



Nano-related materials characterization and problem solving for industry

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Oxford Materials Characterisation Service

“... has been supporting industry and research for the last decade, providing unrivalled facilities for materials characterisation and consultancy....”

Begbroke Science Park

- ◆ Supporting Start ups
- ◆ Quality Control
- ◆ Contract analysis
- ◆ Problem Solving
- ◆ Industrial R&D
- ◆ Interdisciplinary Research Projects



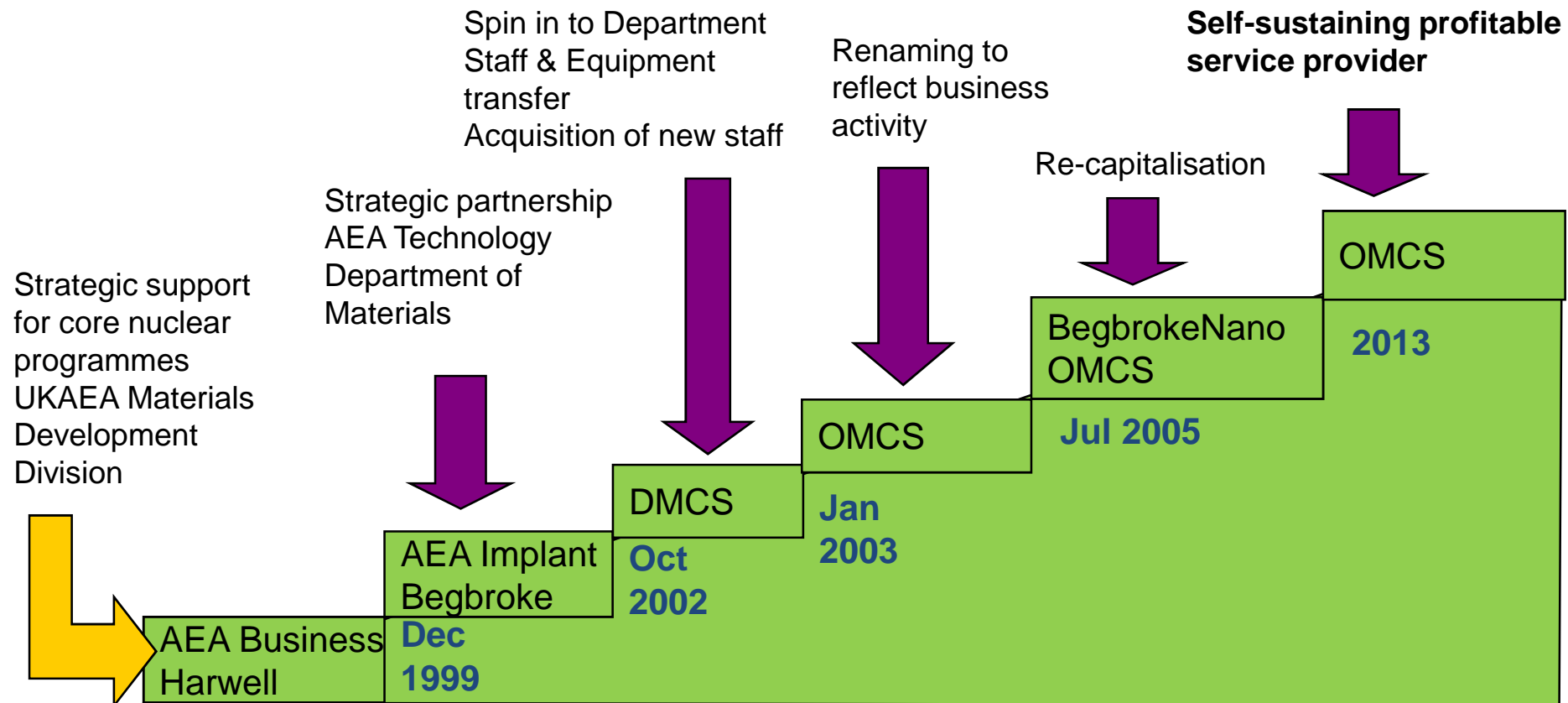
Fully funded by serving industry



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A brief history...

Lead by Dr Alison Crossley



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Service Delivery

WHAT WE DO...

- ◆ Analysis
- ◆ Consultancy
- ◆ Research and Development
 - In house
 - Support for companies / organisations
- ◆ Training
 - Industrial users
 - Students
- ◆ Project Partnership
- ◆ **Quality Assurance**
 - **ISO9001:2008**
 - **ISO17025:2005**
 - UKAS accredited- Glass**



HOW WE DO IT

- ◆ Diverse equipment base
 - Complemented by recent opportunistic investment
- ◆ Expert staff
 - including recruit training (by equipment manufacturers etc)
 - access to extra work force (experts and students)
 - staff encouraged to join outside committees
 - eg standards, RMS, IOP, RSC
- ◆ Outsource when required
 - Approved suppliers



Capabilities

MICROSCOPY

SEM & EPMA
TEM & STEM
Optical
Atom probe

SURFACE ANALYSIS

XPS
AFM
Profilometry
BET
NanoSIMS

BEGBROKE
SCIENCE PARK
WHERE SCIENCE AND INDUSTRY MEET



OTHER

XRD
XRF
Thermal Analysis, inc.
microcalorimetry
Nanomechanical testing
X-ray tomography

SPECTROSCOPY

FTIR
Raman
UV-Vis NIR

PARTICLE ANALYSIS

Dynamic Light Scattering
Differential Sedimentation
Laser diffraction
Nanotracking Analysis



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Typical nanomaterials characterisation requests

◆ Nanoparticles

- What is it?
- What is the particle size distribution?
- What is the surface like?
- Specific questions, e.g. surface area, pore volume, UV-vis absorption?

◆ 1 & 2D materials

- Is it what I think it is?
- How many layers or size is it?
- What else can you tell me about it, e.g. defects?

◆ Embedded nanomaterials

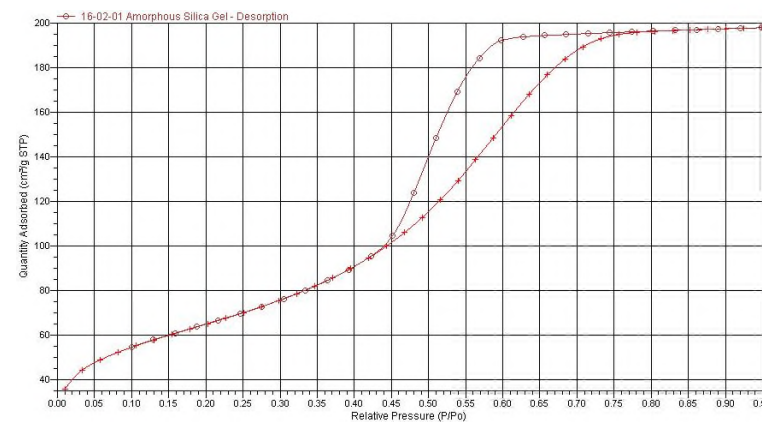
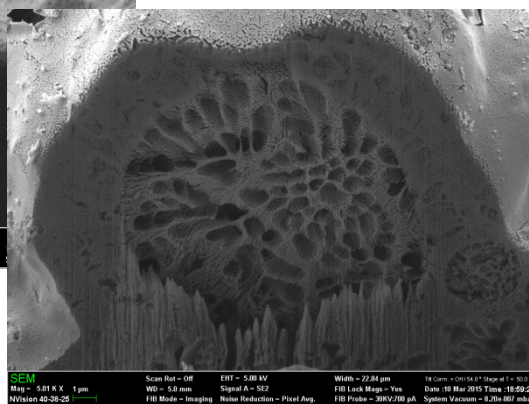
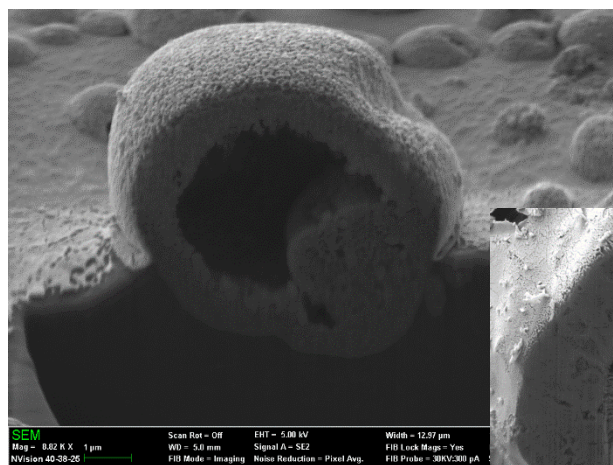
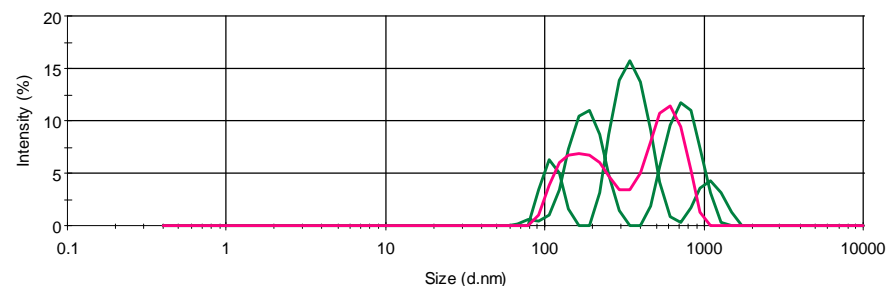
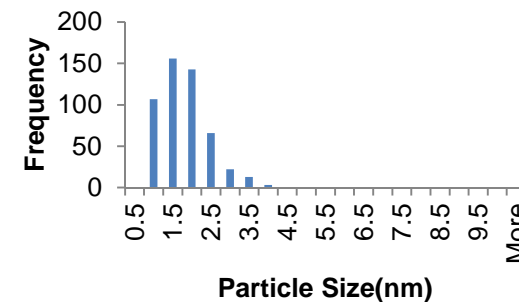
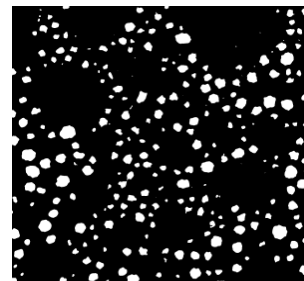
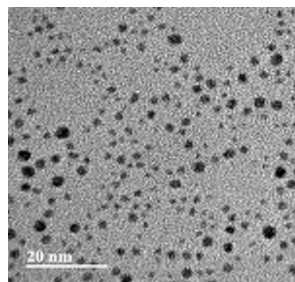
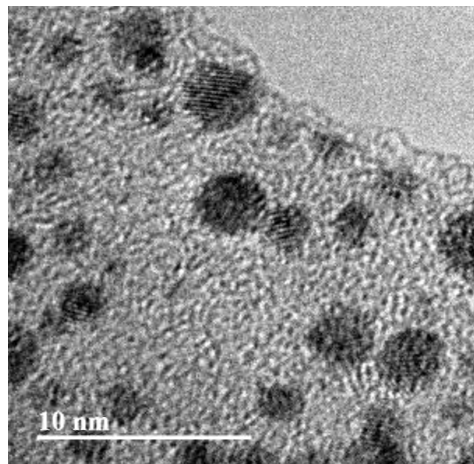
- What is it, where is it and how much of it is there?

◆ Thin films

- Structure and composition?



Nanoparticles



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Graphene powder

◆ Original questions

- Is it graphene?
- How many layers?

◆ Solution

- Raman spectroscopy

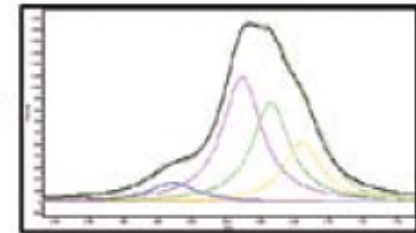


From Horiba JY application note RA50

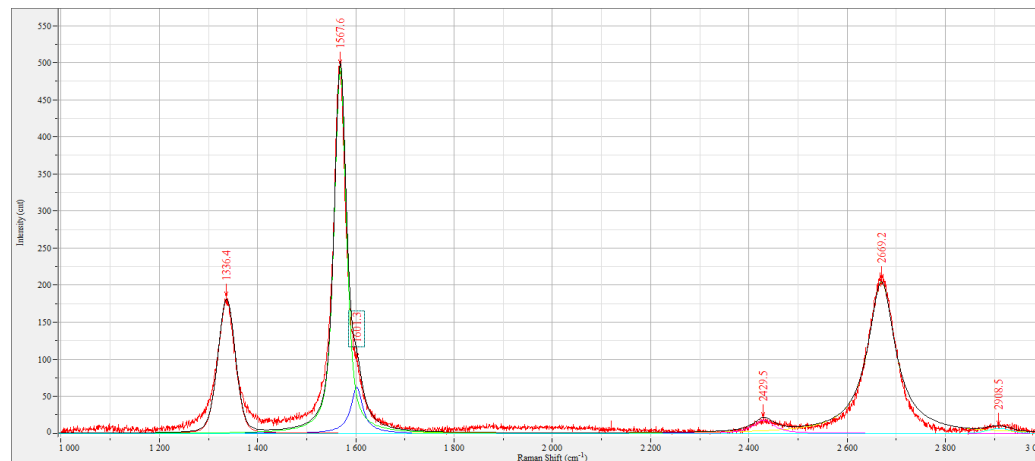
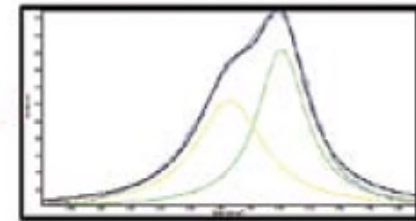
Single layer



Bilayer



Graphite



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Mission creep

- ◆ The customer was hoping to get information about the defect density
 - They had read a paper
- ◆ Defect density
 - Follows on from original work on graphite crystallite size correlated to $I(D)/I(G)$ [Tuinstra and Koenig]
 - D (and D') peak increases with number of defects and edges
 - Excitation energy dependence
 - Theory developed from studies on carefully controlled oriented flakes
- ◆ Real problem here was that the samples were powders
 - Uncontrolled volume sampled
 - Uncontrolled number of edges presented to beam
 - How was the customer going to use the numbers?

Carbide nanoparticles

◆ The problem

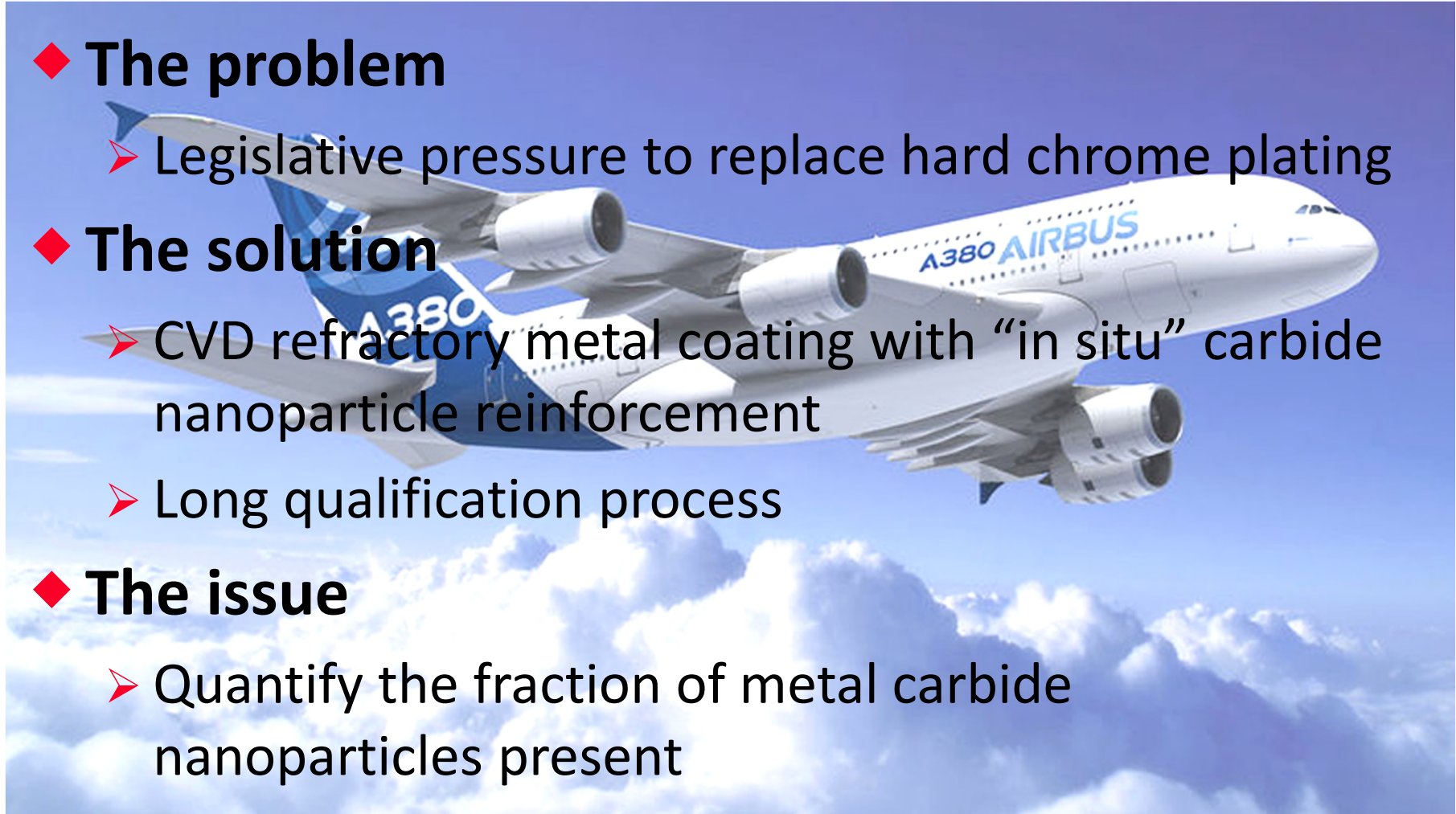
- Legislative pressure to replace hard chrome plating

◆ The solution

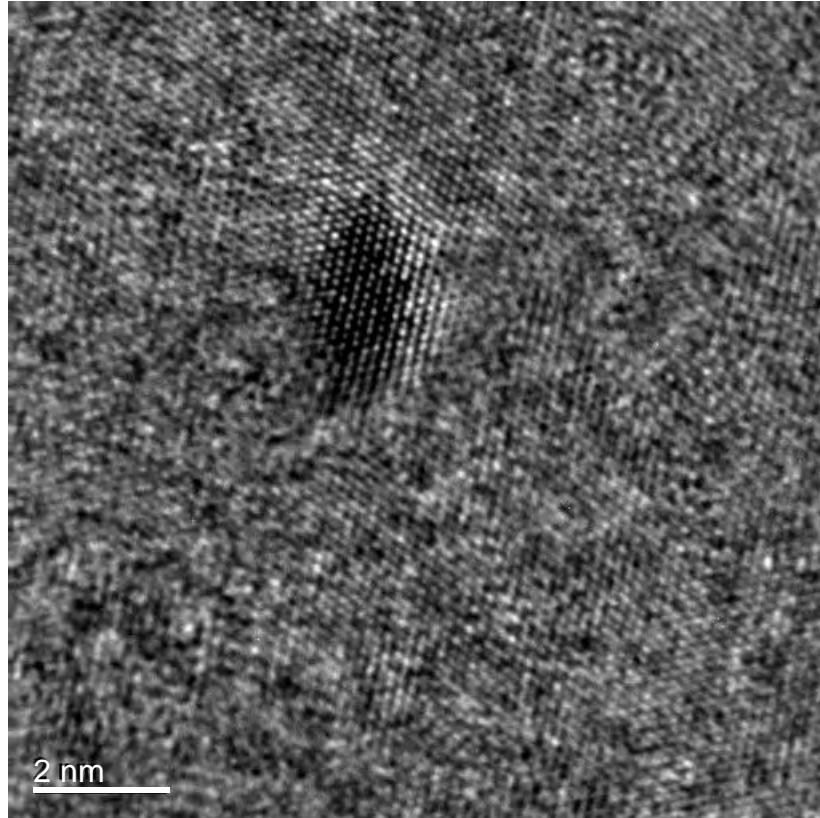
- CVD refractory metal coating with “in situ” carbide nanoparticle reinforcement
- Long qualification process

◆ The issue

- Quantify the fraction of metal carbide nanoparticles present



2 nm precipitated carbide particle

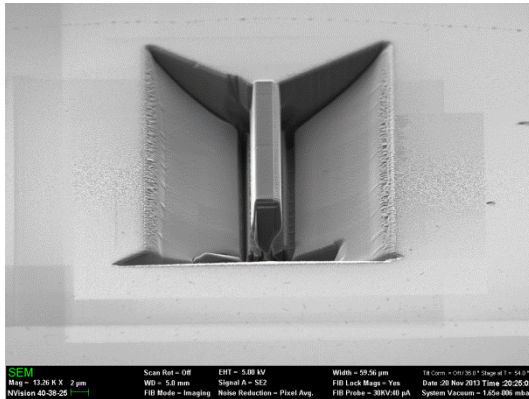


- ◆ High resolution TEM
- ◆ FIB – 10 x 2 μm x-section
- ◆ Questions
 - How representative is that?
 - What is the volume fraction?
 - How many fields of view would be needed?

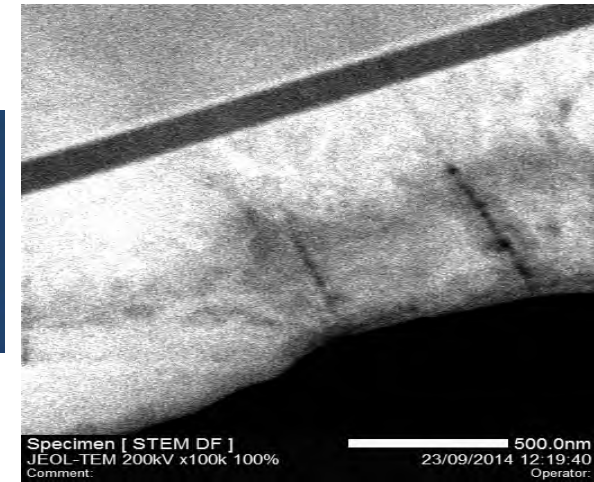
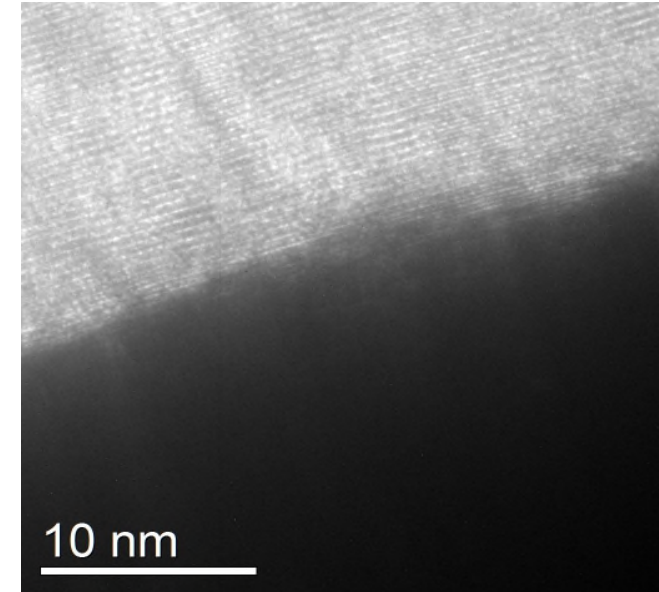
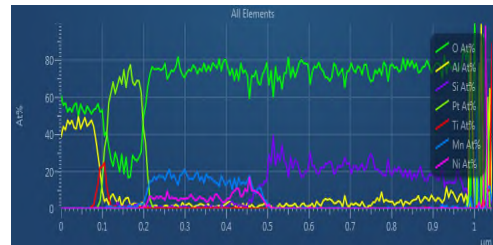
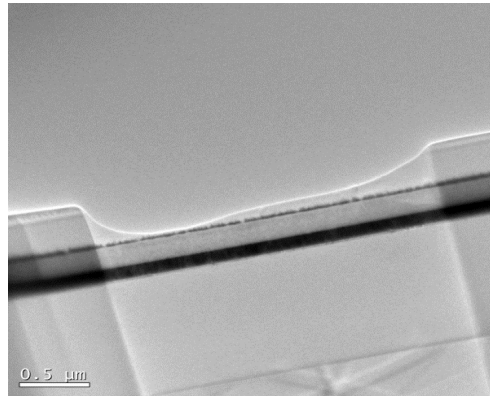
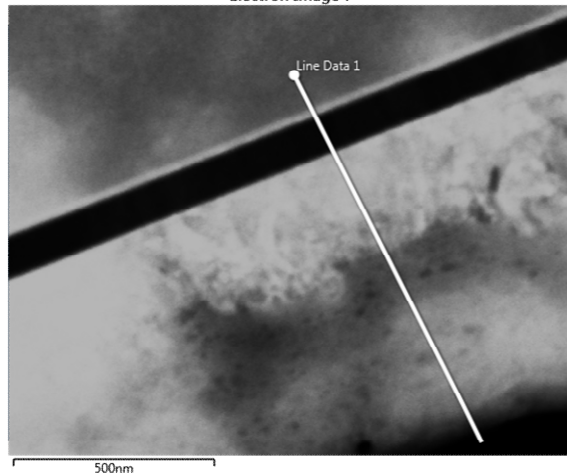
50,000 images required

Typical thin film work

- ◆ Tends to be knowledgeable customer
- ◆ Highly specific requests



Electron Image 7



Advanced techniques

- ◆ Atom probe facility
 - 3 instruments
- ◆ UK National centre

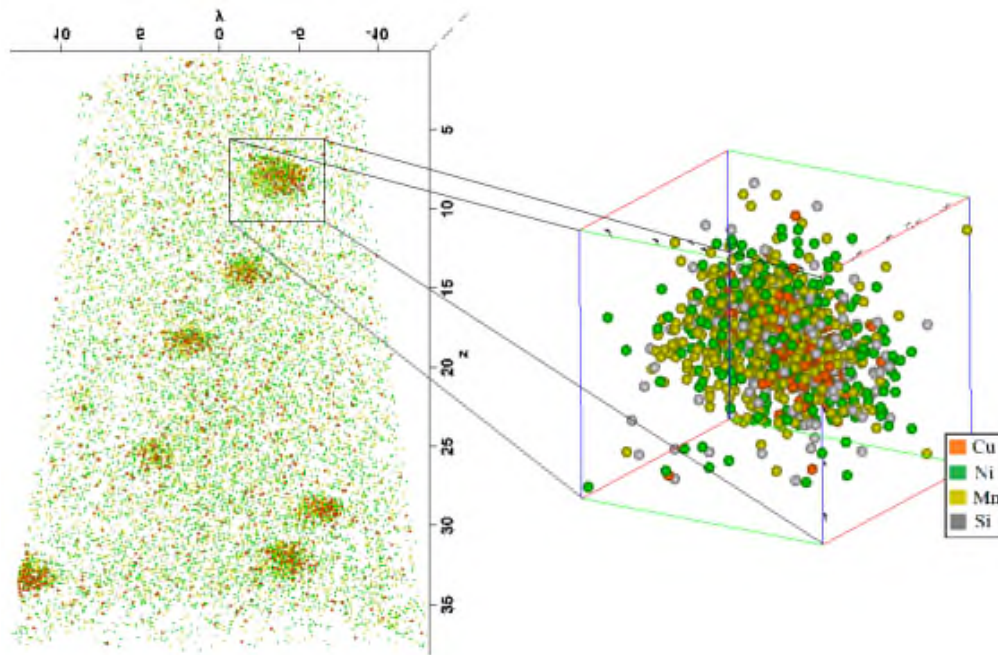


- ◆ Aberration corrected TEM
 - JEOL strategic partner
- ◆ electron Physical Science Imaging Centre (ePSIC)



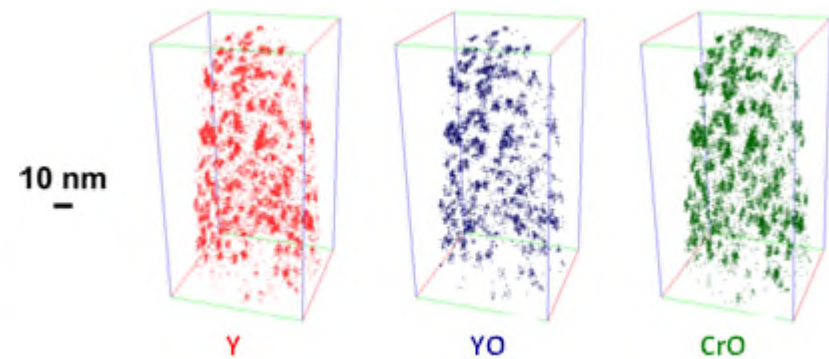
Steels

Embrittlement



AP map of a high copper RPV steel with copper enriched precipitates – highlighting the role of Ni & Mn

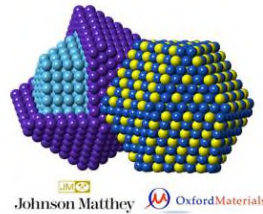
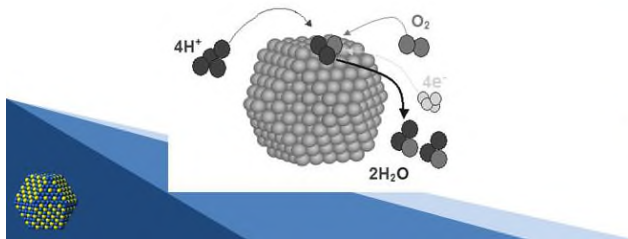
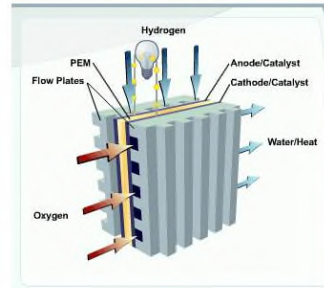
Strengthening



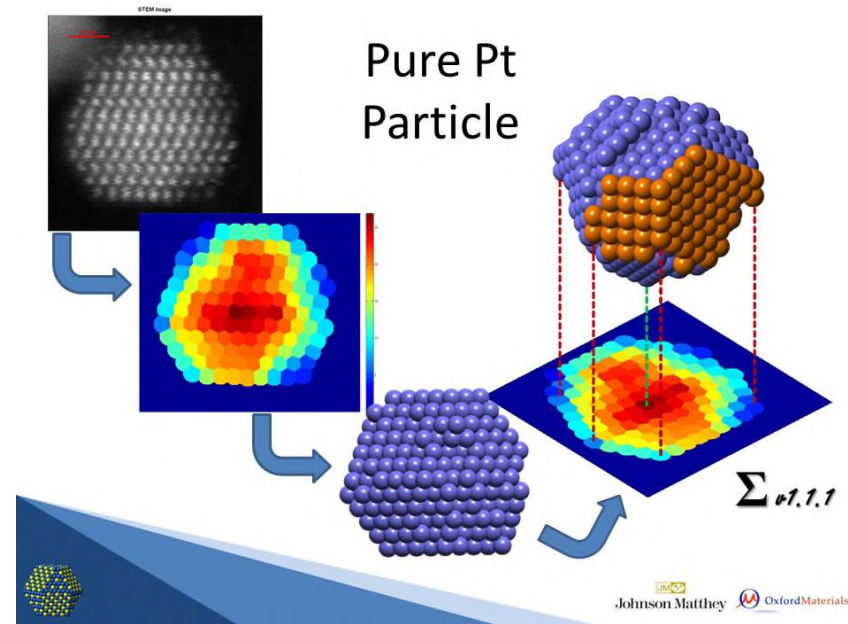
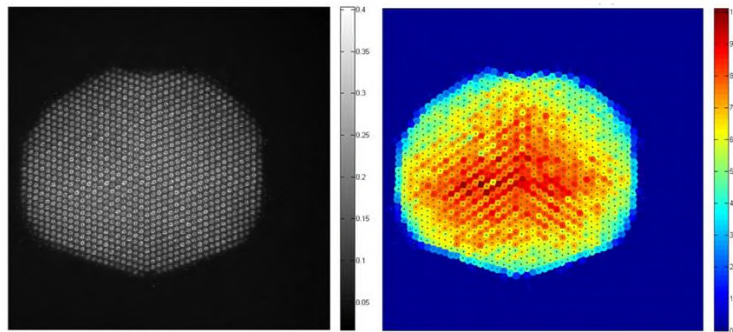
Nanoparticle dispersion in a series of Oxide Dispersion Strengthened (ODS) Fe-14Cr alloys

Catalysts

- Polymer Exchange Membrane Fuel Cells show a great deal of promise as a future energy solution.
- Pt Catalyst needed for the oxygen reduction is very expensive!



Pt-Co Particle

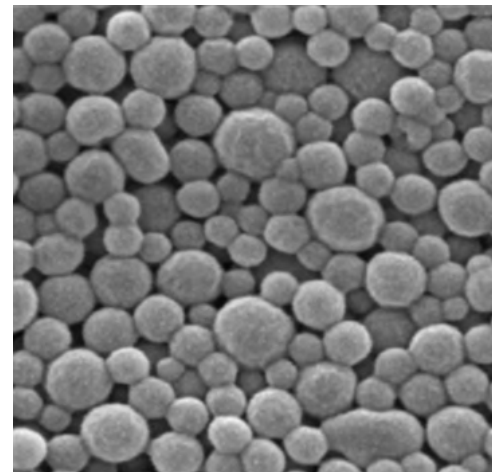
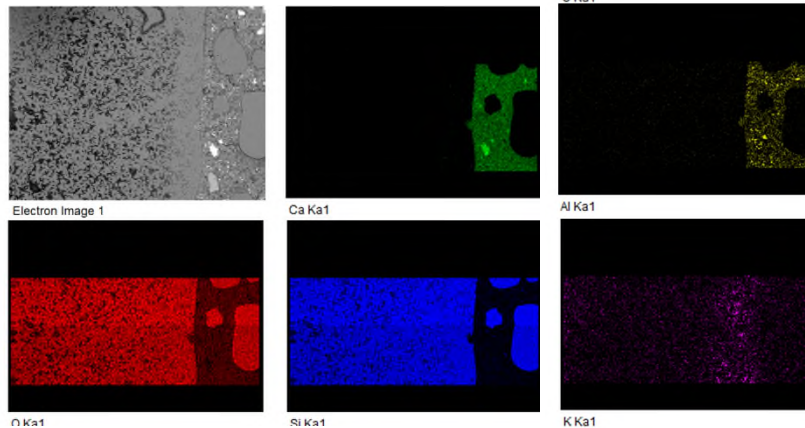


Electron beam
tomography

Concrete



- ◆ Bulk use of nano(fumed) silica
- ◆ Low concentrations (1 – 5wt %)
- ◆ Increase strength and durability
- ◆ It reacts Calcium Silicate Hydrate
- ◆ No longer nano?



Summary

- ◆ Need a suite of complementary tools/techniques
 - Not a one fits all
 - Need to appreciate the limitations
- ◆ Need detailed knowledge
 - Don't over interpretation
- ◆ Manage expectations
 - Realistic targets
 - A little knowledge can be dangerous!



Questions?



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