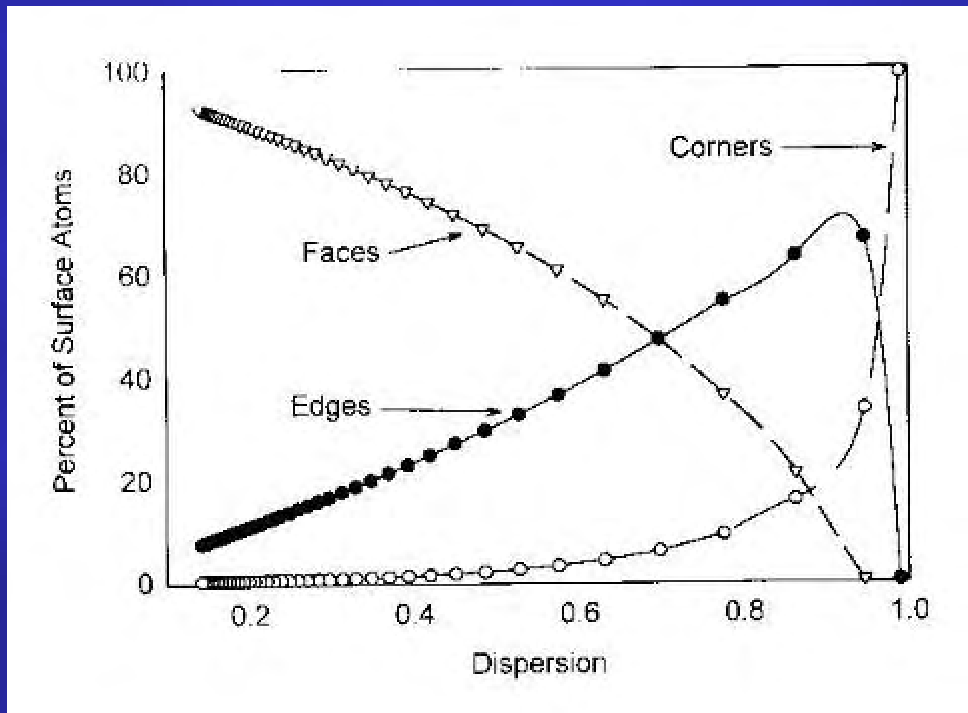
Oxides on Pt constitutes a problem due to poisoning of the surface. It is been reported that binary alloys PtCo are surface-rich in Pt, but possesses a minimum amount of oxides on the surface.



a) TEM image of PtRu nano-alloy prepared by 1,2-hexadecanediol in the presence of oleic acid and oleylamine stabilizers (b) A selected area electron diffraction pattern. (c) TEM images of PtRu/C (d) electro-methanol oxidation by PtRu/C catalyst (solid line) compared with JM PtRu/C catalyst (dashed line). Scan rate=0.010V/s

Size effect of platinum nanoparticle

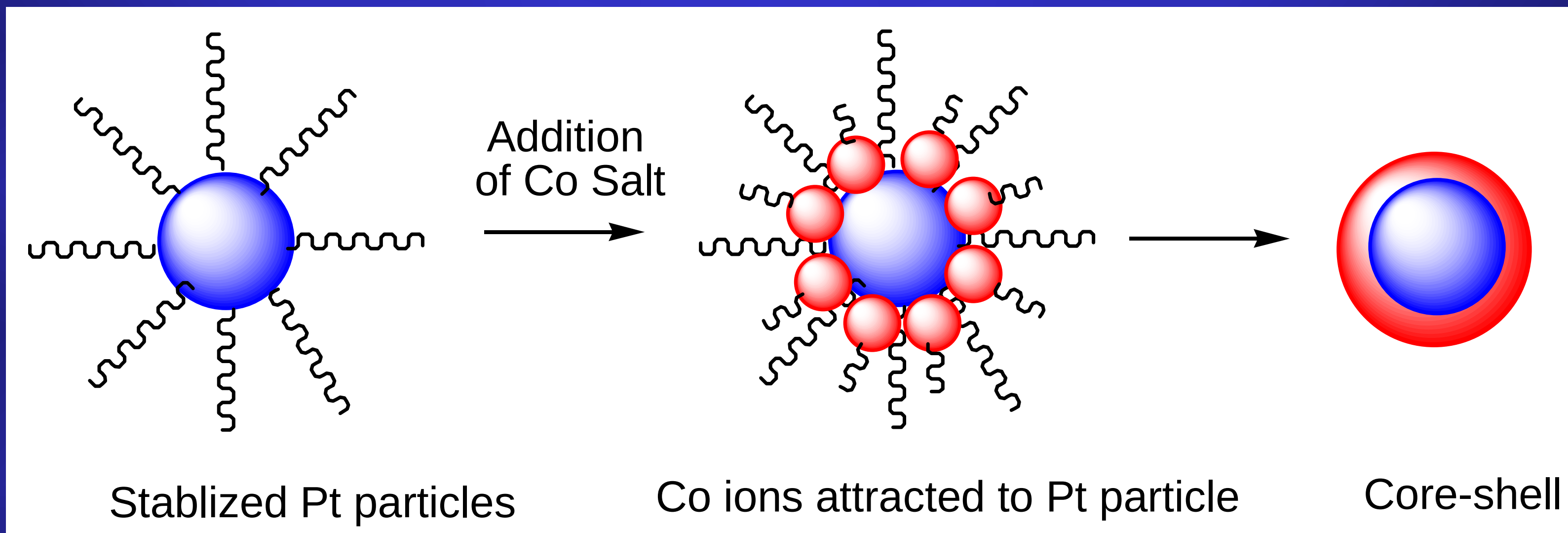


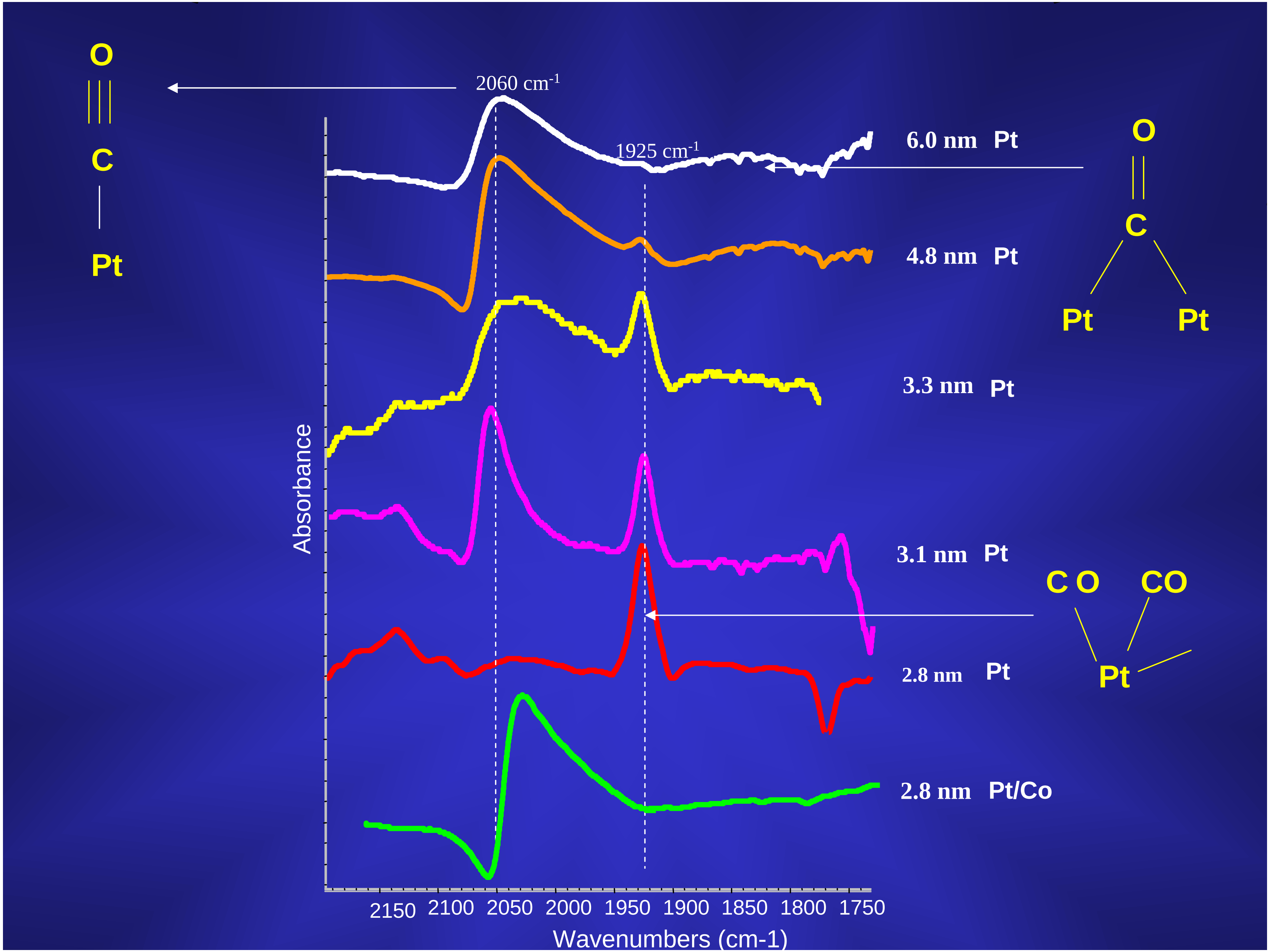


Nanoparticle Decoration

Pre-formation of platinum nanoparticle;

followed by a control deposition of foreign atoms on the platinum particle.

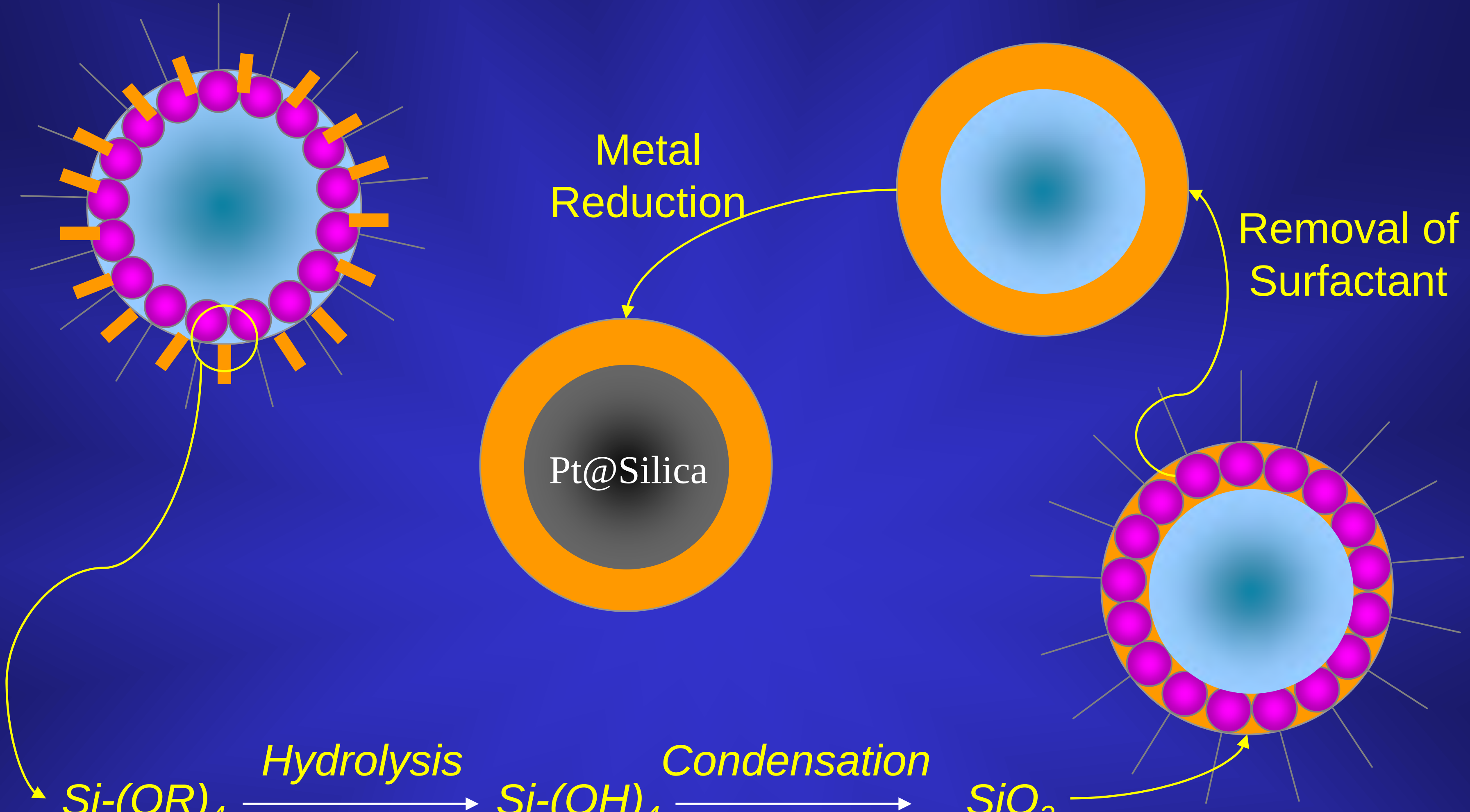




O ≡ C — Pt
2060 cm-1
1925 cm-1
Absorbance
6.0 nm Pt
4.8 nm Pt
3.3 nm Pt
3.1 nm Pt
2.8 nm Pt
2.8 nm Pt/Co
O
C
Pt Pt
C O CO
Pt
2150 2100 2050 2000 1950 1900 1850 1800 1750
Wavenumbers (cm-1)

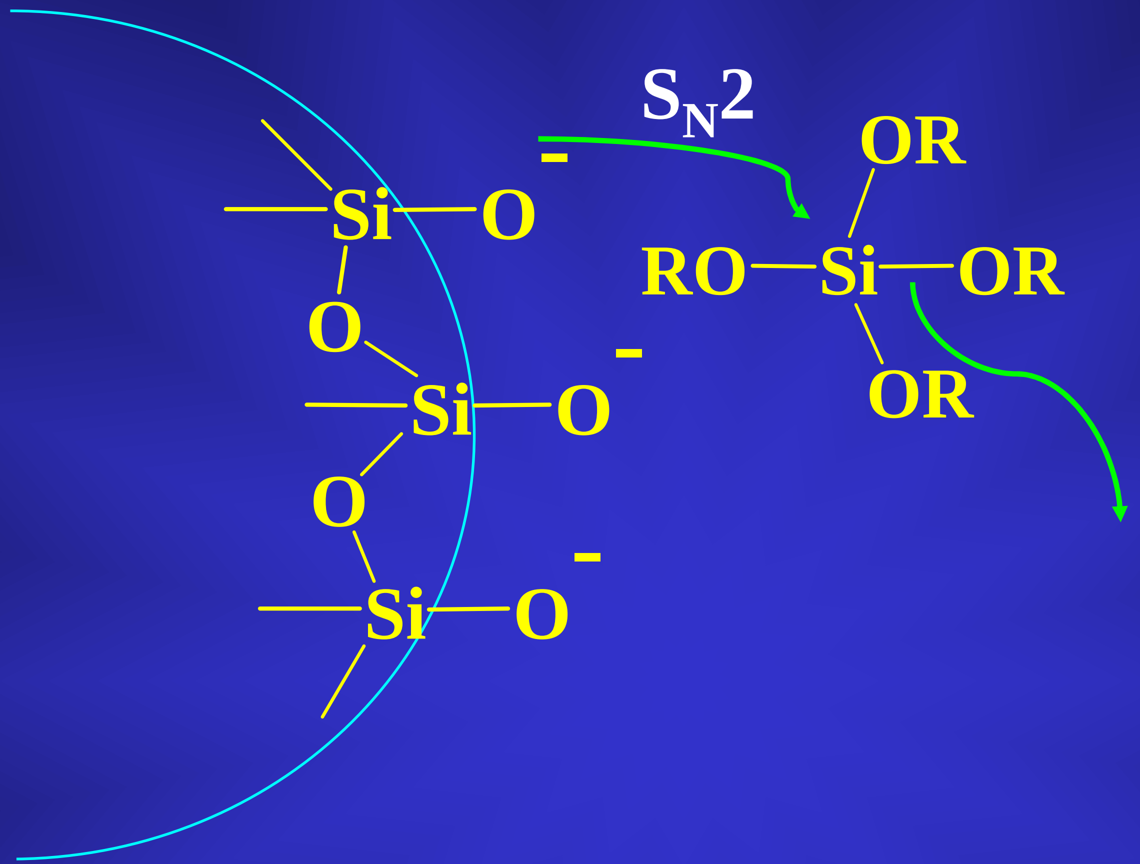
Methodology

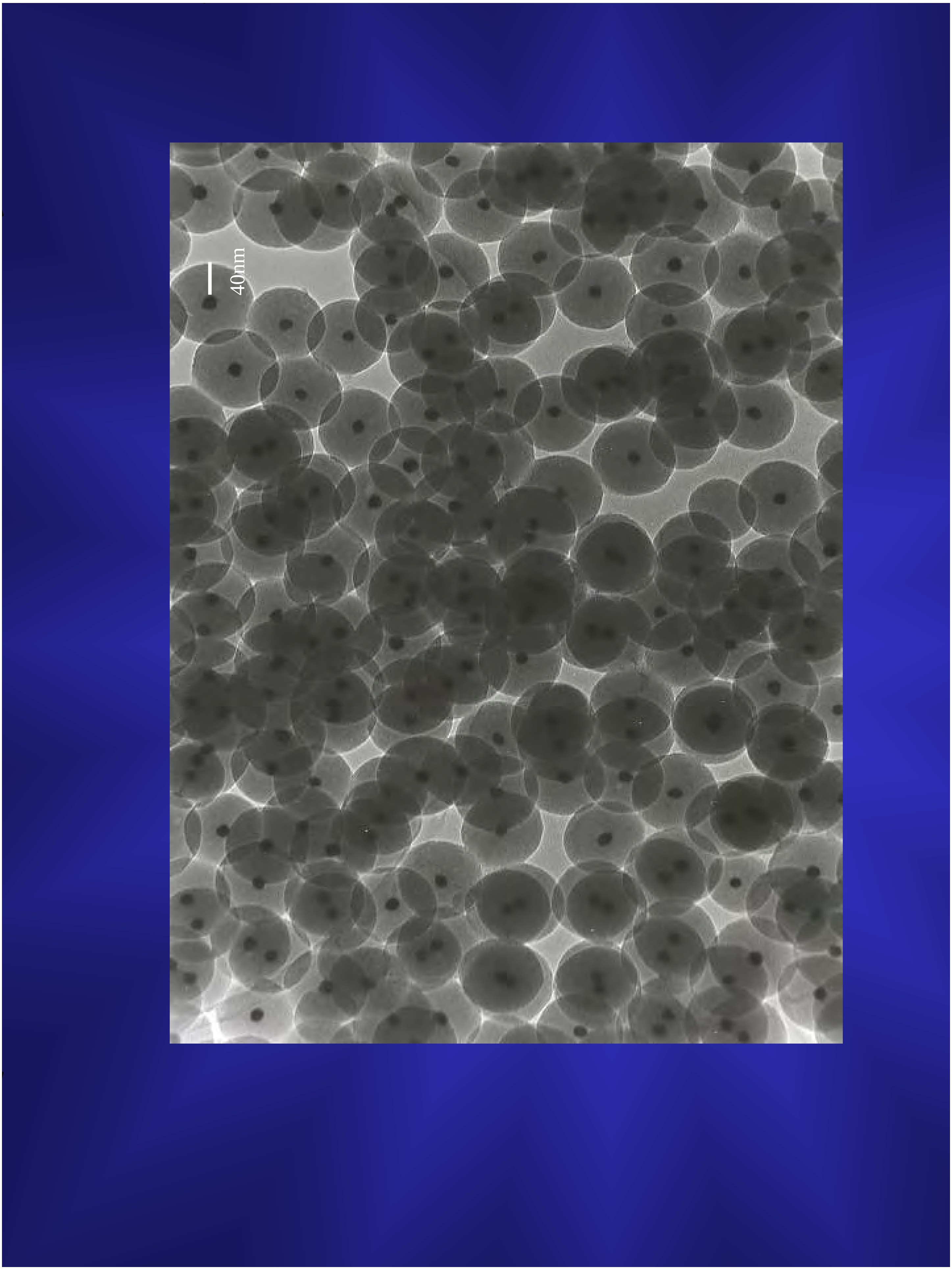
Silica encapsulated metal nanoparticles



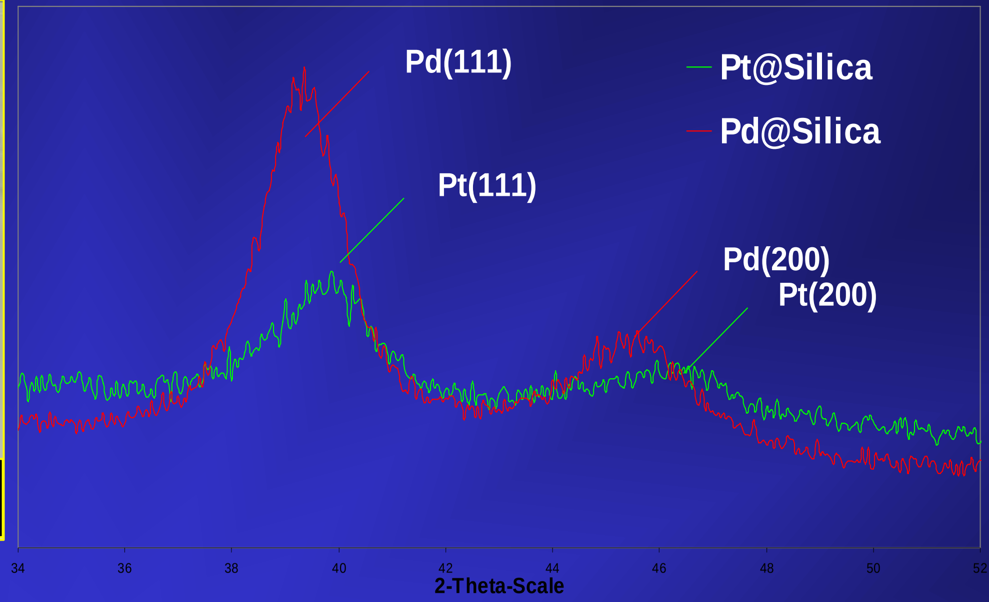
$$Si\text{-}(OR)_4 \xrightarrow[OH^-]{\text{Hydrolysis}} Si\text{-}(OH)_4 \xrightarrow{\text{Condensation}} SiO_2$$

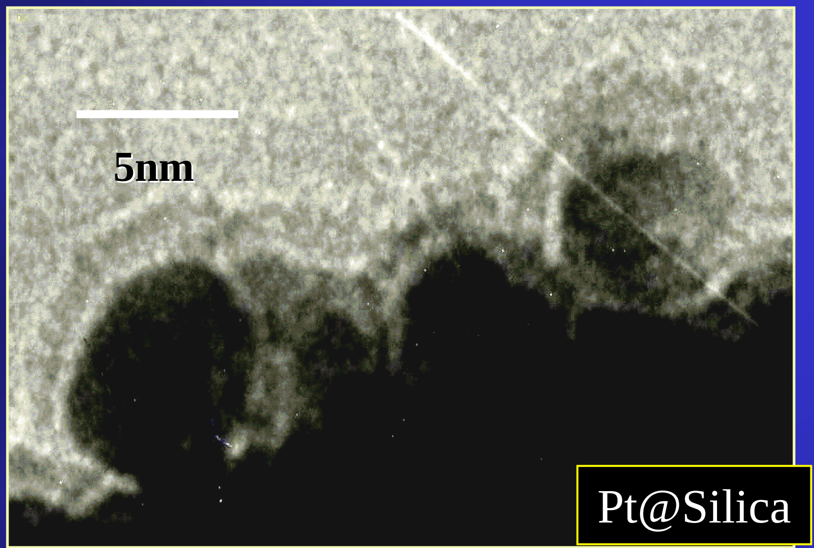
Yu, Yeung, Thompsett and Tsang, *J.Phys.Chem. B* 107, **2003**, 4515.


S$_N$2
Si
O
RO
OR


40nm

Characterisation



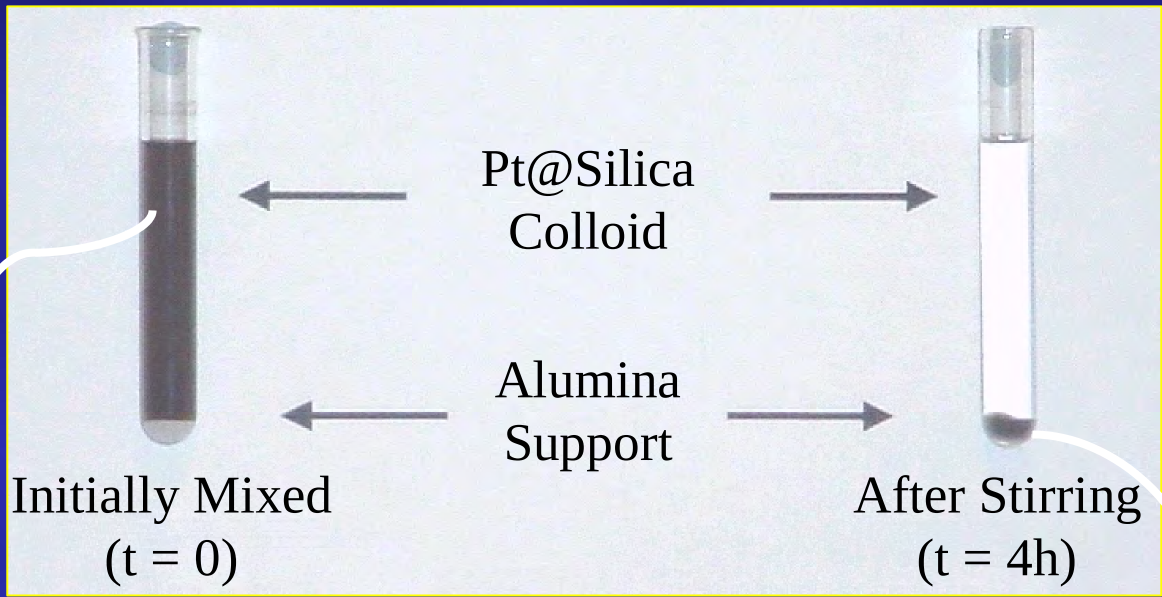


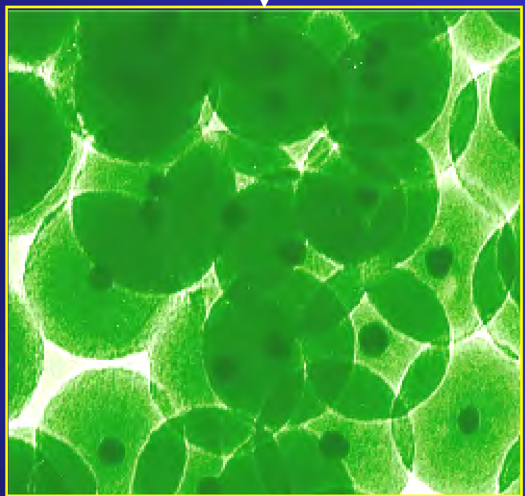
Pt: $(NH_4)_2PtCl_4$,
4.5 ± 0.4 nm (metal core)

Pd: $(NH_4)_2PdCl_4$
3.9 ± 0.4 nm (metal core)

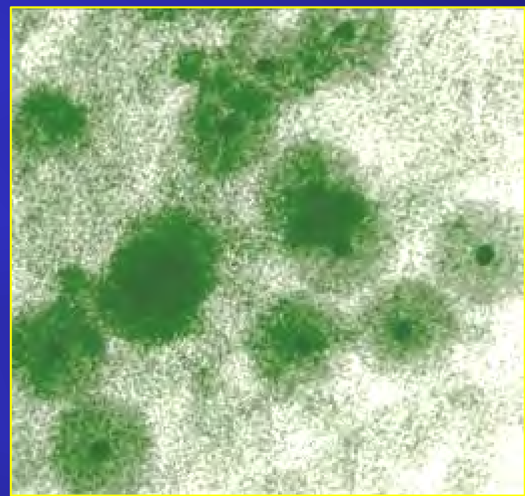
Tsang, Yu, Steele, Zhu and Fu *Catal.Today* **2003**, 81, 573.

Immobilisation of Pt@Silica onto Alumina

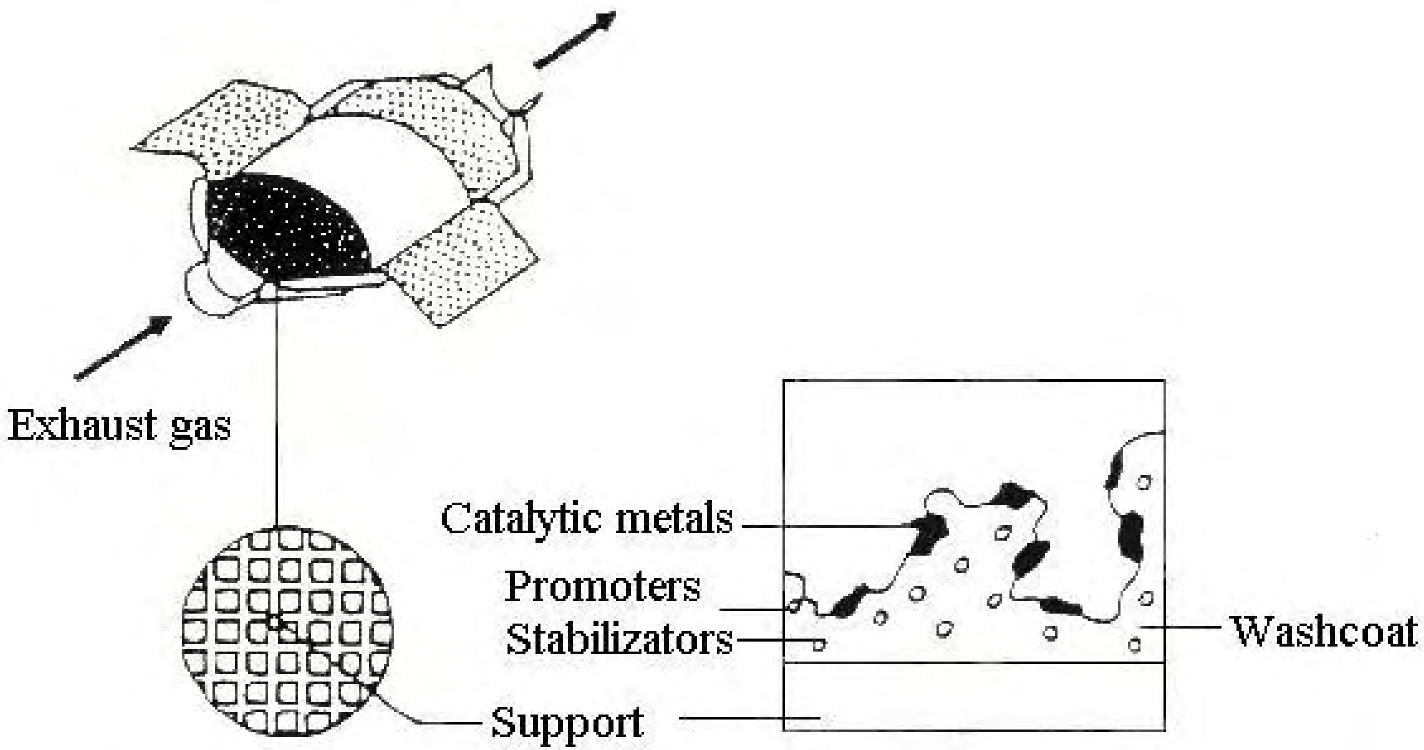




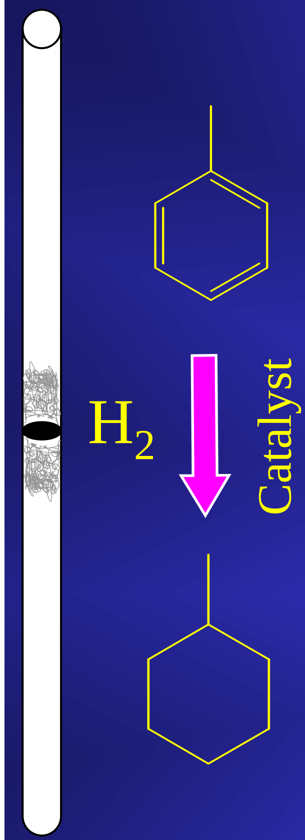
~1wt%Pt uptake by the alumina



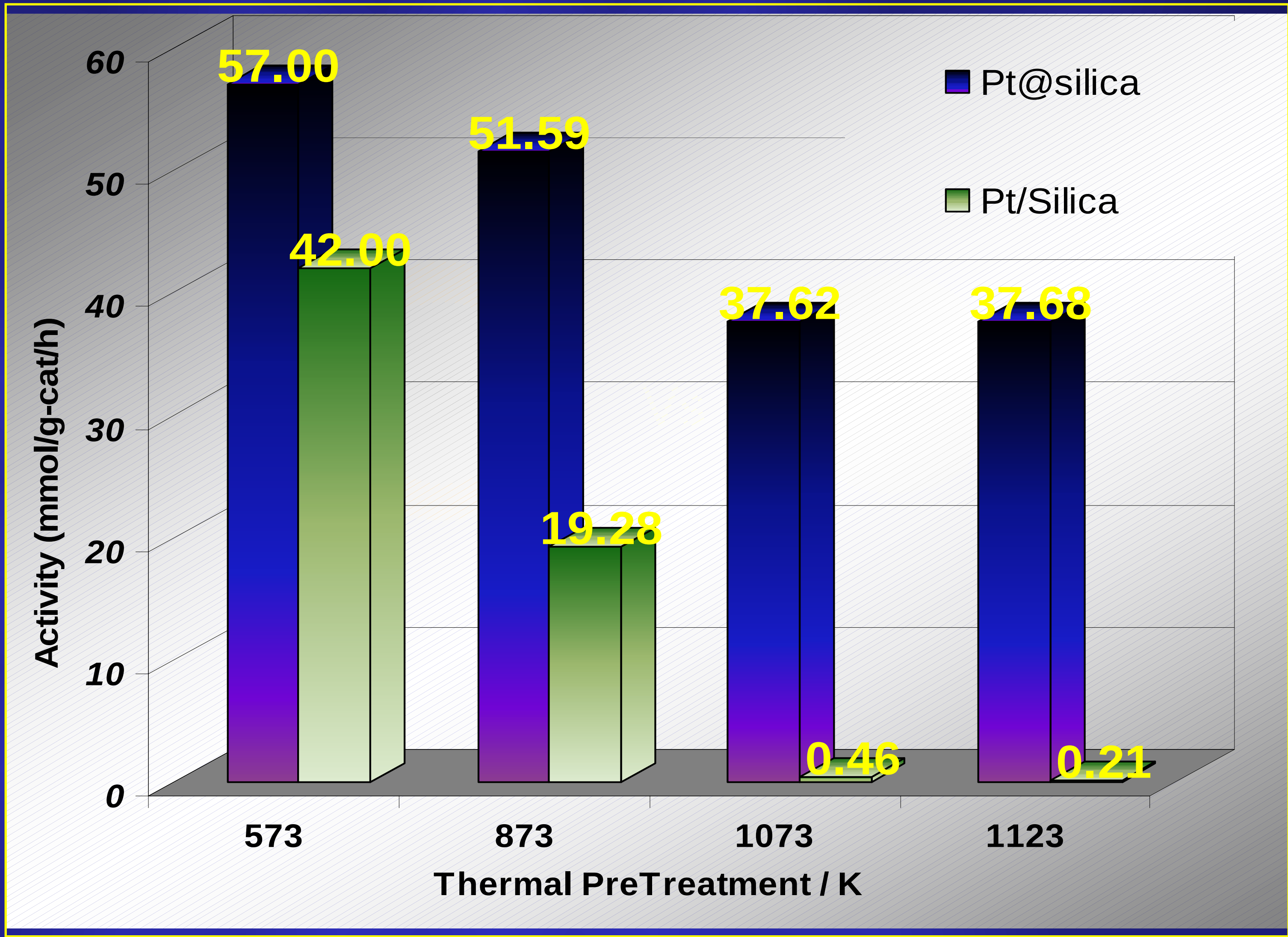
Yu, Thompsett and Tsang. *Chem.Commun*. **2003**, 1522


Exhaust gas
Catalytic metals
Promoters
Stabilizators
Support
Washcoat

Catalytic Activity Tests



Toluene Hydrogenation - Thermal Stability

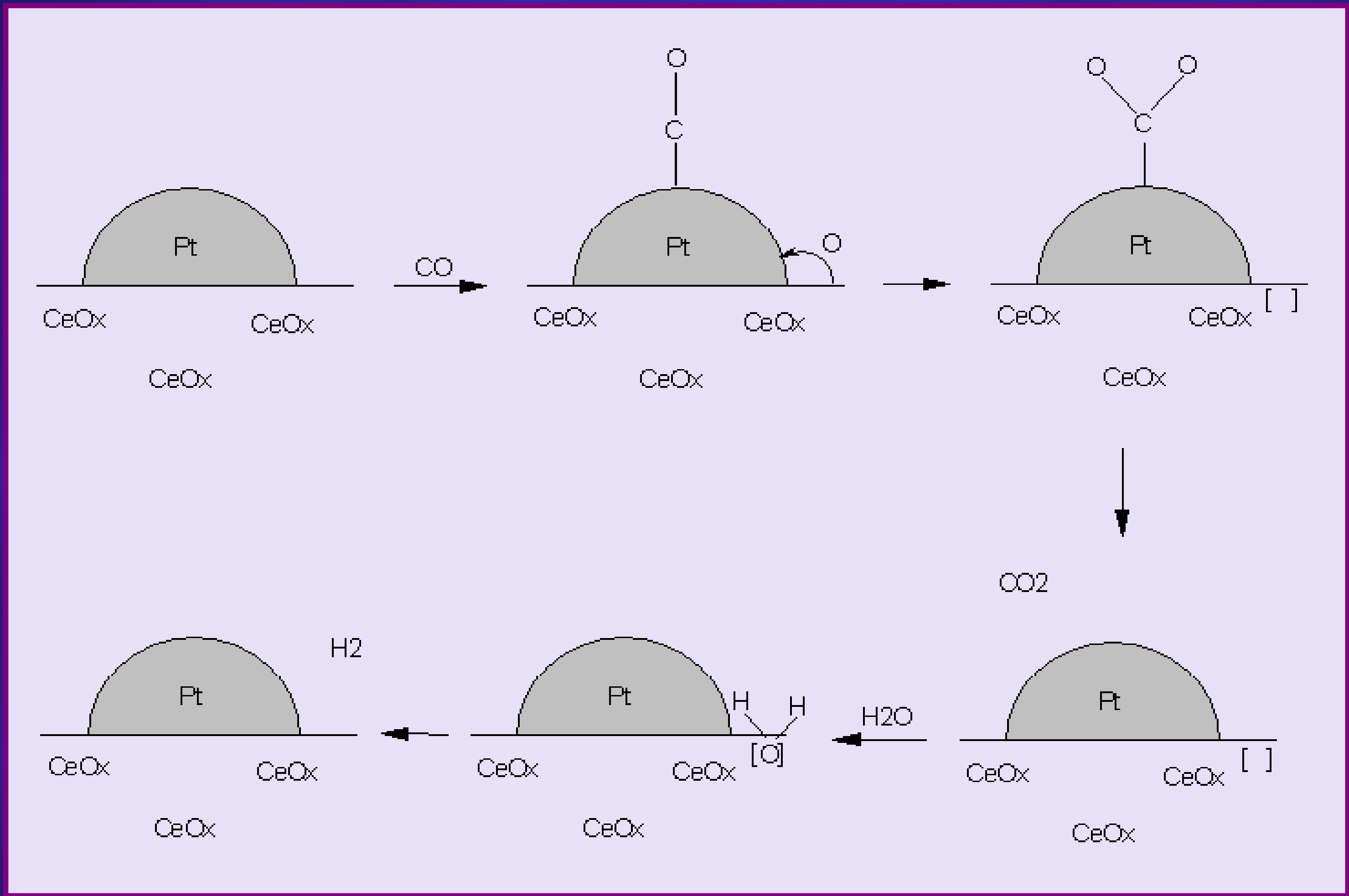


Yu, Thompsett and Tsang. *Chem.Comm.* **2003**, 1522

Water Gas Shift (WGS) Reaction

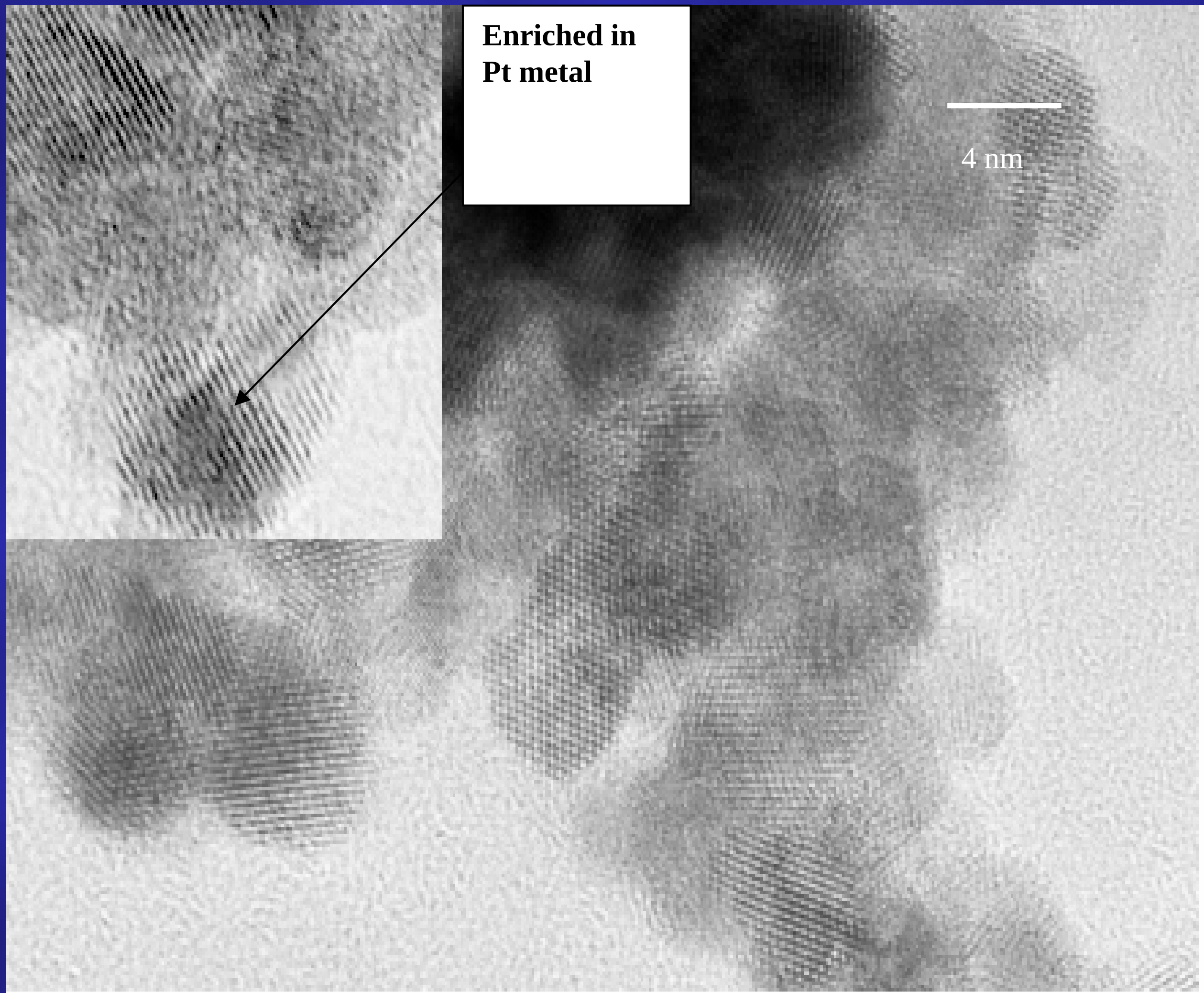
$$CO + H_2O \rightleftharpoons CO_2 + H_2$$

$$\Delta H = -41.2 \text{ kJ/mol}, \ \Delta G = -28.6 \text{ kJ/mol}$$



Davis & Gorte

An enlarged TEM showing typical Pt nanoparticle encapsulated in a ceria sphere



Water Gas Shift (WGS) Reaction

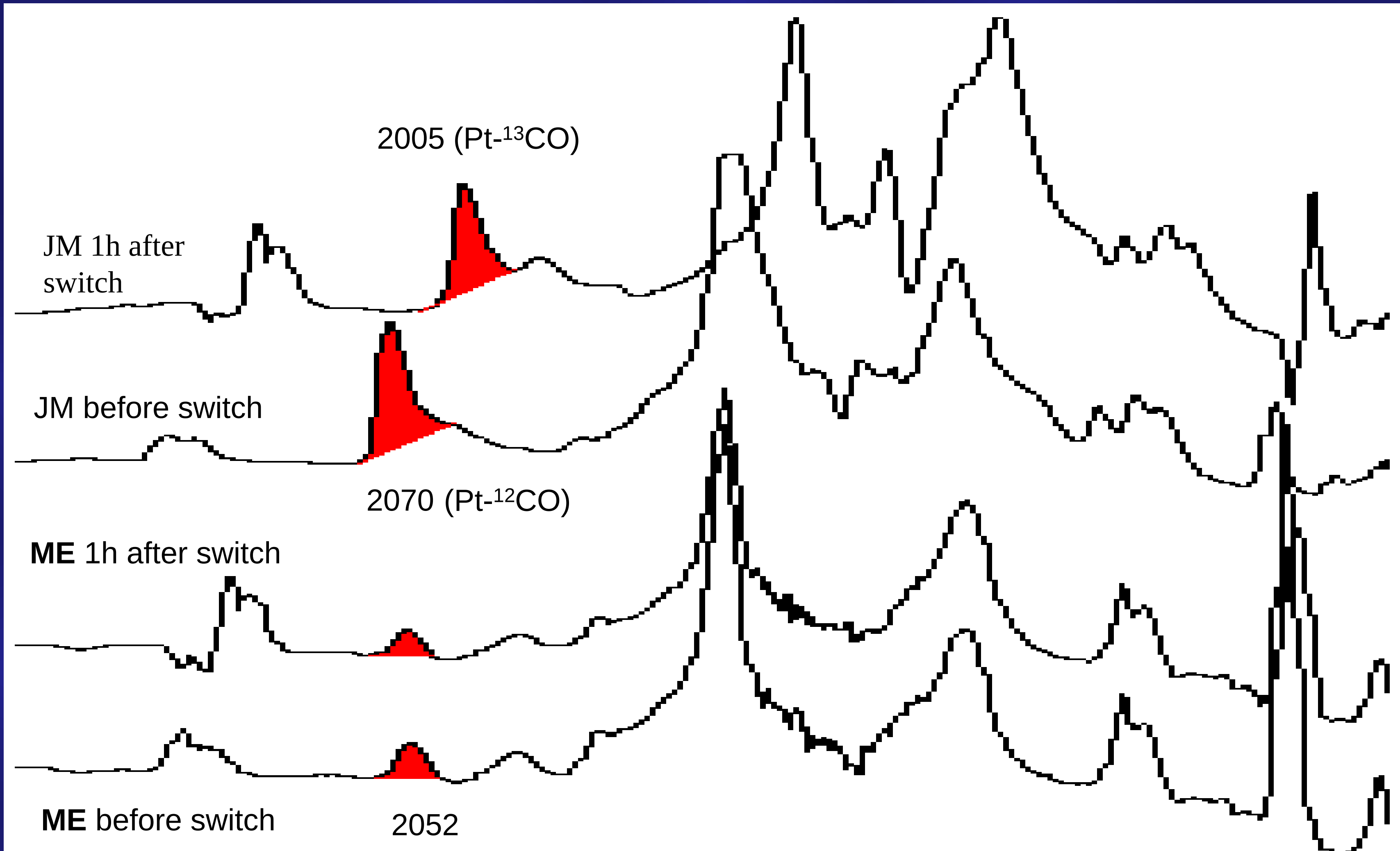
$$CO + H_2O \leftrightarrow CO_2 + H_2$$

$$\Delta H = -41.2\ kJ/mol,\ \Delta G = -28.6\ kJ/mol$$

Catalyst	WGS activity	CH_4 (%)
$Cu/ZnO/Al_2O_3$	55.4	0.03
Co-precipitated 2%Pt/ceria	58.6	1.01
Impregnated 5%Pt/ceria	53.2	1.43
5%Pt@ceria	59.2	0

6 % CO, 7.5% CO_2,, 0.78 CH_4, 25 % H_2, 23% H_2O balanced with N_2 at GHSV of ~100, 000 h^{-1} at 400°C

S.C. Tsang, C.M.Y. Yeung and D. Thompsett, *GB patent* 0402104.4 filed on 6 Feb (2004).



(left) Typical DRIFT spectra obtained under steady-state RWGS conditions at T = 498 K with 1%12CO2 + 4% H2 in Ar and switching to 1% 13CO2 + 4% H2 in Ar over both JM (2Pt%/CeO2) and 5Pt%MEs sample

C.M.Y. Yeung, F. Meunier, R. Burch, D. Thompsett and S.C. Tsang, *J Phys. Chem. B*, **2006;** *110*(17) 8540.

Optimisation in the core-shell geometry

Catalyst	Ceria thickness	Band transition/ eV	WGS activity/%
Ceria	3.7	3.18	-
MEs-1.0 Pt%	3.6	3.20	0.4
MEs-2.8 Pt%	3.0	3.29	6.6
MEs-5.0 Pt%	1.7	3.33	62.5
MEs-10 Pt%	4.4	3.31	47.7
MEs-27 Pt%	6.3	3.22	7.6
MEs-5%Pt/Au	-	3.42	70.6

Yeung, Yu, Fu, Thompsett, Petch, Tsang . *J. Am. Chem. Soc.*, **127** (51), 18010, 2005.

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Dr Paul Collier and Dr Stan Glounski