High Throughput Nanomaterials Discovery - a UK perspective

Steve Fletcher, InsightFaraday Partnership

Jawwad Darr, Queen Mary University London

Chemistry Innovation
Knowledge Transfer Network

Insight
High throughput technologies

The view of the UK Government

- UK industry has a poor innovation performance
  - low R&D spend
  - patent output
- but it has a good science base
  - number of citations per head
- and there are no longer any government laboratories…

The Solution

- Some government funded activity aimed at improving the take-up of new technology by industry, aimed at increasing and accelerating innovation

Chemistry Innovation
Knowledge Transfer Network

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High throughput technologies

The solution from 1997 - 2005

- **Faraday Partnerships**

Government funded activities aimed at

- developing industry-academic partnerships in order to generate increased innovation
- helping the government to channel its spend on science and technology
- identifying requirements for skills and training
- all in focused areas of technology

Chemistry Innovation
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Insight
High throughput technologies

InsightFaraday

- Started in September 2002
- Purpose (version 1)
  - **to promote the development and exploitation of <u>high throughput technologies</u> for product and process development for the benefit of <u>UK industry</u> and the <u>UK science base</u>**
- Purpose (version 2)
  - **to transform the research and development processes in a range of industry sectors in order to**
    - **increase the rate of product & process innovation,**
    - **identify new product options with greater confidence**
    - **reduce time to market and**
    - **improve the competitive position of UK companies.**

What did InsightFaraday achieve?

- Generated a research portfolio of value > £23M, including projects funded by
  - **The UK research councils**
  - **The UK Department of Trade & Industry (DTI)**
  - **The European Commission**
- Developed a commercial product ('LabSmart') to help laboratories improve their implementation of high throughput technologies
- Organised a series of events - targeted workshops, High Throughput Formulation Screening etc.
- Promoted HTT through a quarterly newsletter etc.
- Produced the UK Roadmap for High Throughput Technologies
- Influenced government funding agencies, particularly EPSRC

Chemistry Innovation
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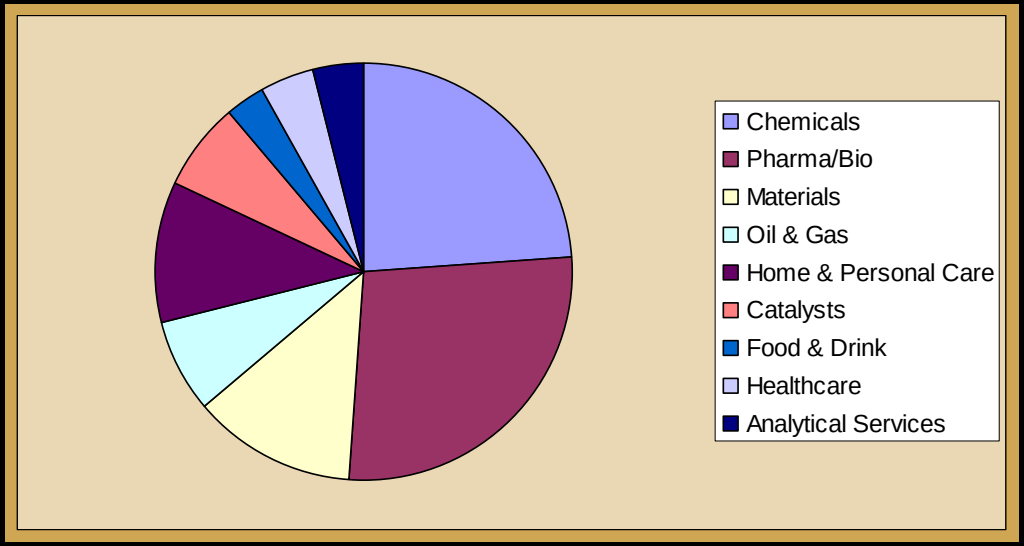
The future ?

- The DTI decided to change the funding in 2005 - introducing 'Knowledge Transfer Networks'
- Insight (plus two other Faradays) is now incorporated in the 'Chemistry Innovation KTN'
- Aim is to improve the innovation performance of the 'Chemistry Using Industries'
- Focus changed from HTT to Chemistry
  - But HTT recognised as a key innovation platform within the KTN

The InsightFaraday Network

- Contacts with over 100 companies and 50 UK university departments
- Can quickly identify partnerships and assemble consortia
- Hence develop collaborative R&D, enable technology transfer, or whatever it takes



Jawwad Darr, Queen Mary University London
Malcolm Connah, Malvern Instruments

Chemistry Innovation
Knowledge Transfer Network

Insight
High throughput technologies

NIST, Washington, USA, 25th April

High Throughput Nanomaterials Discovery

Dr Jawwad A. Darr

Clean Materials Technology Group
http://www.qmul.co.uk

Contents

- Selected UK nano / HT
- Nano-materials, photocats
- HT-nano research
- Wish list for HT-nano
- Summary

Combi & Nano

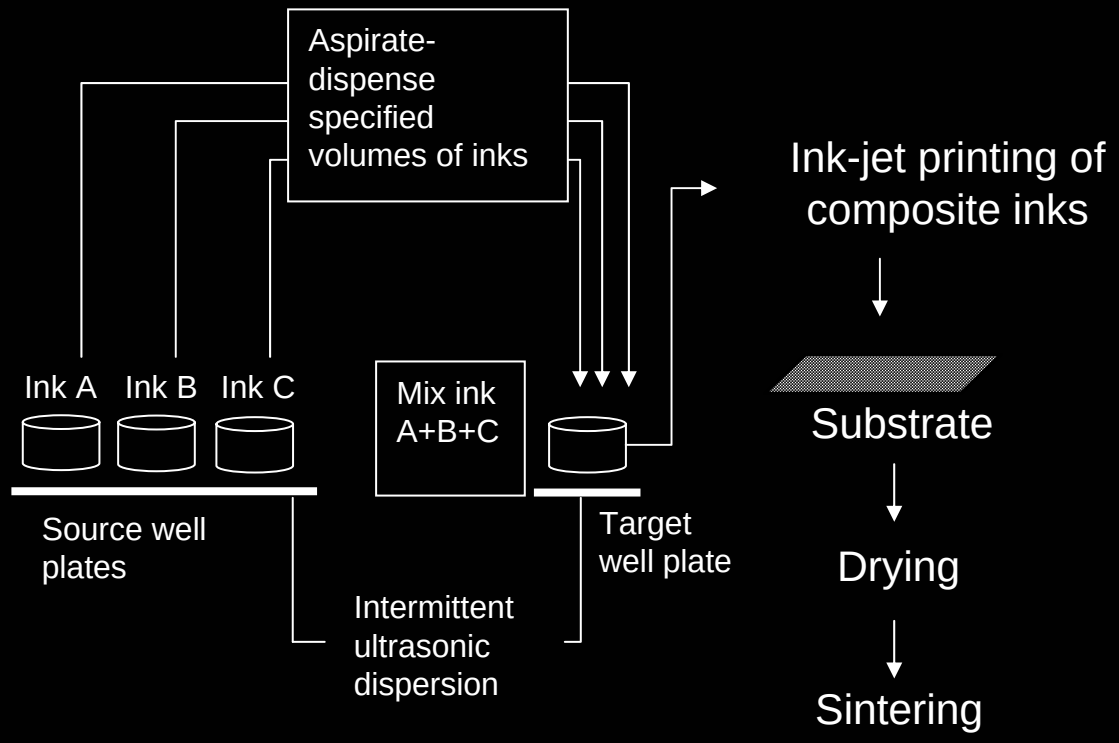
EPSRC

http://gow.epsrc.ac.uk/

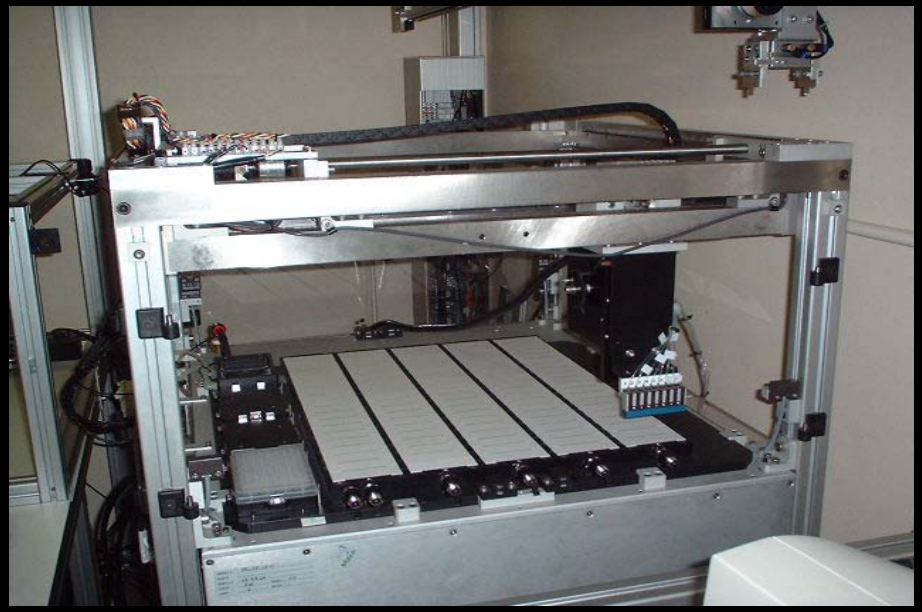
then click on “grants portfolio”

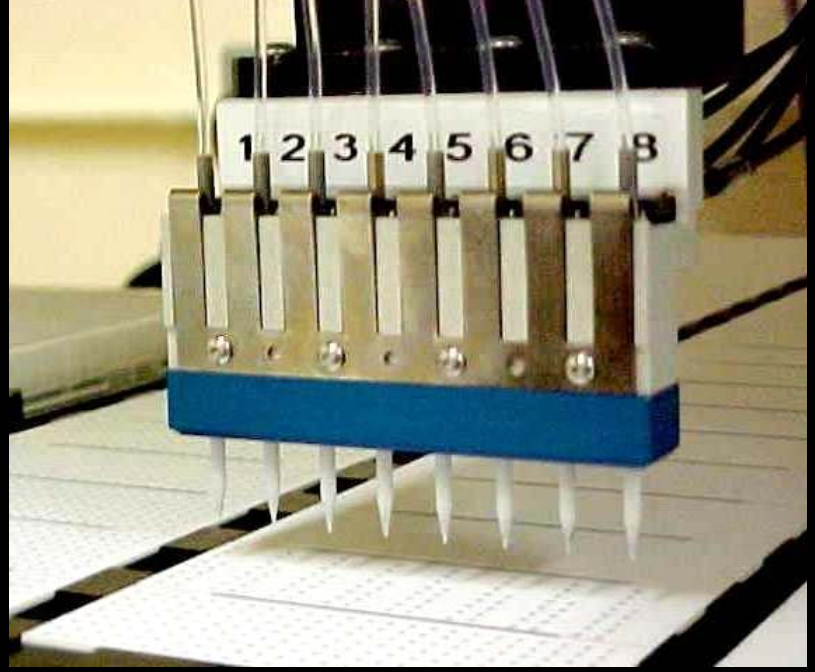
LONDON
2012

LUSI



Sequence for forming samples by well plate reformatting and printing

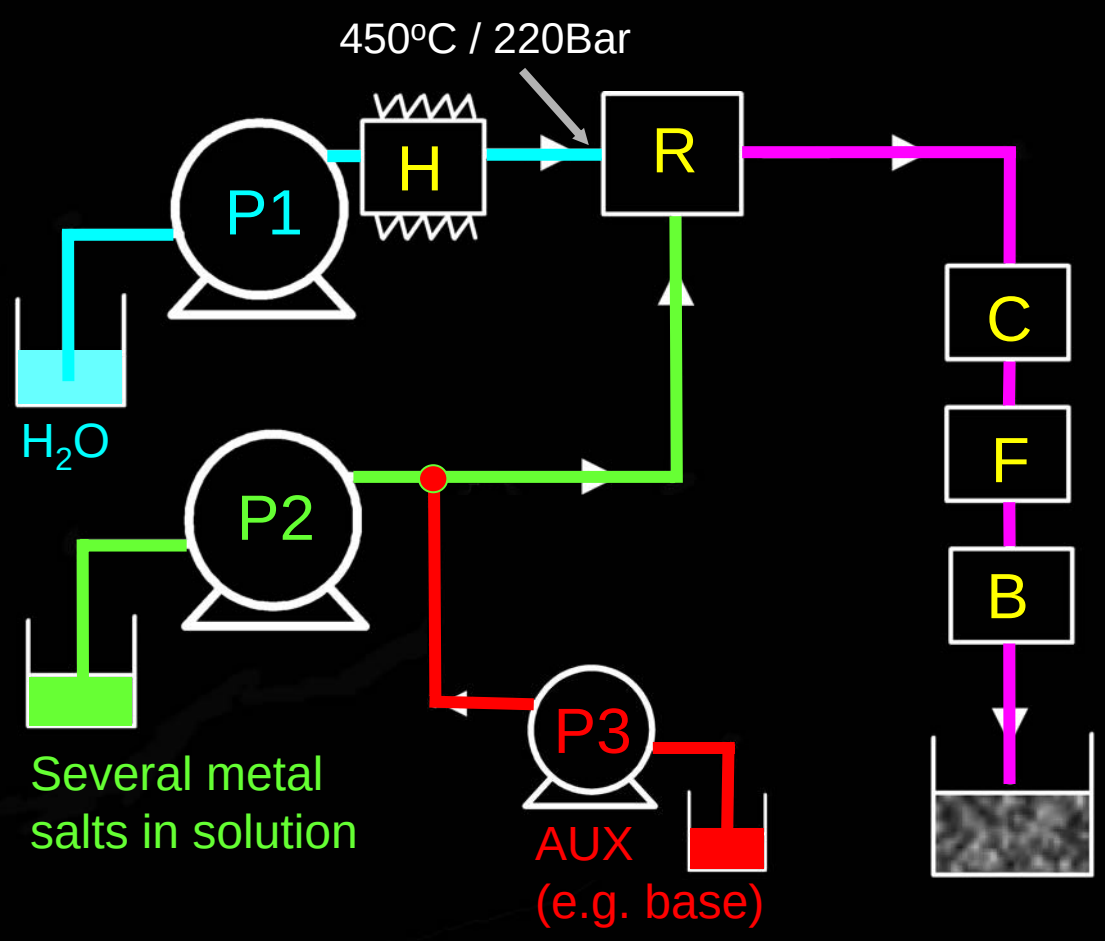




The printer has eight aspirating-dispensing nozzles each controlled by an electromagnetic valve and a 200,000 step linear stepper acting on a 1.5 ml syringe. These nozzles first reformat well plates (left of picture), then aspirate from the target wells and print onto ceramic tiles. The robot gripper can just be seen (top right).

Continuous Hydrothermal Syntheses of Nanoparticles

Hydrothermal flow synthesis



System Variables

- T, P, [conc]
- Metal ratio
- pH, ε, density
- Aux feeds
- Water degas ?
- MS_x, M_yPO_4

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

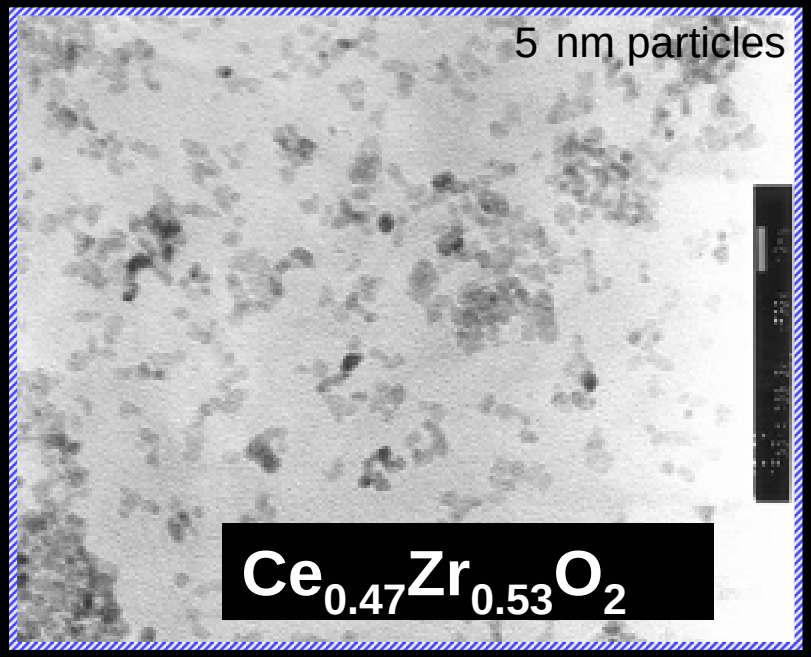
Advantages; Hydrothermal Flow

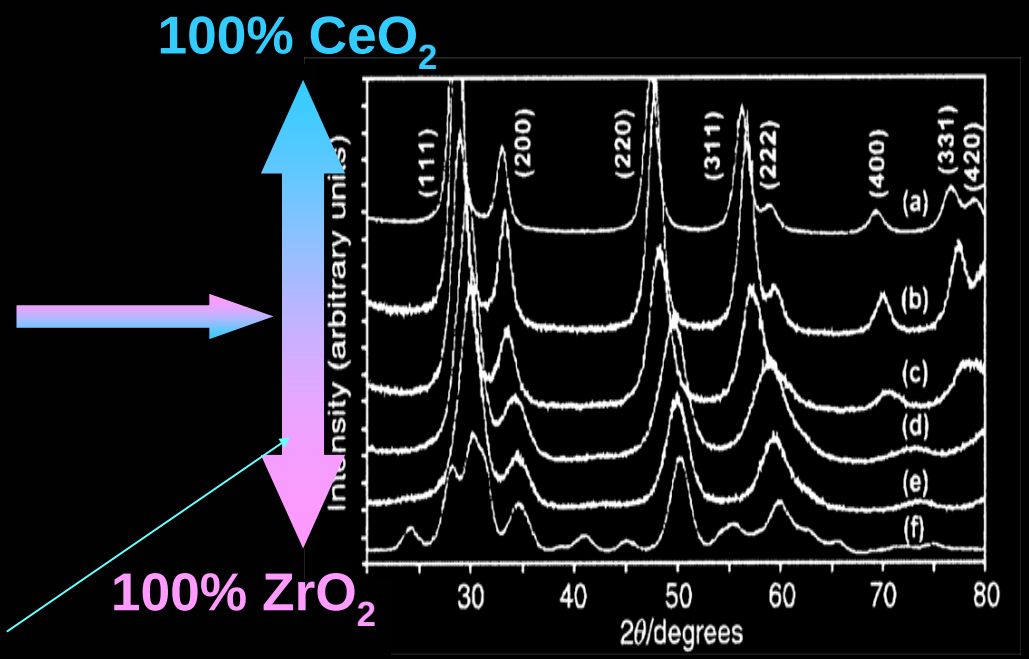
- Nanoparticles
- Green technology
- Few steps, Rapid
- Controllable
- Unique composition
- Crystalline, High SA
- Homogenous doping
- Heat treated to single phase

Current Nano Research

- Fuel cell components
- Li battery materials
- Photocatalysts / sunscreens
- Bioceramics
- Dielectrics / semiconductors
- Magnetic nanoparticles
- Metallics, alloys

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





A CONTINUUM OF COMPOSITIONS ARE POSSIBLE

Darr, J. A.; Poliakoff, M. ACS, Chemical Reviews 1999, 99, 495-541.
Darr, J. A.; Cabanas, A.; Lester, E.; Poliakoff, M., J. Chem. Soc., Chem. Commun., 2000, 901.
Darr, J. A.; Cabanas, A.; Lester, E.; Poliakoff, M., J. Mater. Chem., 2001, 11, 561 – 568.

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

X in ppt	Phase "as ppt"	Phase after 1000^0C HT
0	cubic	cubic
0.2	cubic	cubic
0.5	cubic	c / t mix
0.8	c / t mix	t
0.9	t	t
1.0	t;m 60:40	t;m 20:80

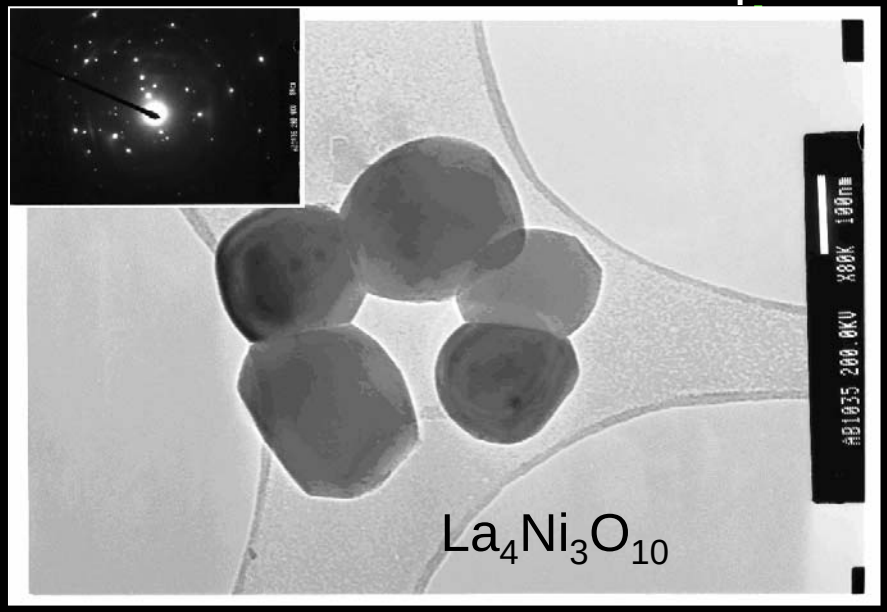
Literature range	Literature assignment
X<0.2	cubic
0.35>x>0.2	t"
0.6>X>0.35	t'
0.8>X>0.6	t
X>0.8	m
X=1.0$	m (usually)$

$ For very small particles has been shown to be tetragonal or cubic

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

$La_x(OH)_{3x}Ni_yO_z$

Nanopowder



$La_4Ni_3O_{10}$

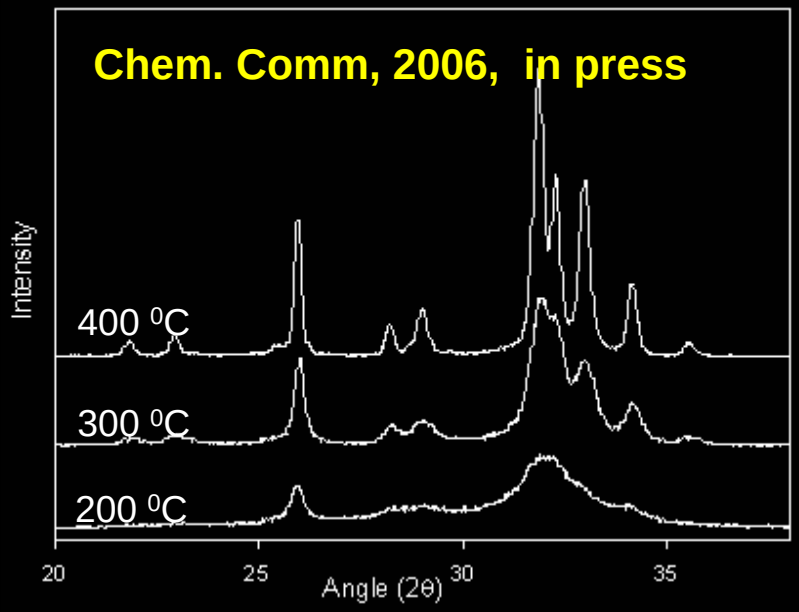
4:3 ratio

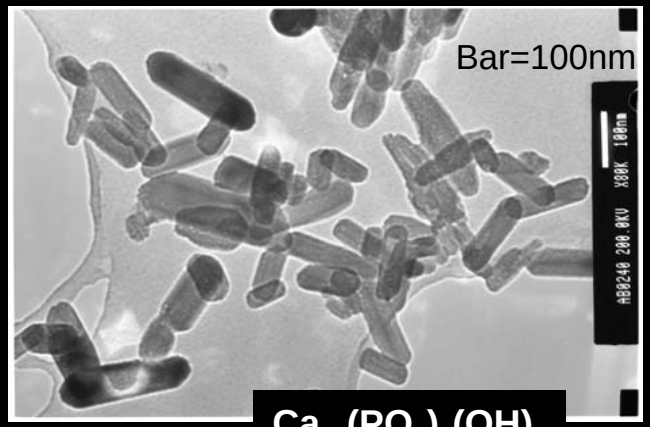
10h 1075 C

$La_4Ni_3O_{10}$

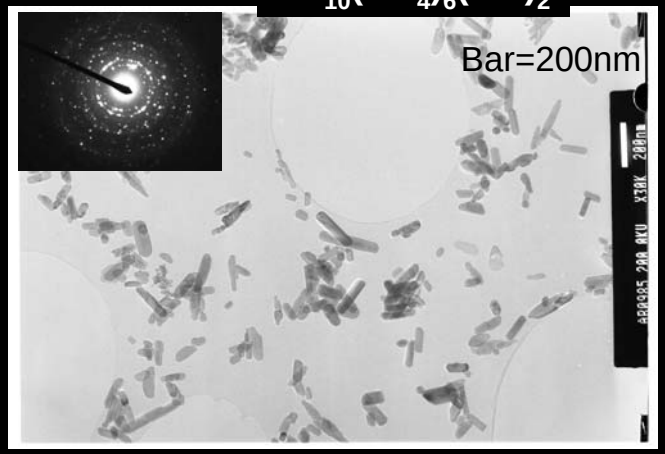
Ion and e- conductor

“Instant Hydroxyapatite”





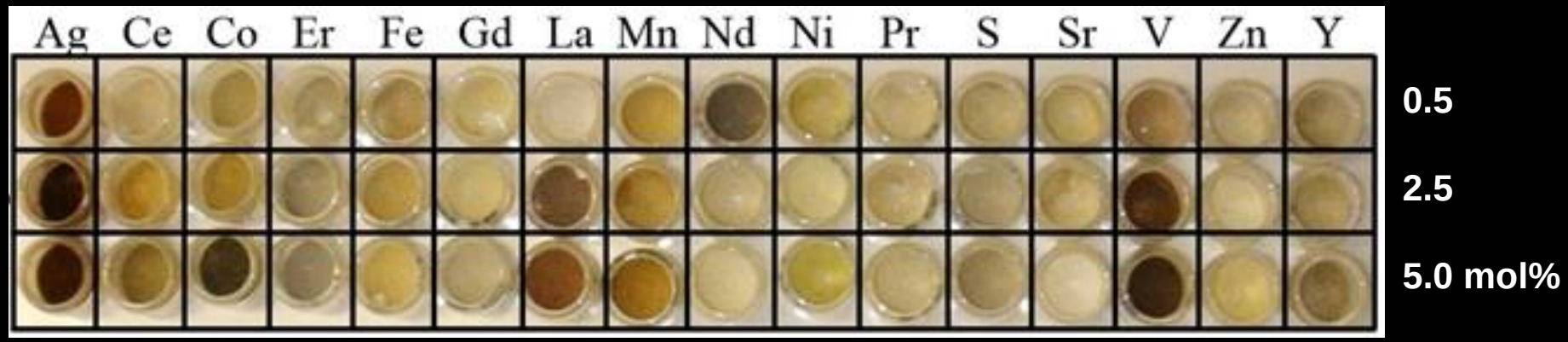
$Ca_{10}(PO_4)_6(OH)_2$



Particles = ca.150x20nm

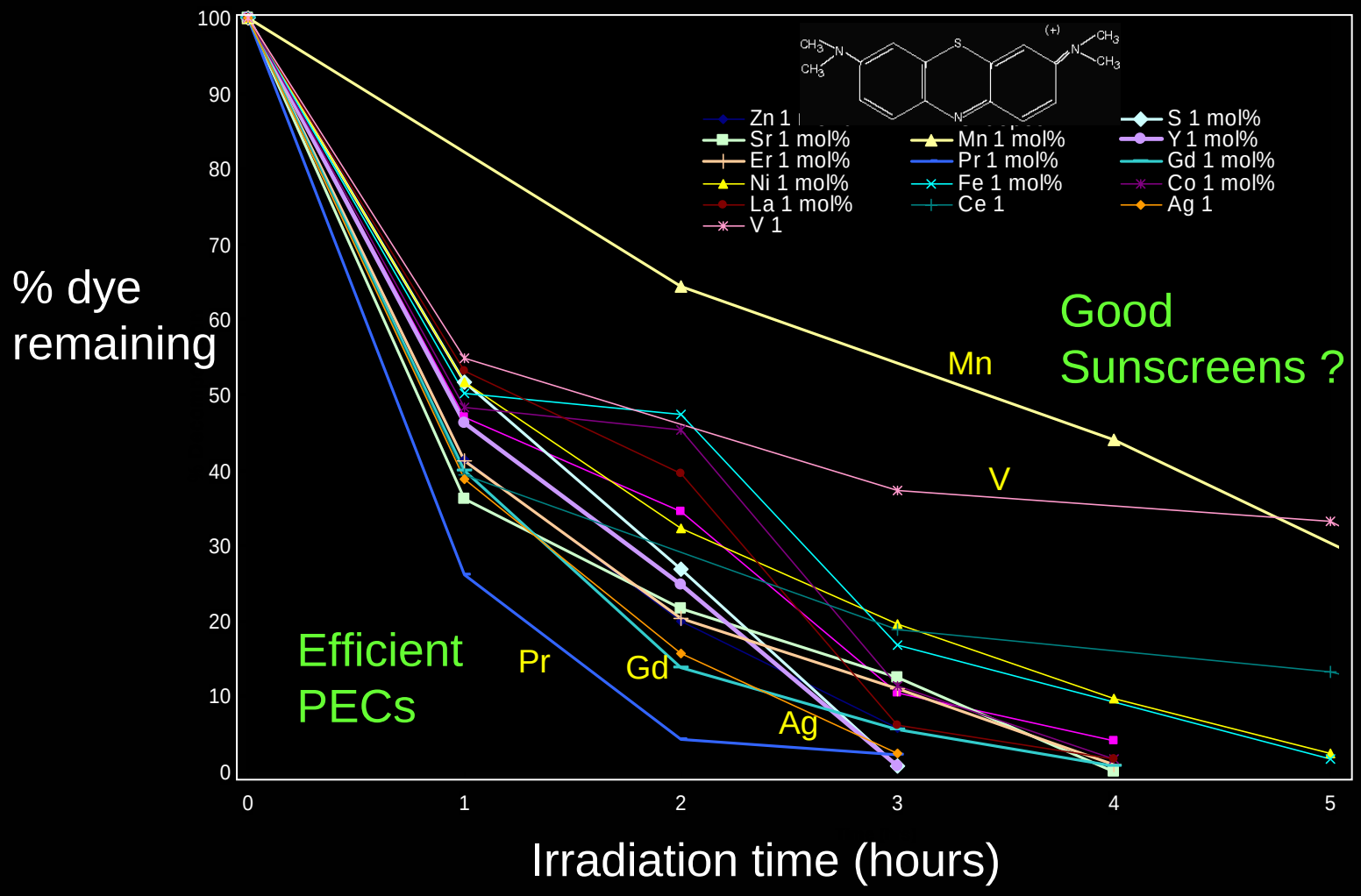
- Continuous synthesis
- Time = 2 mins (18h norm)
- Ca:P ratio / doping

Doped nano-TiO_2 photocatalysts



16 dopants x 3 concentrations
= 48 samples

Photocatalytic Degradation of MB in 0.5 % Doped Nano-TiO_2 Samples vs Time under VISIBLE LIGHT



High Throughput Nanomaterials Discovery

"To identify New Photocatalysts to split water"

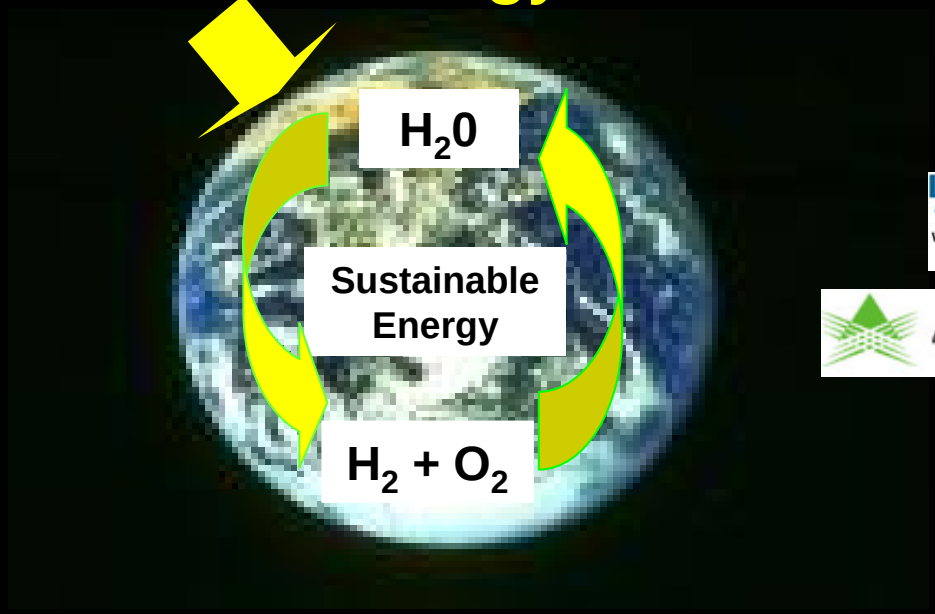
"High Throughput Nanomaterials Discovery"

Queen Mary
University of London

EPSRC

Total = $1.8m
QM = $1.6 m
3 years
Grant reference

Solar Energy



SRI International

Insight
High throughput technologies

Johnson Matthey

London Technology Network
Ideas into Action

Labman Automation
www.labman.co.uk

AMR TECHNOLOGIES INC.

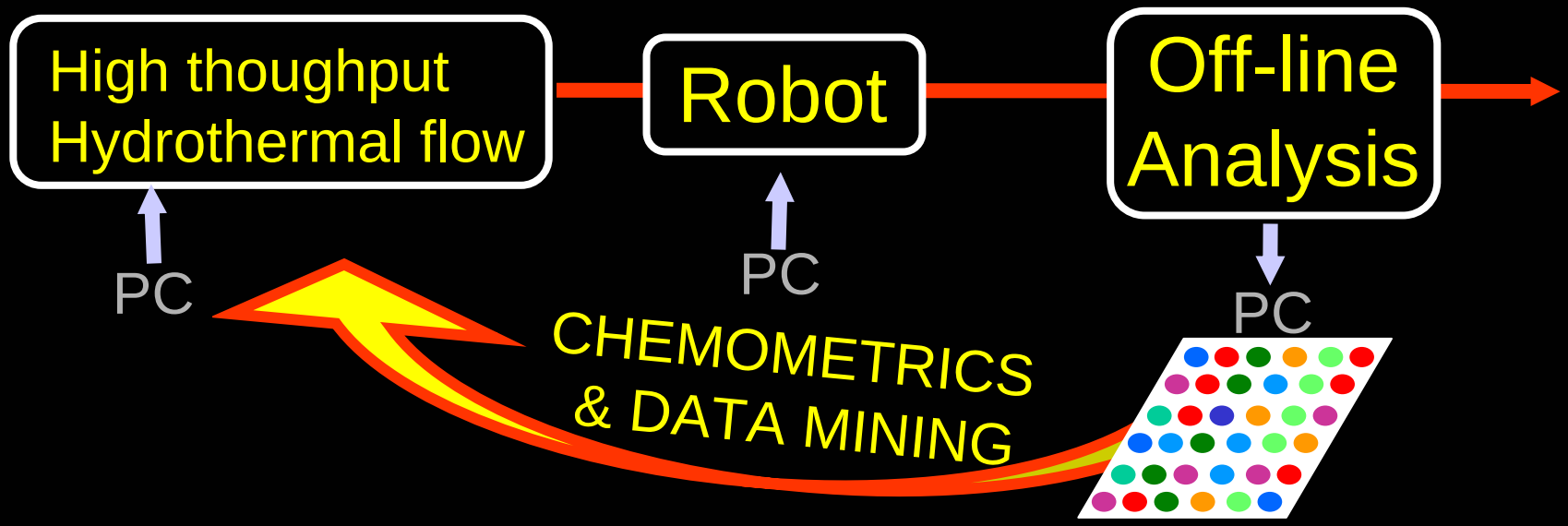
SunChemical

Thermo
ELECTRON CORPORATION

Hydrogen Solar

University of Leeds

Automated HT nanoparticle synthesis and screening

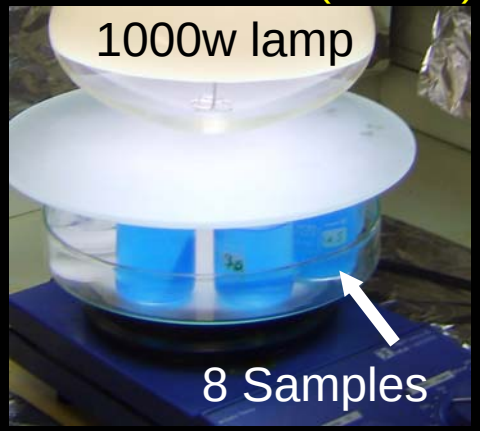


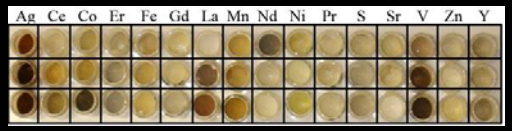
- Automated HT-Raman
- HT-XRD,
- HT photocatalysis

- UV-Vis
- Microwave dielectric measurement
- Ion and electrical conductivity

HT Photocat. testing

1st Generation (UV/Vis)





2nd Generation (pressure sensor)

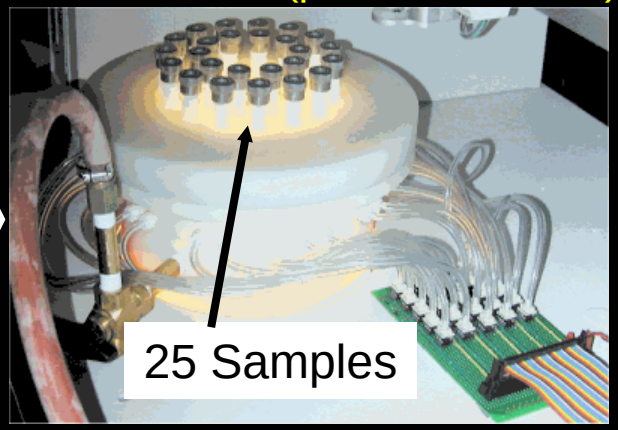


Photo B. Macqueen,
SRI international

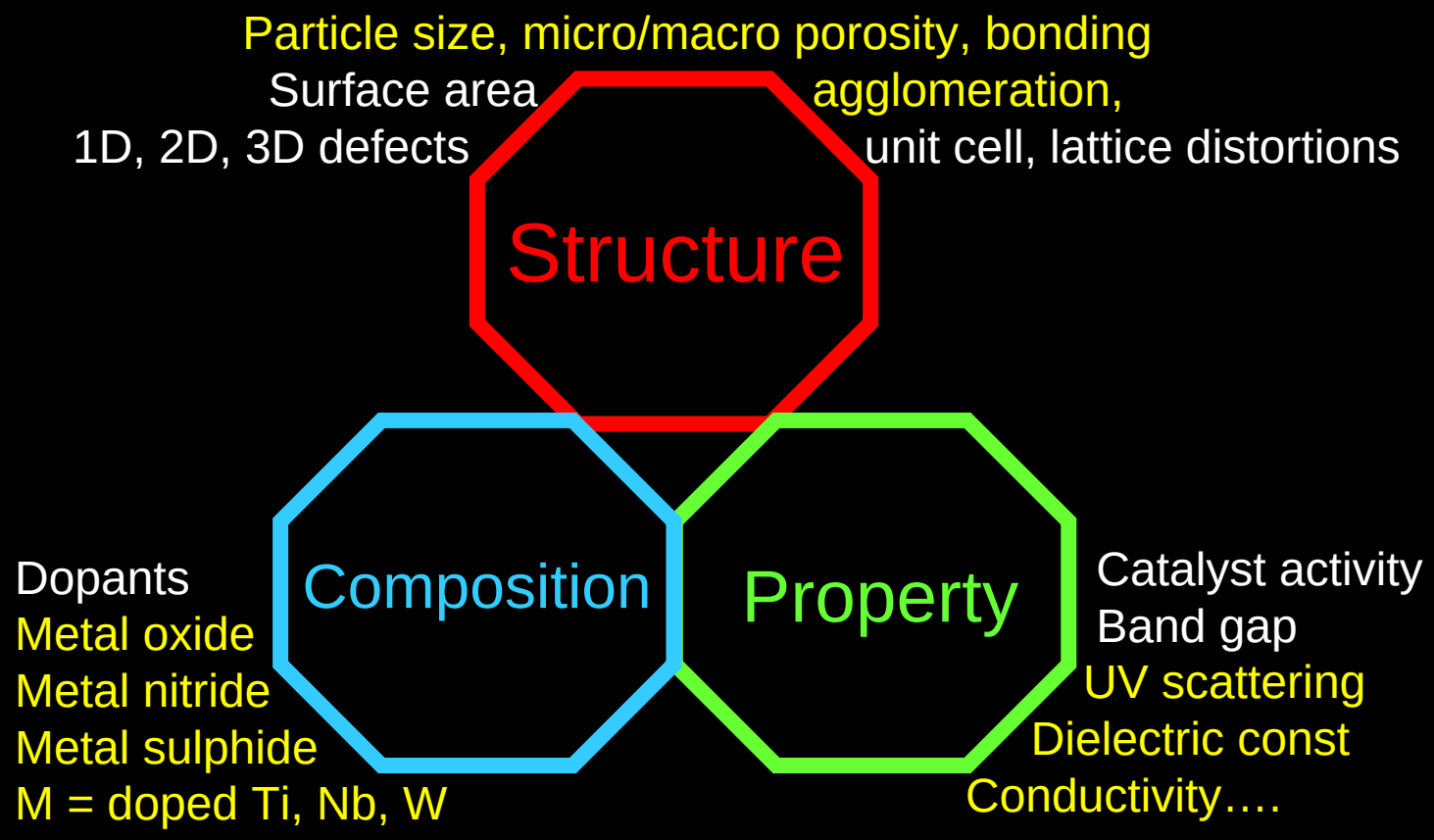
3rd Generation (2006 +)

- Larger array
- Faster equilibration

SRI International

With B. Macqueen 2006

Chemometrics / Data Mining



Seeking….

- HT property measurement or analysis of inorganic nanoparticles / slurries
  - HT-Raman
  - HT-UV/Vis.
  - HT-XRF (metal analysis)
  - HT Imaging of nanoparticles
  - HT other…

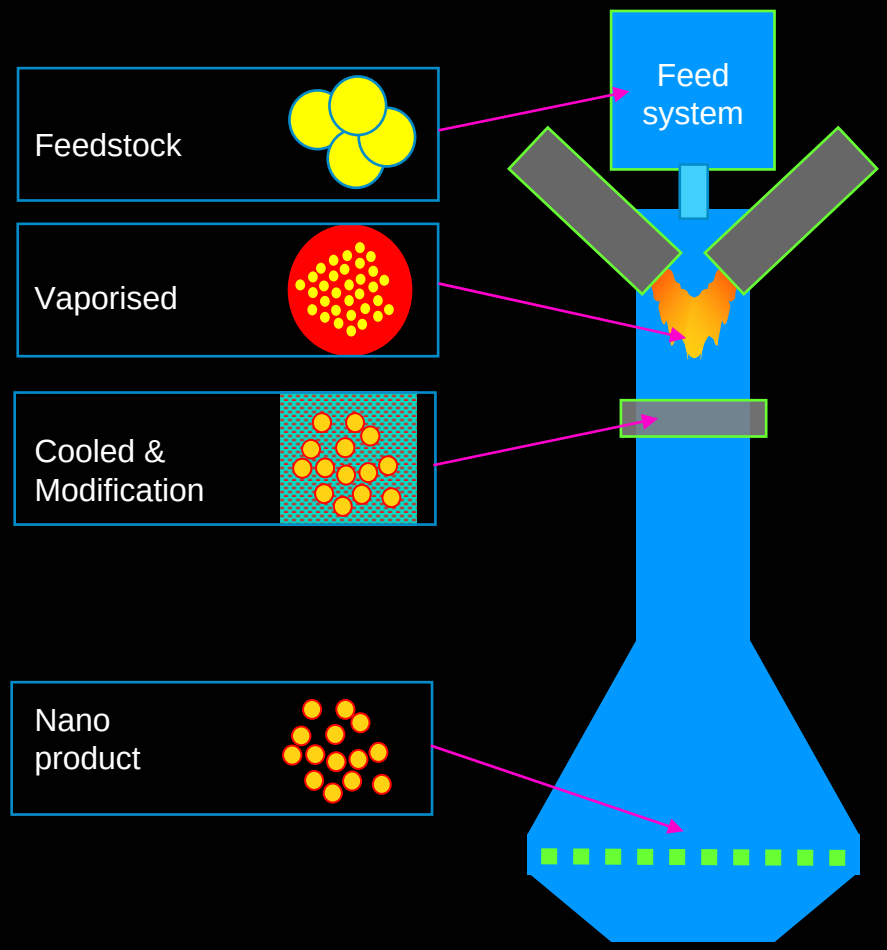
Nanomaterials

ACORN

Foresight LINK Awards

		BNFL	CERAM	DOW CORNING	DUPONT TEIJIN	DYTECH	DYNAMIC CERAMIC	HUNTSMAN	ICI / UNIQEMA	PROCTER & GAMBLE	QINETIQ	SCHLUMBERGER	SYNGENTA
BIRMINGHAM	B1 Supercritical [Jonathan Seville]												
	B2 Nanoadhesion [Jon Preece]												
BRISTOL	BR1 Polymerisable surfactants [Julian Eastoe]												
	BR2 Nanocomposites [Terry Cosgrove]												
	BR3 Deposition of nanoparticles [Jason Riley]												
	BR4 Nanoscale colloidal systems [Terry Cosgrove]												
LEEDS	L1 Processing nanoparticles [Andrew Bell]												
	L2 Nano-assemblies for controlled release [M Ghadiri]												
	L3 Aerosol reactor [Dave Goddard]												
LOUGHBORO	LO1 Lab-on-a-Tip [Mike Reading]												
	LO2 Formation of compacts [Jon Binner]												
	LO3 Retention of nanostructure [Jon Binner]												
NEWCASTLE	N1 Modelling calcination [Terry Egerton]												
	N2 Nanoparticulate titanium dioxide [Terry Egerton]												
UMIST / ANCHESTER	U1 Rapid precipitation/nucleation templating [Roger Davey]												
	U2 Stabilisation of nanoparticles [Andrew Collett]												

The QNL Tesima® plasma process



- Advantages of using plasma
  - material uniformity
  - chemical composition
  - no contamination
  - better control
  - scalability
  - flexibility
  - controllability
  - safety

Sample materials fabricated so far

Tesimet ®

Metals

Aluminium

Copper

Silver

Nickel

Cobalt

Titanium

Stainless Steel

Tungsten *

Boron *

Tantalum *

Tesimox ®

Oxides

Zinc Oxide

Copper Oxide

Cerium Oxide

Tin Oxide

Cuprous Oxide

Titanium Dioxide

Nickel Oxide

Zirconium Oxide

Tesimide ®

Carbides and Nitrides

Tungsten Carbide

Silicon Carbide*

Boron Nitride*

Silicon Nitride*

Boron Carbide*

* planned

UHV methods (PVD) to HT synthesis

Ilika Technologies Ltd

EPSRC Grant Reference: GR/S27238/01 **Ends:**31 July 2006

Accelerated Discovery of Li Battery Materials

Professor J Owen, Professor M Weller, Southampton

1. Chemical synthesis, vacuum deposition and pulsed laser ablation techniques
2. 64 distinct compositions for parallel analysis.
3. HT XRD, eight 64-channel electrochemical stations

EPSRC Grant Reference: EP/D062381/1 (Jan 2009)

High Throughput Synthesis and Screening of Novel Hydrogen Storage Materials

PI: Professor WIF David of ISIS

HT thin film techniques, structural & H absorption
•gram scale quantities of the most promising compositions.

•refine our theoretical and modelling studies, to optimize result or identify new families.

High-throughput intracellular import

Mark Bradley, Edinburgh

- Small polymer beads that mammalian cells take up; dyed with up to 100 different colours
- Biologically active molecules like enzymes or DNA can be attached onto beads
- explore the internal working of the cell in real time
- e.g. special nucleic acids (called RNAi) can be used to "shut" down specific genes (which control function) within the cells.
- In theory any gene could be turned off with resulting in changing of cell phenotype (e.g. how a cell appears under a microscope),
- To control the tissue type formed via a stem cell

.

Summary for HT-Nano

- A few HT-nano groups in UK (current)
- Spread out - low interaction (current)
- Increasing realisation from Industry
- Need more interactions in UK-Other
- Investment growing in MNTs
- QM London

Acknowlegements

- S. Yang,
- S.Shevelin,
- J.Evans,
- J. Elliot,
- I. Rehman,
- ZX Guo

- Dr M. Patel
- Prof M. Braedon
- Dr G. Davies
- Prof Asim Ray

EPSRC

- HEFCE,
- Royal Society,
- HEC Pakistan,
- DTI,
- LDN,
- LTN

- Sun chemical
- Johnson Matthey
- AMR Europe Ltd.

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

- Xue Wang (Leeds)
- Prof Poliakoff (Notts)
- Dr Lester (Notts)
- Stephen Skinner (IC)
- Prof Seifallian (UCL)
- Dr J. Jones (IC)
- Dr J. Anwar (Kings)
- K. Arai (Japan)

www.qmul.co.uk

Nanotech in the UK- various

Blue: Electronics Green: Biomaterials BLACK: characterisation or fabricat

UK Research Activity in Nanotechnolgy

- **Scotland**
- HERIOT WATT UNIVERSITY opto-electronics
- ROBERT GORDON UNIVERSITY biocompatibility, surface charactersiation
- UNIVERSITY OF ABERDEEN biomolecule labelling
- UNIVERSITY OF DUNDEEsemiconductors&sensors
- UNIVERSITY of EDINBURGH silicon technologies, sensors, surface properties
- UNIVERSITY OF GLASGOW opto and bio electronics, biocompatilbility, catalysis
- UNIVERSITY OF PAISLEY thin films & sensors, structural composites
- UNIVERSITY OF STRATHCLYDE polymers, nanoparticles, nanogels, photonics
- UNIVERSITY OF ST ANDREWS photonics
- **Wales**
- UNIVERSITY OF SWANSEA dispersed colloids
- UNIVERSITY OF WALES ABERYSTWYTH nanocrystals, optoelectronics
- UNIVERSITY OF WALES, BANGOR optoelctronics, ferro-fluids, bioelectronics, lab-on-a-chip
- **N. Ireland**
- THE QUEEN'S UNIVERSITY OF BELFAST monolayer magneto-optics
- UNIVERSITY OF ULSTERbio-engineering and advanced functional materials institute

λ**England**

λUNIVERSITY COLLEGE LONDONthin films and sensors

λUNIVERSITY OF BATH nanoparticles, nano-tubes, ceramics

λUNIVERSITY OF BIRMINGHAM nanoscale physics research centre clusters/macromolecules at surfaces:Nanotech Centre

λUNIVERSITY OF BRISTOLnanoparticles, self assembly, interfacial analytical

λUNIVERSITY OF CAMBRIDGEbeam lithigraphy, nanoelectronics, surface properties,

λUNIVERSITY OF CRANFIELDbiosensors, biomimetics, ferro and piezoelectrics

λUNIVERSITY OF DE MONFORT opto-electronics

λUNIVERSITY OF DURHAM Centre for molecular and nanoscale electronics

λUNIVERSITY OF EXETER photonics

λUNIVERSITY OF HULL photovoltaics

λ IMPERIAL COLLEGE OF SCIENCE, TECHNOLOGY AND MEDICINE lithography/epitaxy

λ UNIVERSITY OF KENT electrode/battery devices characterisation

λ LANCASTER UNIVERSITYproximal probe microscopies

λUNIVERSITY OF LEEDS self assembly, biomaterials, nanoparticles, mathematical modelling

λUNIVERSITY OF LEICESTER magnetic nanoclusters

λUNIVERSITY COLLEGE LONDON

λUNIVERSITY OF LONDON, ROYAL HOLLOWAY photonics

λUNIVERSITY OF LIVERPOOL nanoparticles, catalysis, opto-electronics

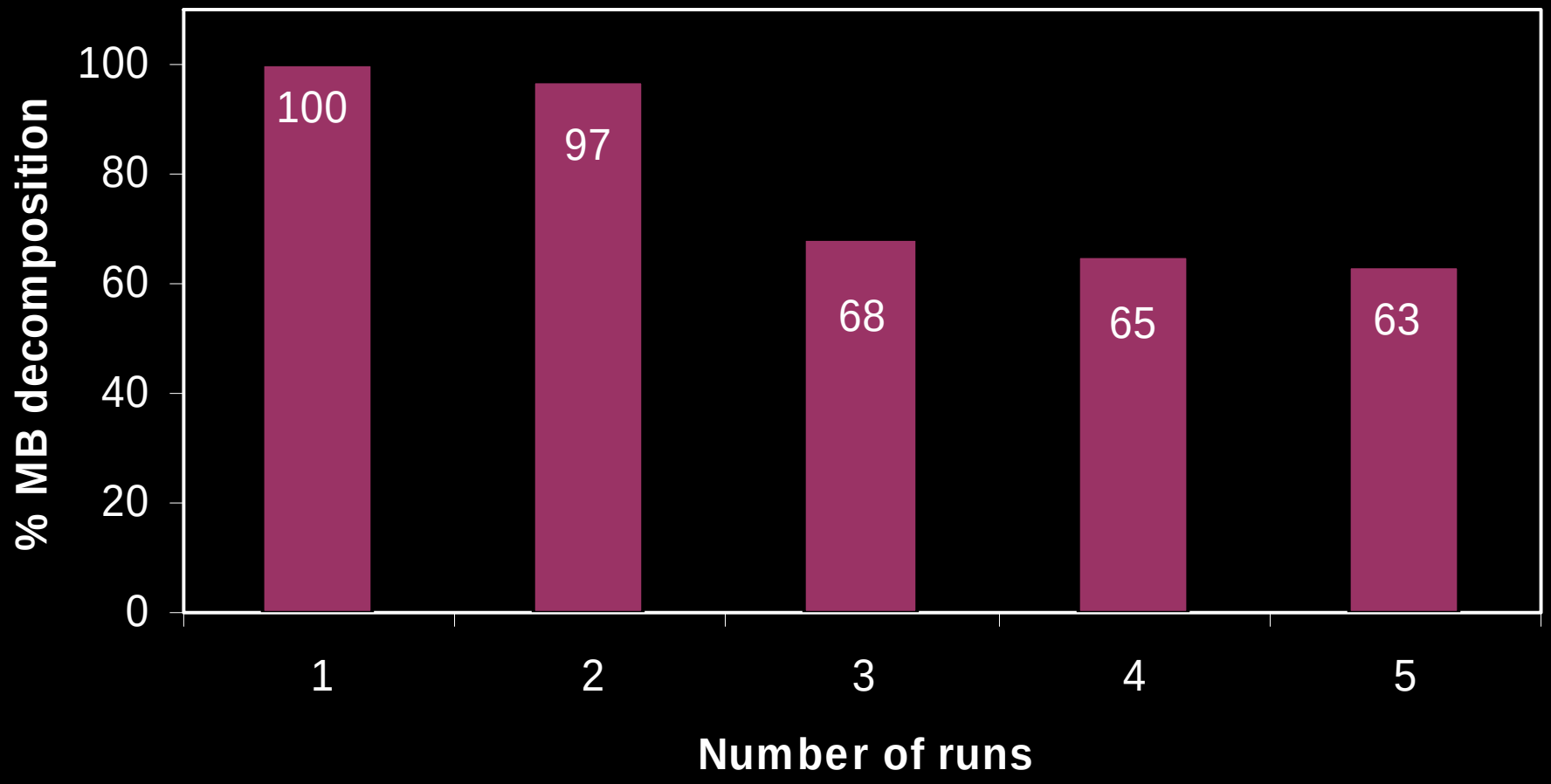
- UNIVERSITY OF MANCHESTER metal clusters, smart materials, magnetoelectronics
- UNIVERSITY OF NEWCASTLE UPON TYNEbiosensors mems
- UNIVERSITY OF NORTHUMBRIA crystal growth
- UNIVERSITY OF NOTTINGHAM biophysical, surface analysis, biocompatibity
- NOTTINGHAM TRENT UNIVERSITY structural nanocomposites
- UNIVERSITY OF OXFORD molecular machines, bioelectronics, MEMS,
- UNIVERSITY OF PORTSMOUTH biomolecular machines
- UNIVERSITY OF READING surface analysis, solid state sensors, catalysis
- UNIVERSITY OF SOUTHAMPTON templated nanostucture fabrication
- UNIVERSITY OF SHEFFIELD semi and optoelectronics, biomimetic soft solids
- UNIVERSITY OF SURREY silicon C nanotube electronics
- UNIVERSITY OF SUSSEX nanotubes/fullerenes
- UNIVERSITY OF TEESIDE TiO2 composites
- UNIVERSITY OF WARWICK nanometrology, MEMs,
- UNIVERSITY OF YORK biomimetics, molecular motors, cell and sub-cell ablation, magnetoelectronics
- CCLRC_RUTHERFORD APPLETON LABORATORY optoelectronics, MEMs, nanofabrication
- CCLRC_DARESBURY XPS
- NATIONAL PHYSICAL LABORATORY
- QINETIQ/ QINETIQ NANOMATERIALS

IRC's in Nanotechnology

- IRC in Nanobiotechnology
  - directed by Professor John Ryan, Head of Condensed Matter Physics and Head of the Physics Department, Oxford University
  - Universityof Glasgow (Professor Jon Cooper)
  - University of York (Dr. Justin Molloy)
  - National Institute for Medical Research.
  - links with the Universities of Cambridge, Nottingham and Southampton.
- IRC in Physics of nanotechnology
  - directed by Professor Mark Welland, Head of the Nanoscale Science Laboratory in the Department of Engineering Cambridge University,
  - Professor Richard Friend (Cambridge, Physics); Dr Mark Blamire (Cambridge, Materials Science and Metallurgy); Professor Chris Dobson , (Cambridge, Chemistry)
  - University College London (Dr Andrew Fisher, Physics and Professor Michael Horton, Medicine)
  - University of Bristol. (Professor Mervyn Miles Physics) .

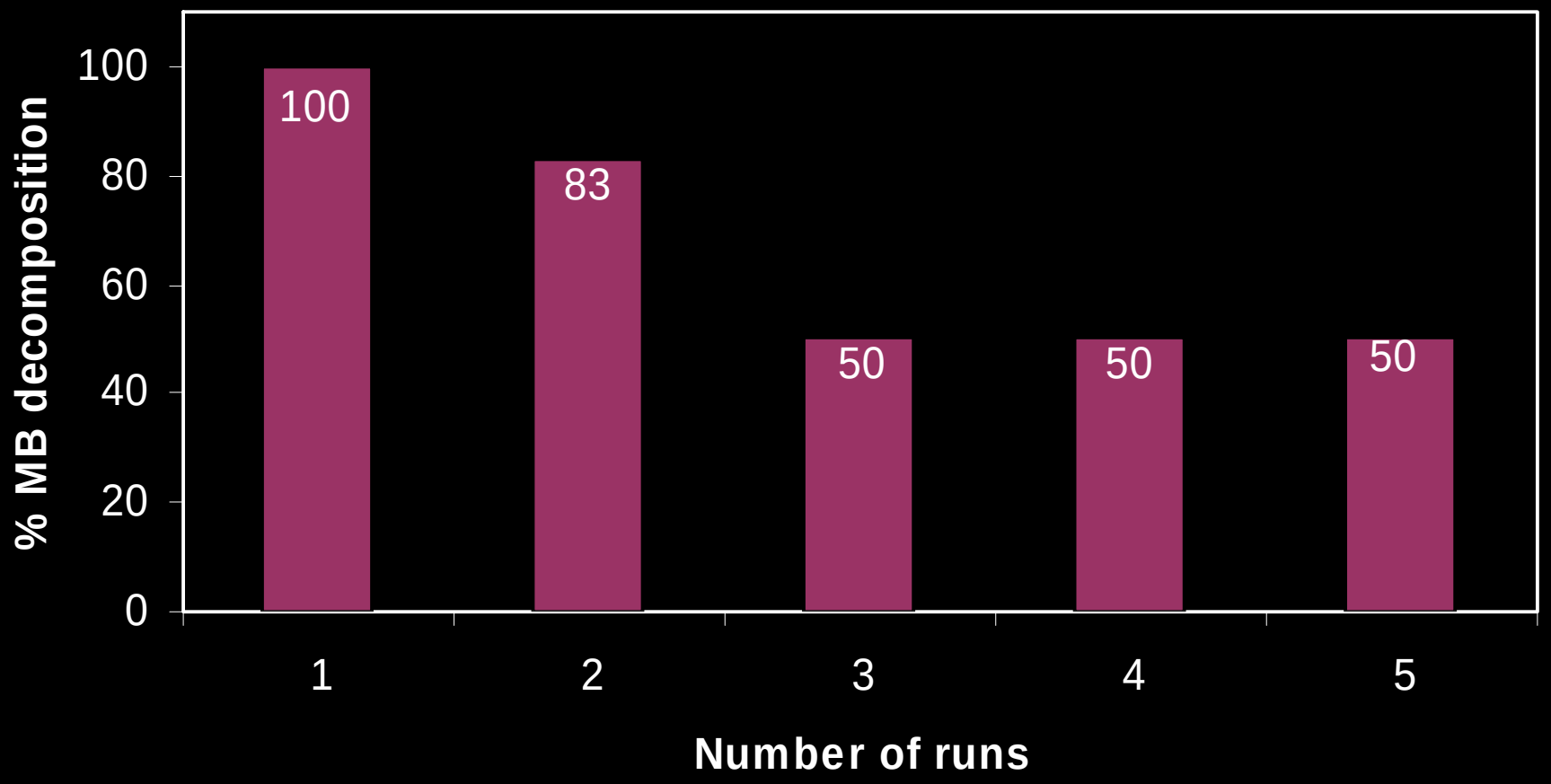
1%Ag in TiO_2

% of MB decomposed in 3h



1% Pr in TiO_2

% of MB decomposed in 3h

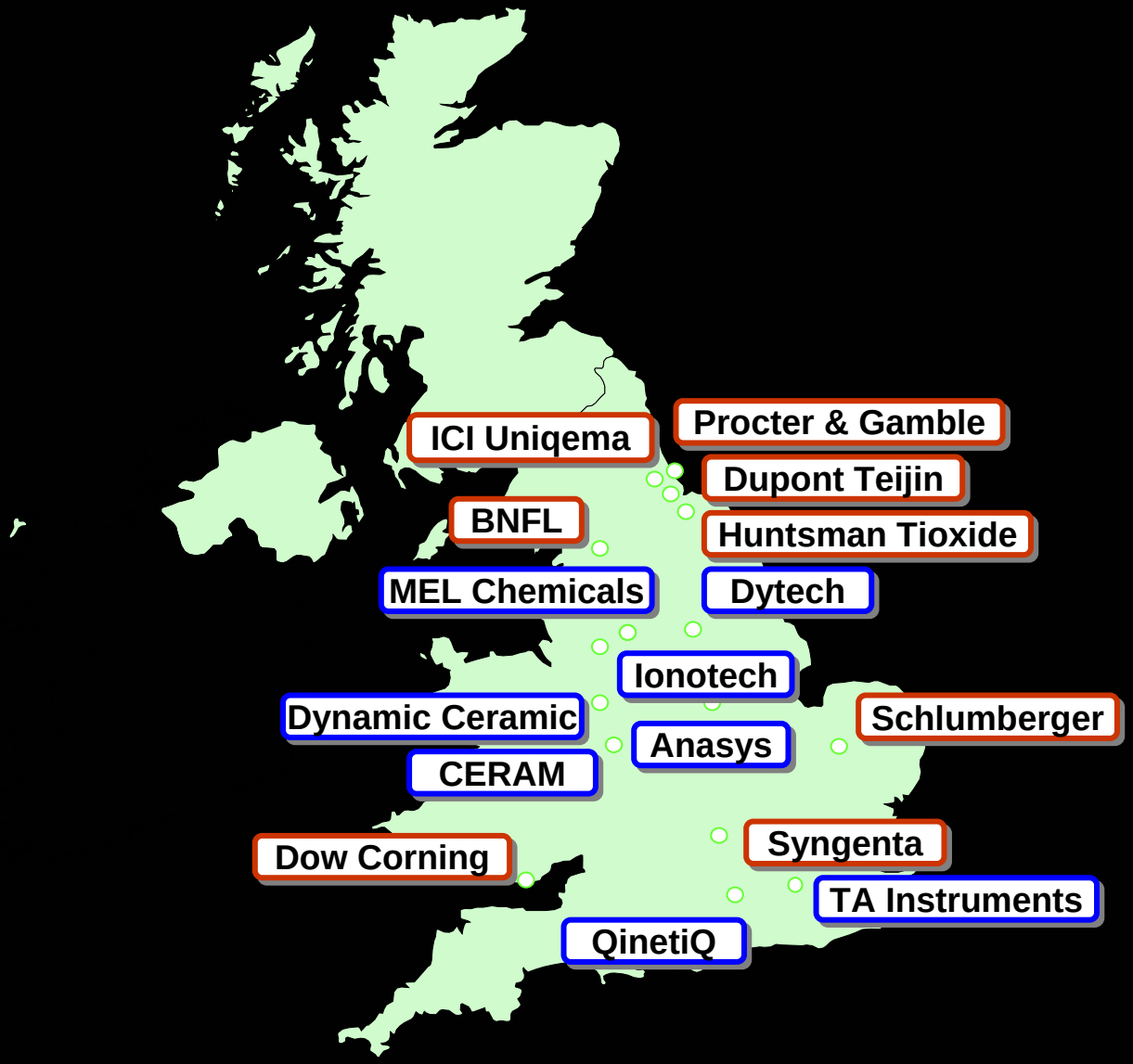


University Innovation Centre for Nanotechnology at the University of Newcastle.

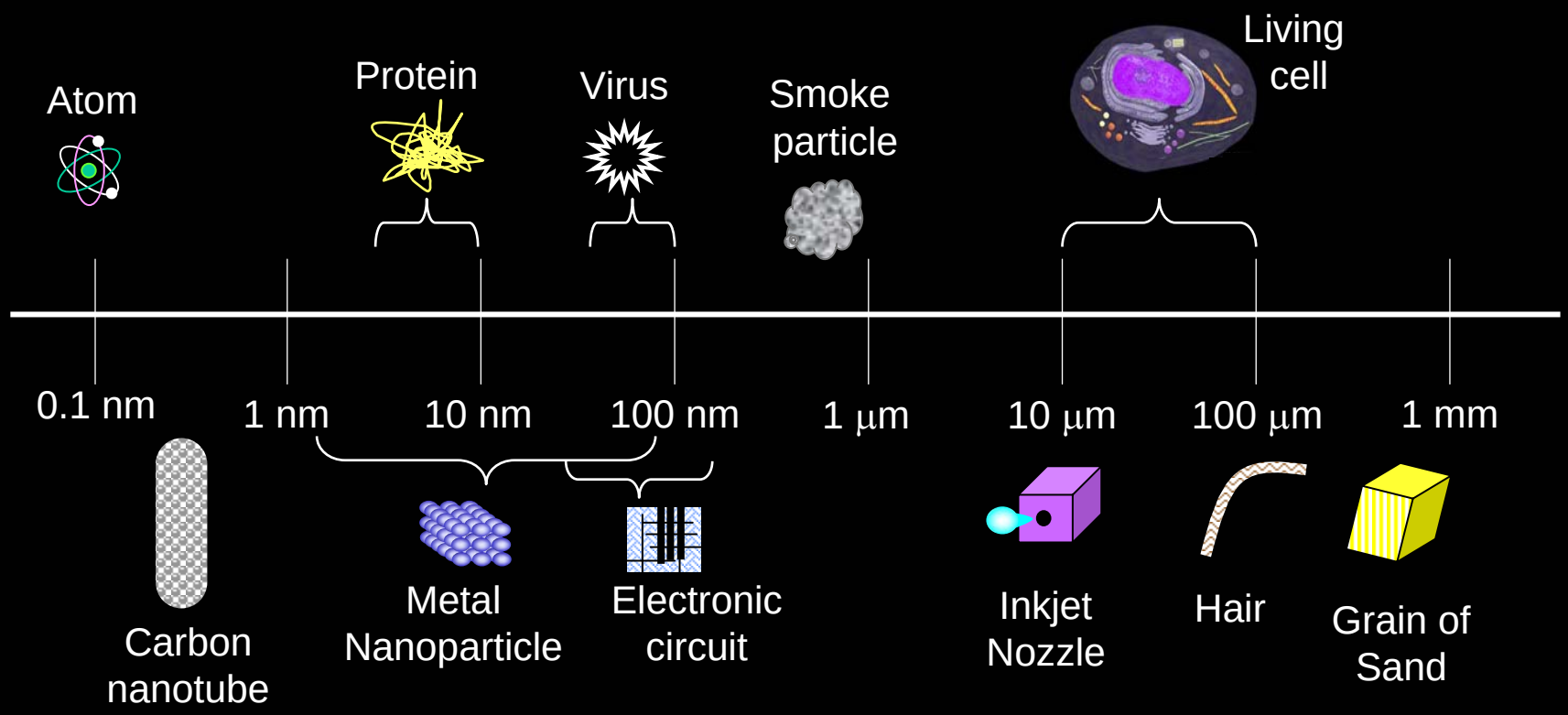
- £25m high-technology cluster development initiative
  - surface engineering (Northumbria)
  - chemical and biological sensors (Sunderland and Teeside)
  - molecular electronics (Durham)
  - biomedical nanotechnology (Newcastle)
  - International Centre for Life
- Director: Prof Ken Snowden

COMPANY PARTICIPANTS

ACORN



The scale of things



Building from the “bottom-up”