Bringing Advanced Characterisation to Industry

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About us

- Finden is a UK Ltd company
- Operating for 1 year
- 4 unique clients
- Consists of two academics
  - Extensive synchrotron experience
  - X-ray & spectroscopic methods for materials characterisation
  - High throughput methods
  - Crystallography & Chemistry
- As academics we are based at RCaH Diamond. Company located in Oxfordshire, UK

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Dr Andrew Beale

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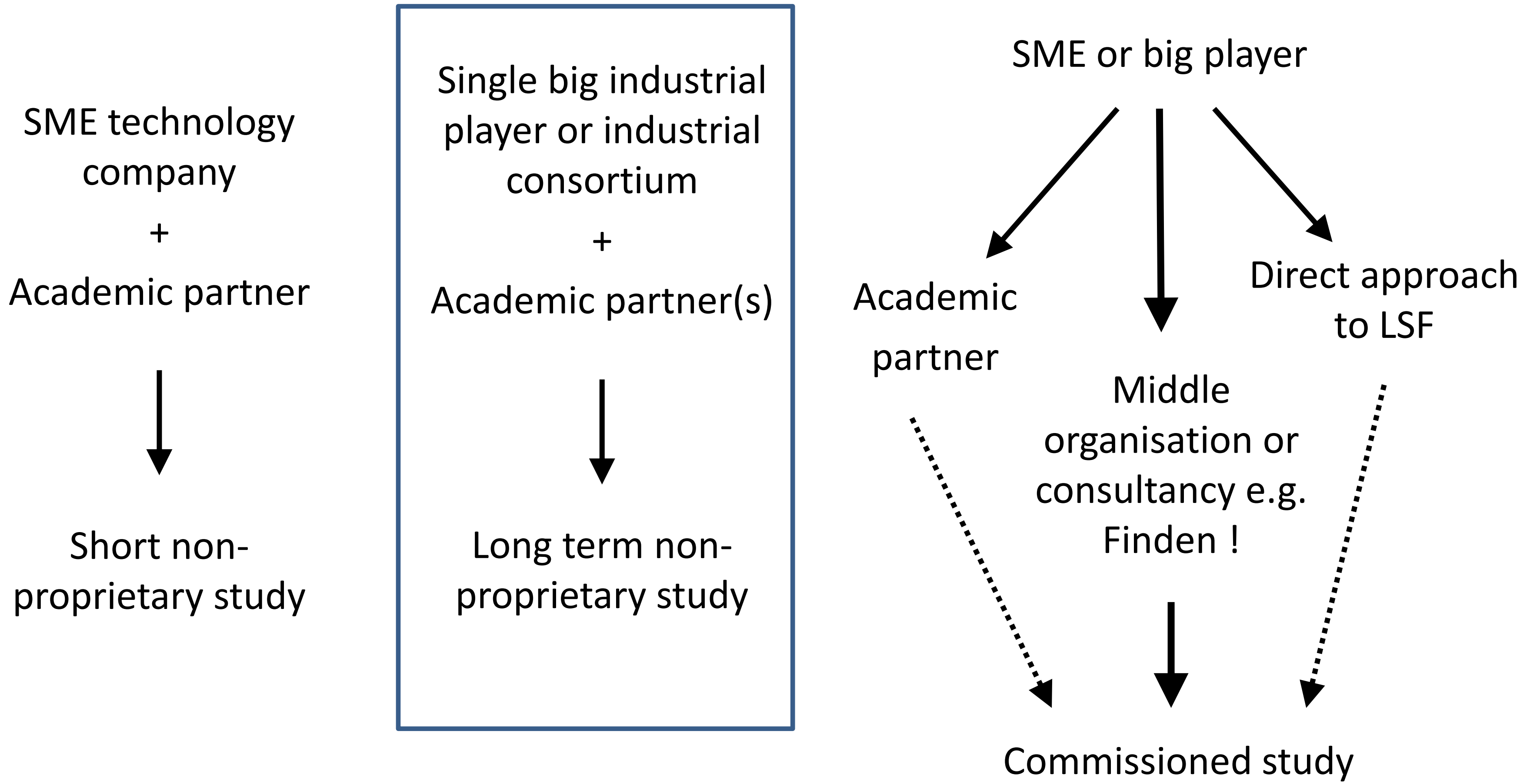
Access to LSF



Project Construct

2 year project – 1 Post Doc ($200 k)

Proposed stationing partners (infrastructure)

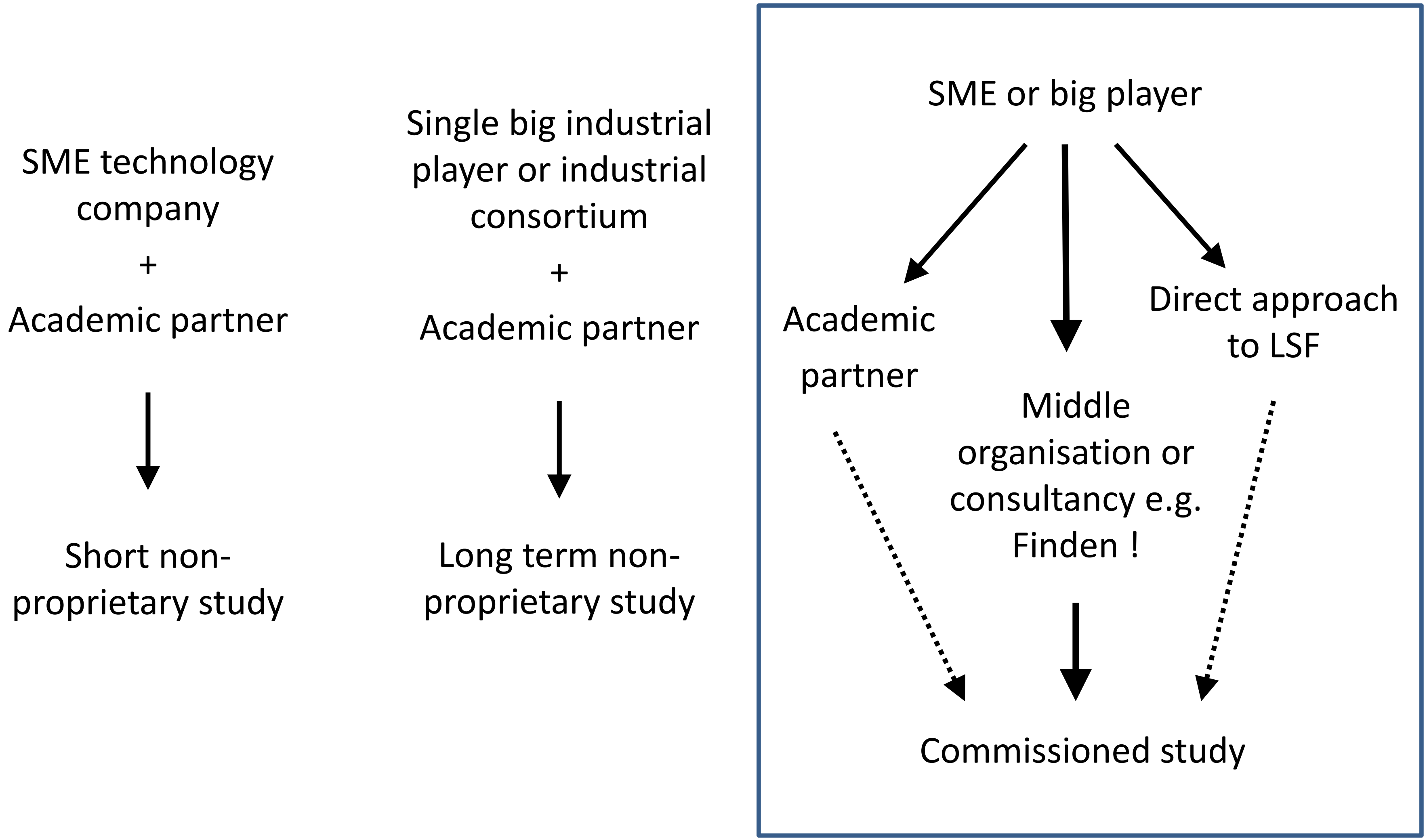
Non proprietary not without its issues or significant costs !

HE institution – overheads (typically > 25 %) in northern Europe

IP – ownership/publication clearance times

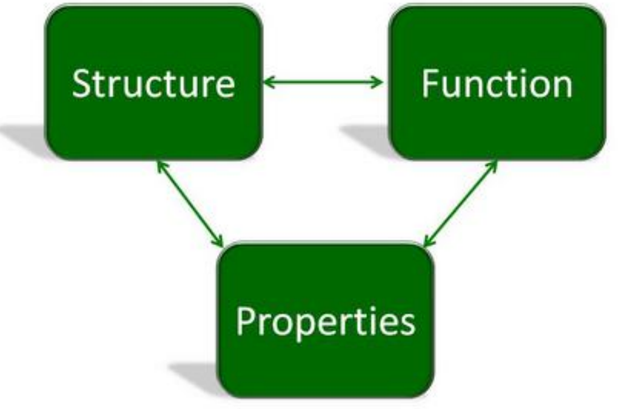
Recruitment – satisfying requirements of project partners

Access to LSF



About Finden

- As Finden Ltd, we aim to provide an efficient alternative
- Standard & advanced characterisation to further understanding of structure-function-property
- Chemical analysis under both static and in real time under operating conditions
- Services
  - X-ray absorption spectroscopy (XANES EXAFS)
  - X-ray diffraction and scattering
  - Infra Red Spectroscopy
  - Raman Spectroscopy
  - UV-Vis Spectroscopy
  - Tomography
  - Diffraction tomography & other advanced methods
- Consultancy on
  - data processing
  - process/materials development



What we do and who we serve ?

- Largely focus on industrial catalytic problems (associated with fine chemical synthesis)
- Nature of catalysts is that they are specialist materials – expensive raw ingredients, often expensive to manufacture
- Improve lifetime, but also inline re-activation (initial vs. permanent deactivation)
  - Ex-situ analysis often misleading (passivation issues – sintering etc.)
  - Classic in-situ is not always representative
- New technology companies (SME) / established big players
- Understand the requirements and the goals
- Help industry:
  - Pose questions
  - Choose characterisation methods
  - Design & perform experiments
  - Analyse and interpret data
  - Advise on how to improve products / processes

Our perception of industry barriers & motives

Barriers

- Industry traditionally hesitant to using large scale facilities (especially direct access)
- ‘High’ cost often quoted as an impediment to commitment
- Real cost is risk of not getting results and getting them in a reliable time frame
- Others !

Motivations

- General shift in focus – requirement to develop efficient processes (cost of raw materials; cost of preparing materials; environmental impact of preparation and process; recycling considerations); exploiting new opportunities
- Coupling of materials to make devices – understanding their function

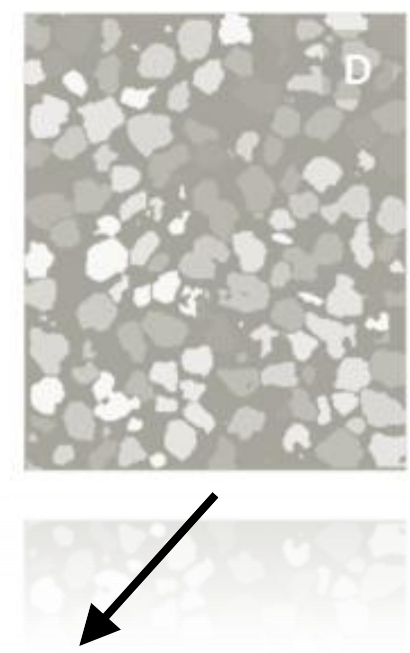
Our perception of industry barriers & motives

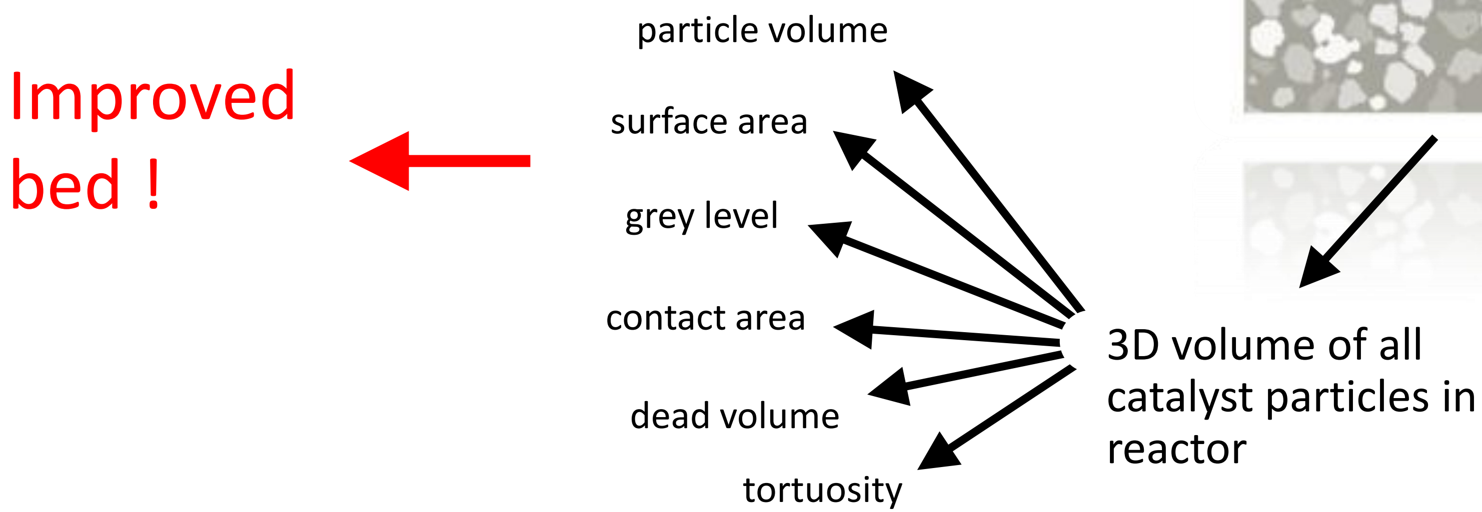
Bias against using LSF largely a misnomer since:

- Asking the right question ? (LSF black box)
- Do expts not achieveable by conventional measurements
- In situ – becomes highly relevant
- Tried and tested methods argument – i.e. those benchmarked (not worse than lab !)
- High flux / recording allows large volume of work to be completed
- On-the-fly results processed data into recognisable format (what you get in the lab) allow adaption
- Ways to mitigate risk – free or low cost proof-of-concept studies
- Rapid access ?

Example of traditional characterisation

- Industrial partner wants to investigate the poor performance of a reactor (prescriptive)
- Whole field reactor (3 x 2.5 x 30 cm casing) studied using lab based CT (6 & 20µm resolution)
- First carry out small proof-of-concept study





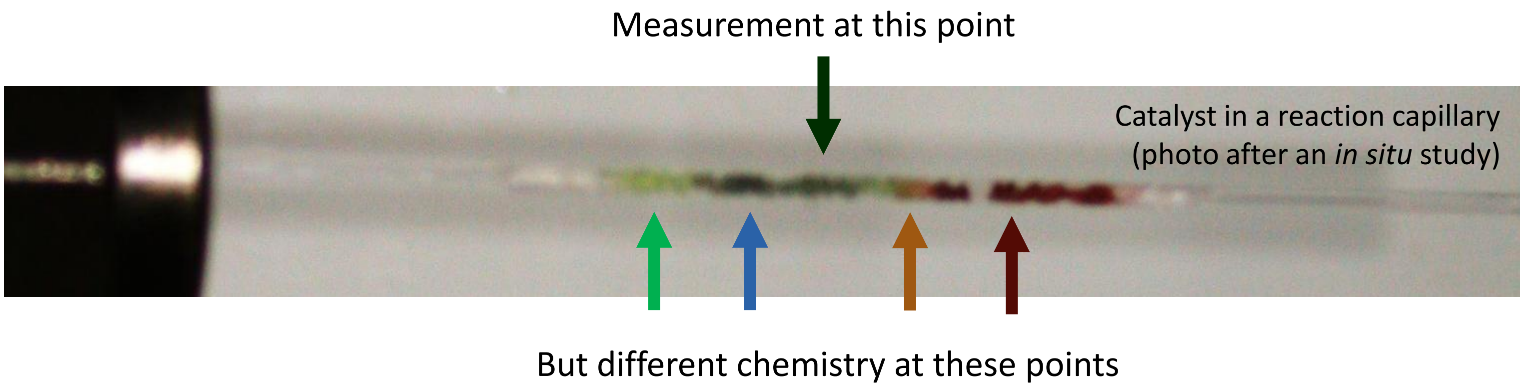
SR better route

- Ca. €7000 study (Whole sample characterisation i.e. post proof-of-concept)
- Including 10 hours scan time @ ca. €2700
- 8 hour SR shift ca. €3300
- Easily 5 x samples
- SR → higher quality results (better contrast; higher resolution)
- Measurement of choice – combine with other measurements
- Raw data quicker (virtue of centralised computing cluster)

Advanced characterisation: Moving from packed bed models to real systems

- Shifting to real samples
- Accepting samples are heterogeneous by accident or design
- Recognising limits of techniques

The packed bed model



- Miniature of industrial process
- Use gas delivery / heating apparatus to model process conditions
- Convenient for characterisation by methods sensitive to chemistry (e.g. in situ XRD)

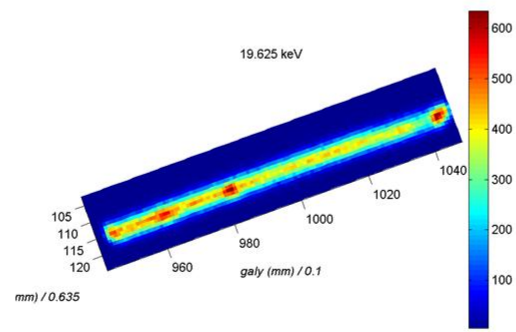
- Real catalysts are rarely crushed and sieved powder fractions !
- Single point studies do not tell us the whole story
    - true on small and large scale (μm → m)
    - true in both the bed and indeed catalyst bodies

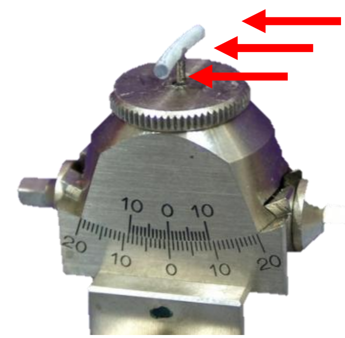
reactant(s)

product(s)

Using real samples

- Supported catalysts – preferred industrial method is to use packed bed structures of catalytic bodies
- mm or µm sized objects
- High energy X-rays can penetrate thick objects
- Efficiency of entire industrial process dependent on the performance *per se*



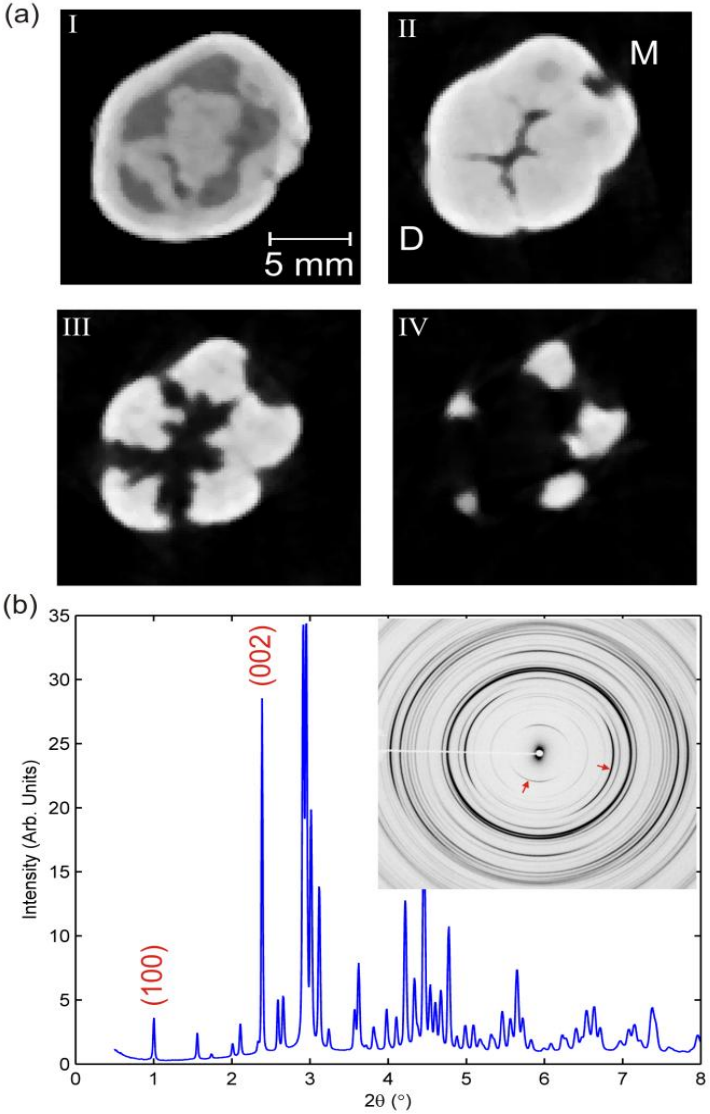


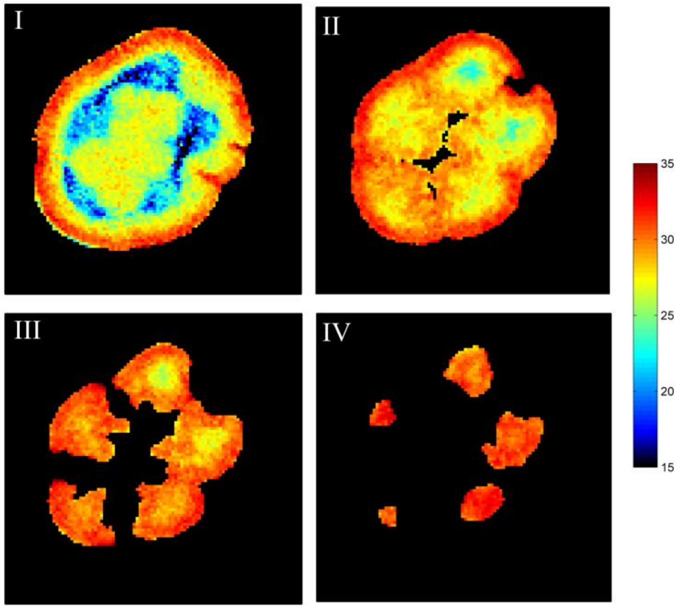
Tomography shows us that there are variations inside the catalytic body; Co/Mo on alumina

Support materials provide gas permeable environments with high SA

Advanced characterisation

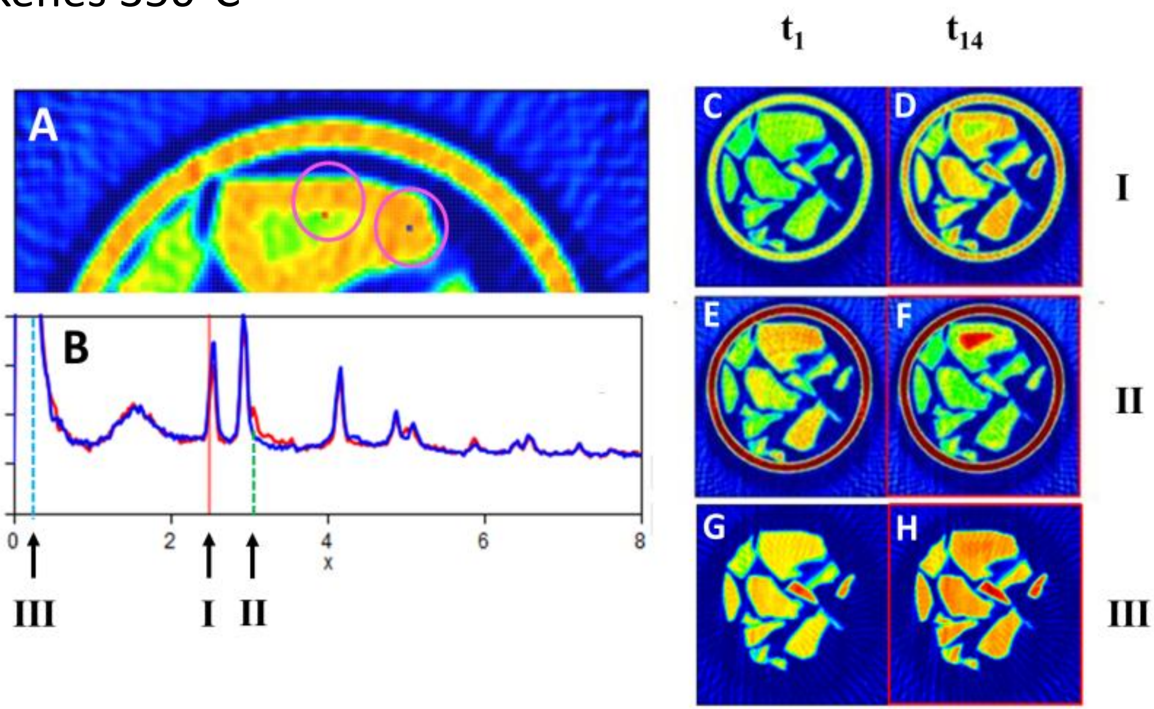
- In situ XRD-CT using high flux high energy X-rays (synchrotron technique)
- Combination of X-ray tomography and XRD
- Powerful non-destructive technique gives XRD signal at each point in **bulk samples**
- Any extractable feature in XRD can be mapped
- Open to conventional analysis





Crystallite size

- **Map evolving chemistry**
- Can discriminate sample from in-situ rig or container
- Industrial catalyst
- Calcination → activation → FT at atmospheric pressure
- Short chain alkanes/alkenes 350°C
- 5 µm resolution



In operando study of industrial FT catalyst using PDF-CT.
A raw intensity components of the reconstructed diffraction for this slice. B shows two reconstructed patterns blue and red spectra from the pixel positions circled in A. The maps C-H are maps of raw diffraction intensity at positions I, II and III in the diffraction pattern; where I and II correspond to positions associated with Bragg peaks for CoO and fcc Co respectively. Position III corresponds to a SAX signal which perhaps can be rationalised with PDF later. C,E & G and D,F & H show these components at times t_1 and t_{14} respectively. These images illustrate that spatial changes in time can readily be observed. .

Our experience

- *Advanced* characterisation is logical progression
- Fairly similar to standard methods
- Easy to convince Industrial partners / clients of the value of such characterisation methods once engaged
- Proof-co-concept is convincing step
- They are <u>not</u> demanding accreditation or benchmarking
- They look to us pre and post-study as to the value of data
- Timely that such characterisations are readily available

Our bottlenecks / issues

- Lag. From initial contact to commissioned study
- Lag. From 1st work to next commision
- Rapid requirements. Results next week !
- Heightened awareness of obtaining the data
- Technical challenges dealing with large data sets

Papers

Pair Distribution Function Computed Tomography
Simon. D. M. Jacques et al.
Nature Communications 4 (2013)

Dynamic X-Ray Diffraction Computed Tomography Reveals Real-Time Insight into Catalyst Active Phase Evolution
Simon. D. M. Jacques et al.
Angewandte Chemie International Edition **50** (43) p10148 (2011)

Probing ZnAPO-34 Self-Assembly Using Simultaneous Multiple in Situ Techniques
Andrew M. Beale et al.
The Journal of Physical Chemistry C **115**(14) p6331 (2011)

Active phase evolution in single Ni/Al2O3 methanation catalyst bodies studied in real time using combined μ-XRD-CT and μ-absorption-CT
Matthew. G. O'Brien et al.
Chemical Science **3**(2) p509 (2012)

Noninvasive Spatiotemporal Profiling of the Processes of Impregnation and Drying within Mo/Al2O3 Catalyst Bodies by a Combination of X-ray Absorption Tomography and Diagonal Offset Raman Spectroscopy
Emma K. Gibson et al.
ACS Catalysis **3** (3) p339 (2013)