



# Nano-related materials characterization and problem solving for industry

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# Oxford Materials Characterisation Service

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*“... 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



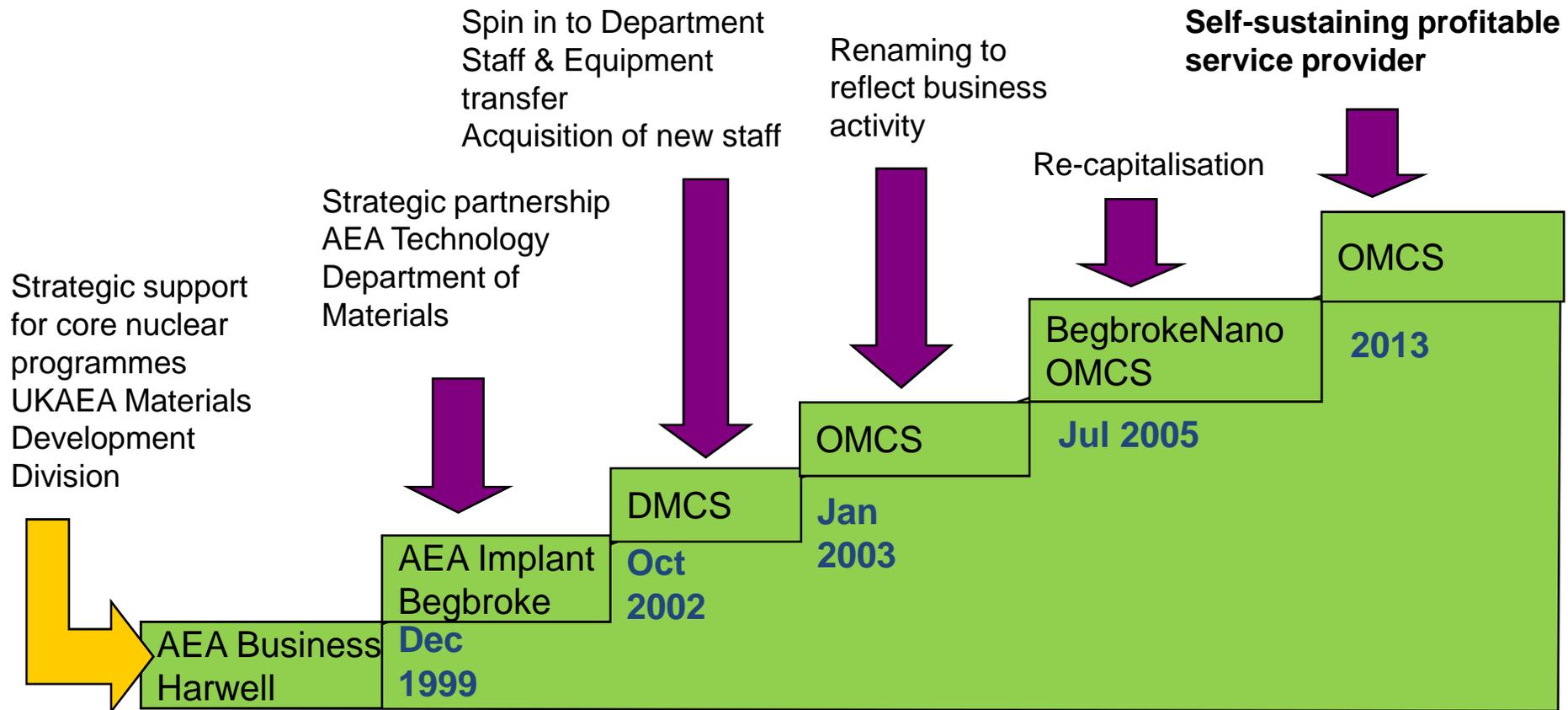
***Fully funded by serving industry***



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# A brief history...

*Lead by Dr Alison Crossley*



# Service Delivery

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## *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



## SPECTROSCOPY

FTIR  
Raman  
UV-Vis NIR

## PARTICLE ANALYSIS

Dynamic Light Scattering  
Differential Sedimentation  
Laser diffraction  
Nanotracking Analysis

## OTHER

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



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# Typical nanomaterials characterisation requests

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## ◆ 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

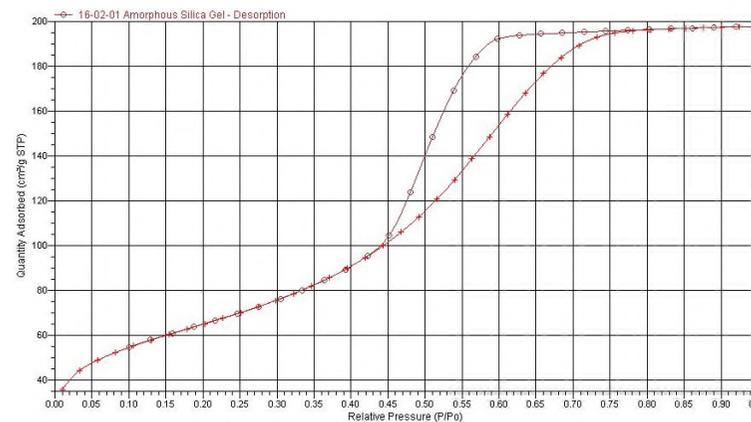
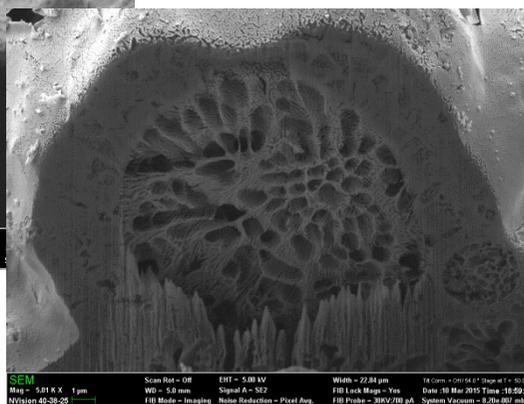
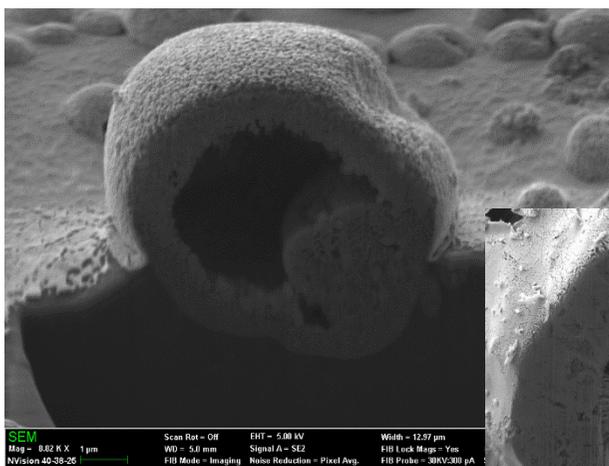
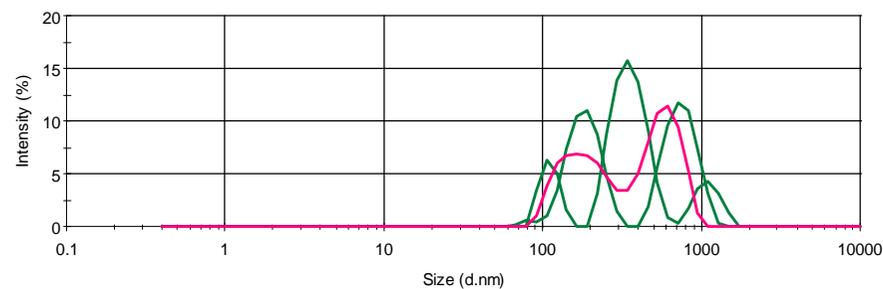
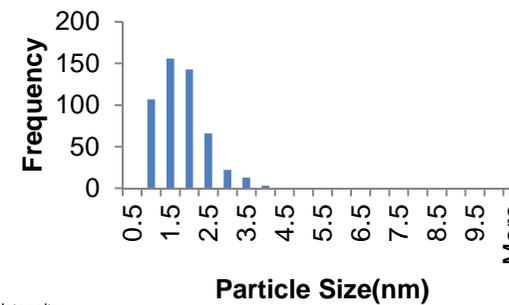
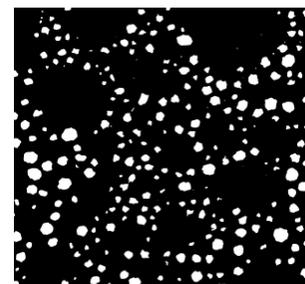
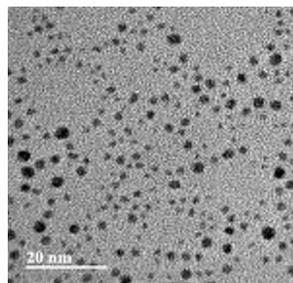
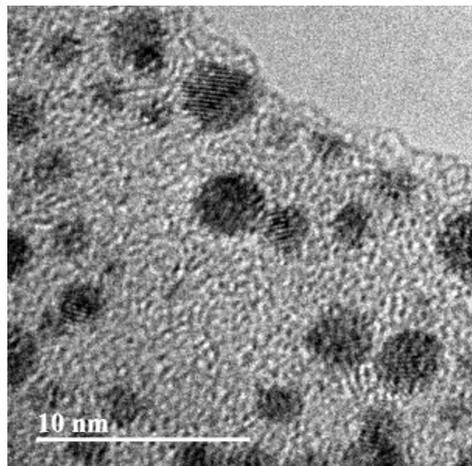
- What is it, where is it and how much of it is there?

## ◆ Thin films

- Structure and composition?



# Nanoparticles



# Graphene powder

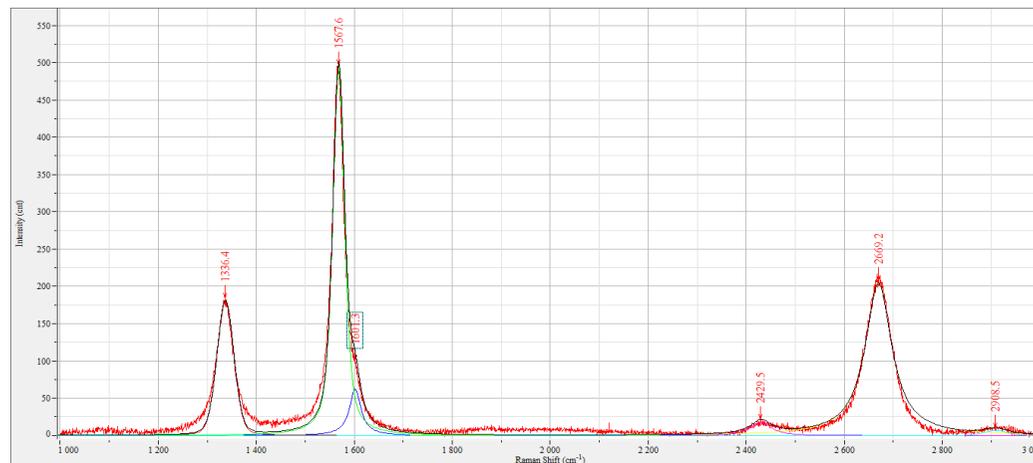
## ◆ Original questions

- Is it graphene?
- How many layers?



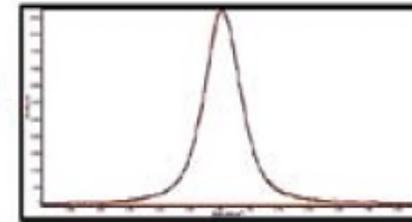
## ◆ Solution

- Raman spectroscopy

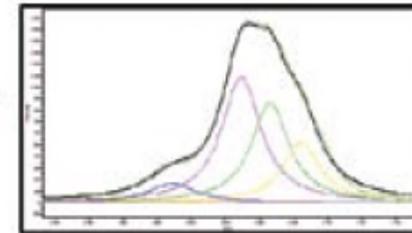


From Horiba JY application note RA50

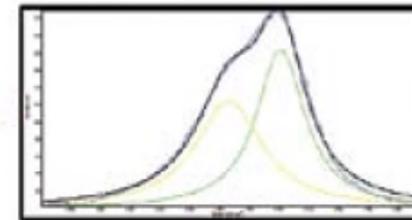
Single layer



Bilayer



Graphite



# Mission creep

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- ◆ 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

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## ◆ The problem

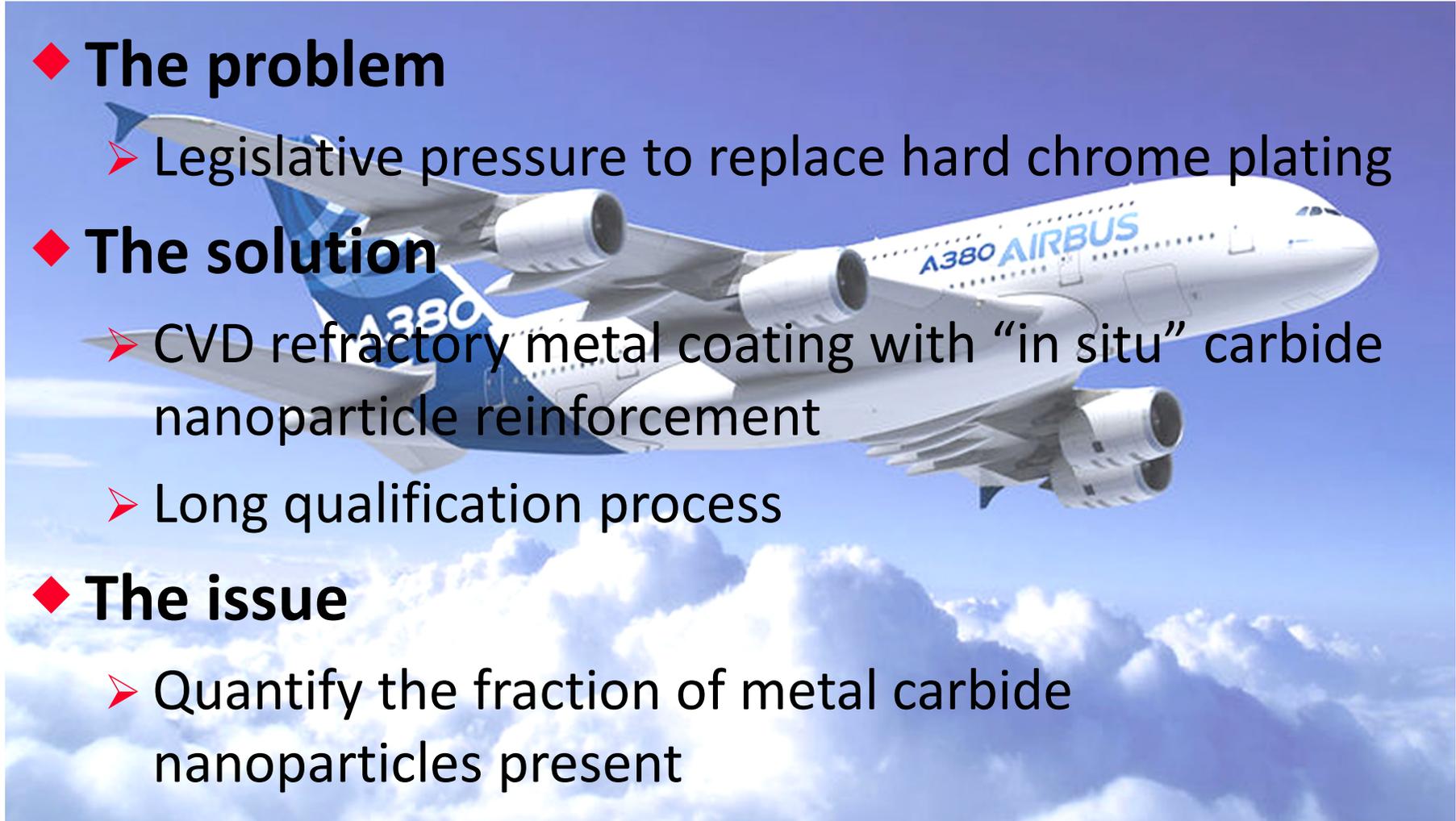
- Legislative pressure to replace hard chrome plating

## ◆ The solution

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

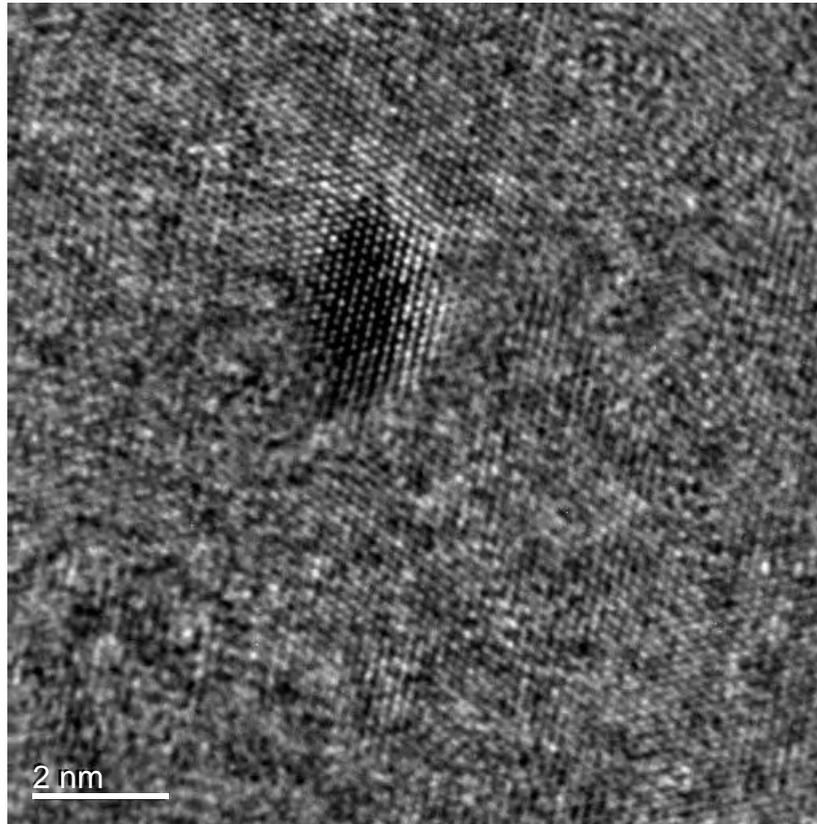
## ◆ The issue

- Quantify the fraction of metal carbide nanoparticles present



# 2 nm precipitated carbide particle

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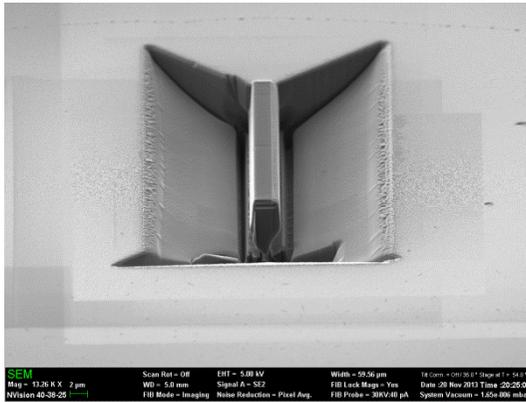


- ◆ High resolution TEM
- ◆ FIB – 10 x 2 um x-section
- ◆ Questions
  - How representative is that?
  - What is the volume fraction?
  - How many fields of view would be needed?

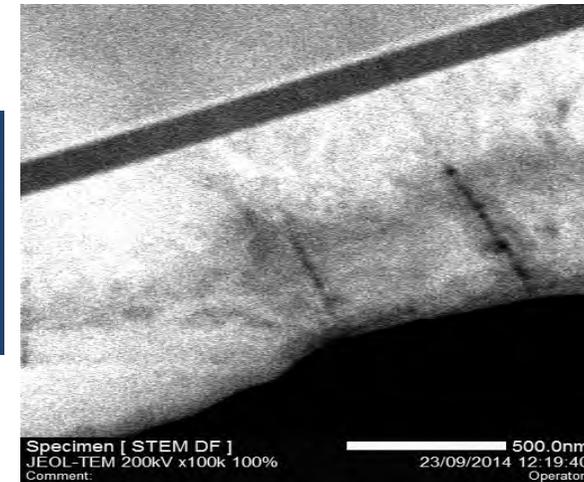
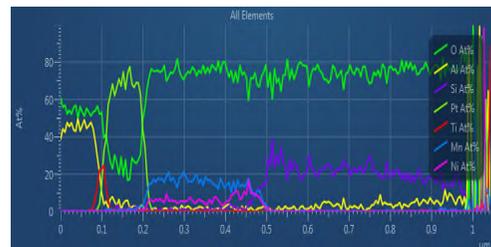
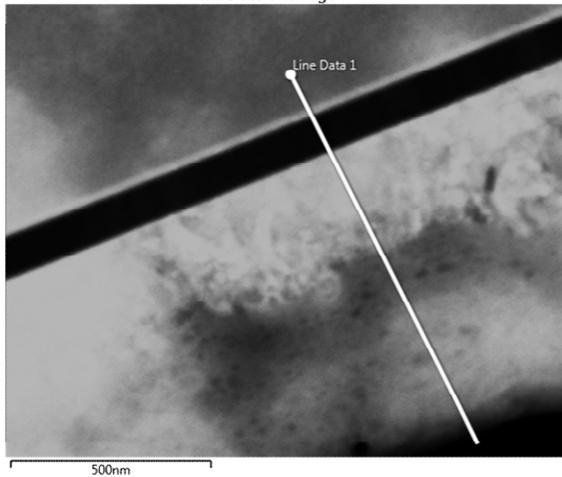
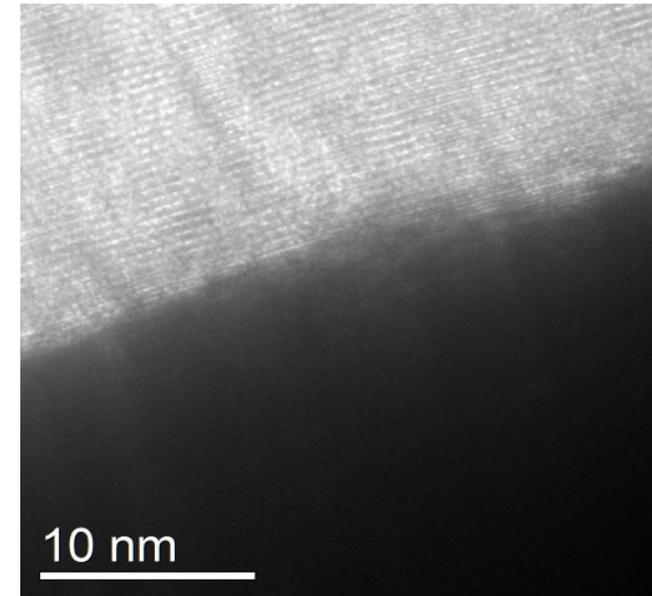
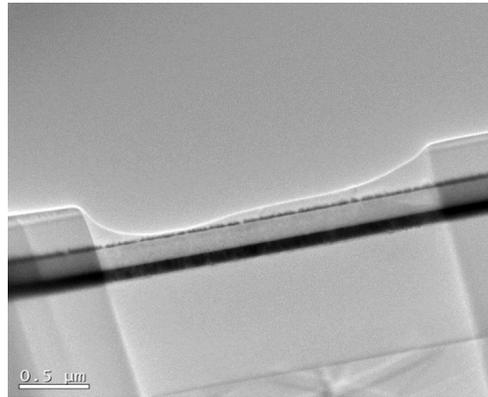
***50,000 images required***

# Typical thin film work

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



Electron Image 7



# Advanced techniques

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

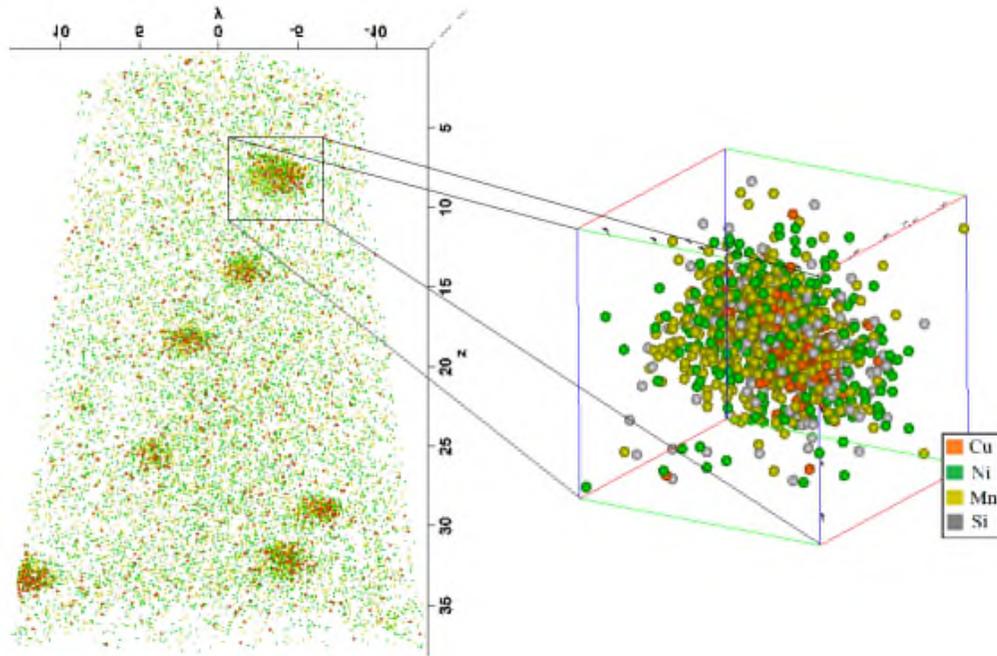


- ◆ Aberration corrected TEM
  - JEOL strategic partner
- ◆ electron Physical Science Imaging Centre (ePSIC)



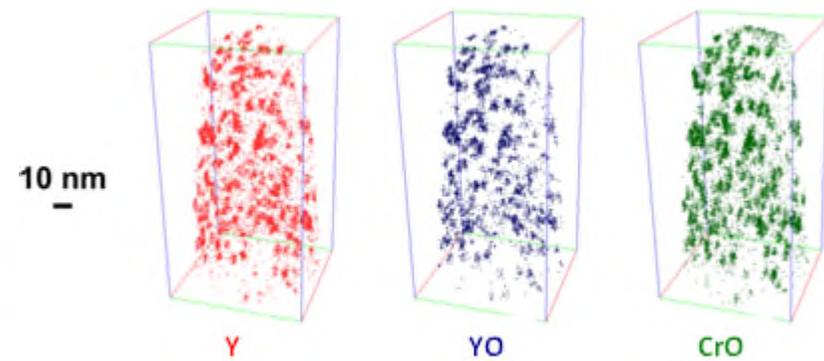
# Steels

## Embrittlement



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

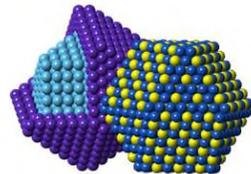
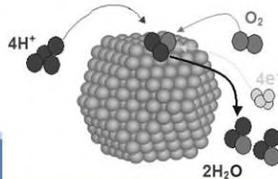
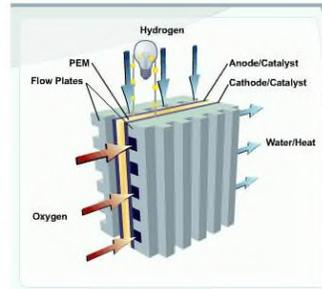
## Strengthening



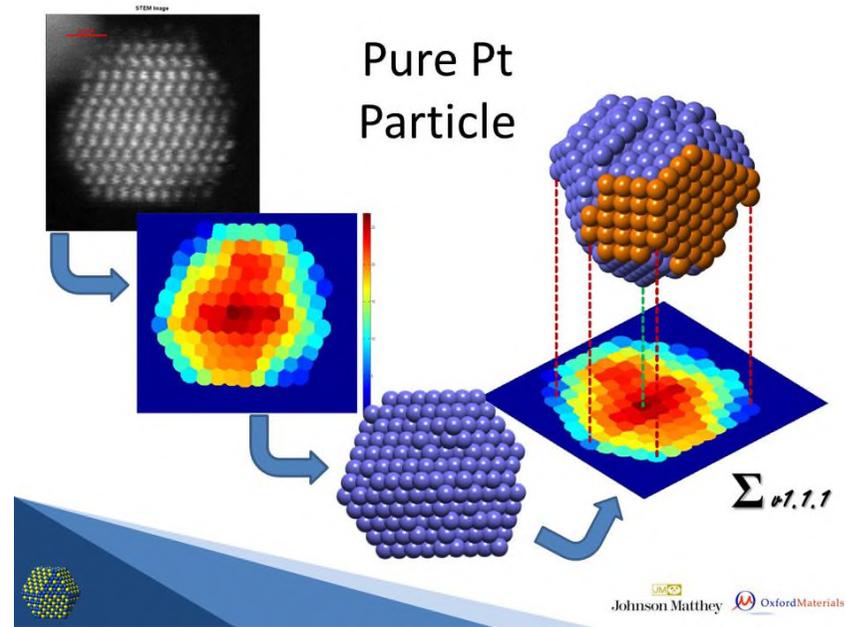
Nanoparticle dispersion in a series of Oxide Dispersion Strengthened (ODS) Fe-14Cr alloys

# Catalysts

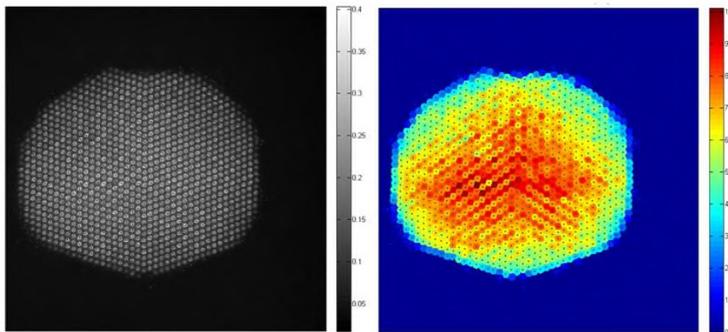
- Polymer Exchange Membrane Fuel Cells show a great deal of promise as a future energy solution.
- Pt Catalyst needed for the oxygen reduction is very expensive!



Johnson Matthey Oxford Materials



Pt-Co Particle

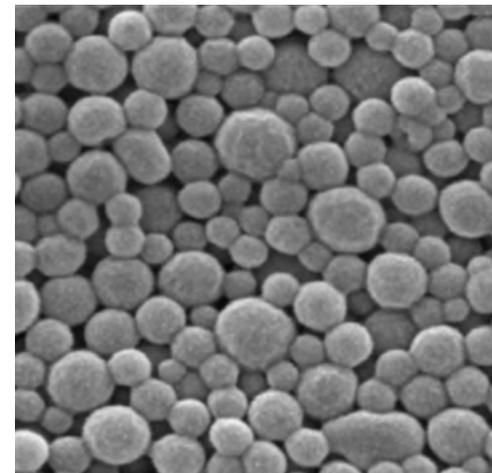
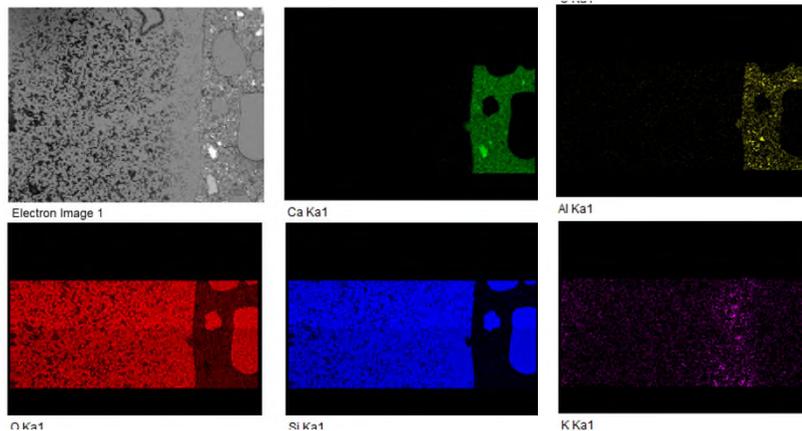


## Electron beam tomography

# Concrete



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



# Summary

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- ◆ 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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