

**MAPP**

Manufacture using Advanced  
Powder Processes  
EPSRC Future Manufacturing Hub

# **In Situ Synchrotron, Neutron and Laser Characterisation of Advanced Powder Processes**

***Peter D. Lee***

***Research Complex at Harwell***

**With: M. Preuss, P.J. Withers, P. Prangnall, CL (Alex) Leung  
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**ICL: Profs Jones & Saiz, Drs Kim, Zhang, Yue, Madi**

**Eric Jones; Chris Sutcliffe; Ulster: Prof. Mitchell**

**And many other colleagues, Companies, PDRA's and students**

**Funding: EPSRC (RCaH, MAPP), FP7 (AMAZE)**

**Didcot**

University of Oxford ~ 20km  
(London 1 hr)

**MRC Mammalian Genetics Unit & Mary Lyon Centre**

**Solid State NMR Centre**

**Research Complex**

**Central Laser Facility**

**Membrane Protein Laboratory**

**Diamond Light Source**

**eBIC**

**ISIS Neutron Source**

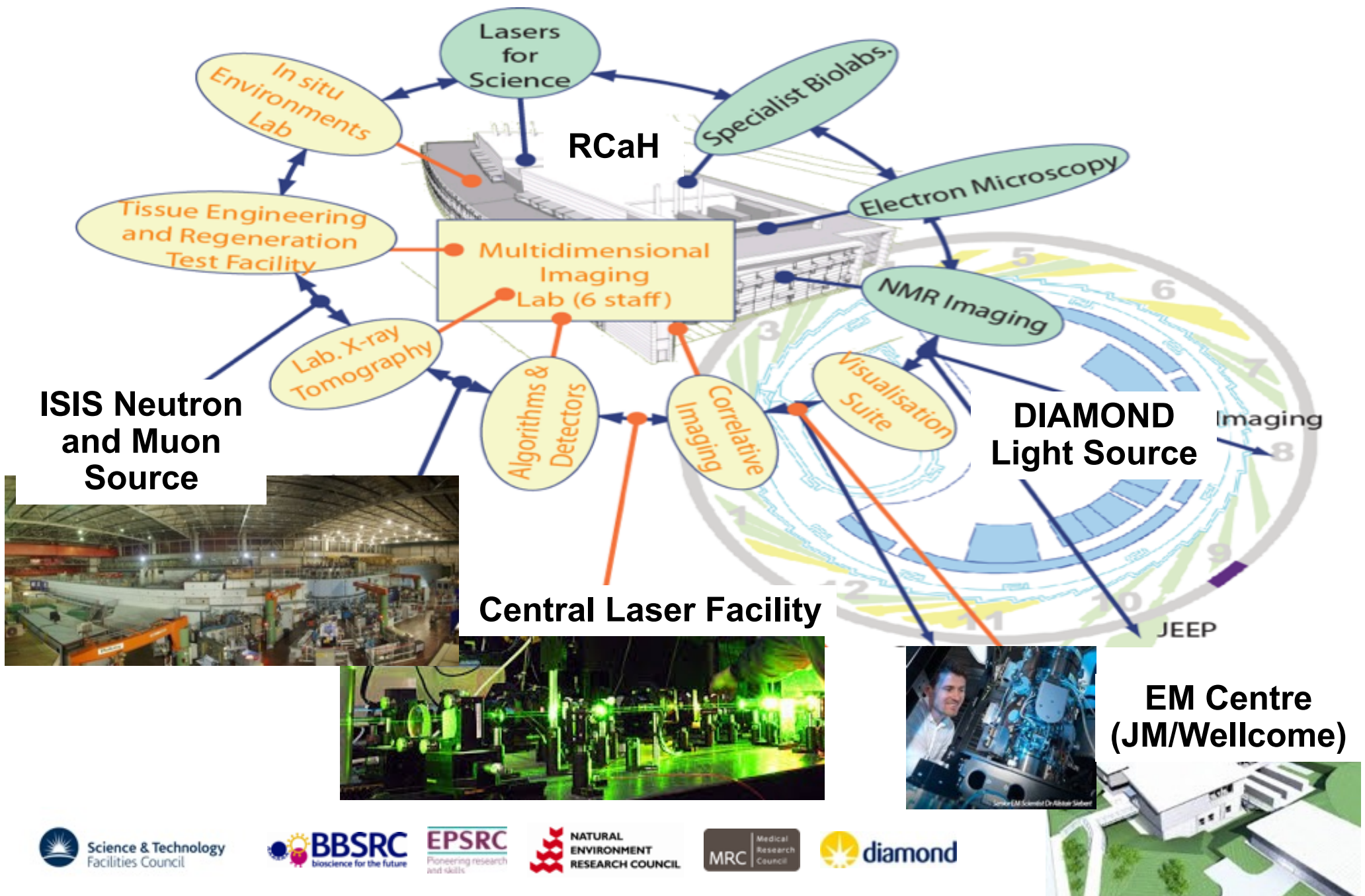
**Research Complex of Oxford**

The image is an aerial photograph of the Didcot Research Complex. A blue circle highlights the central area, which includes the Central Laser Facility, the Membrane Protein Laboratory, and the Diamond Light Source. A yellow arrow points from the University of Oxford towards the complex, indicating a distance of approximately 20km and a 1-hour drive to London. Various other facilities are labeled, including the MRC Mammalian Genetics Unit & Mary Lyon Centre, the Solid State NMR Centre, and the ISIS Neutron Source. A small inset image in the bottom right corner shows a modern building with large glass windows, likely part of the research complex.

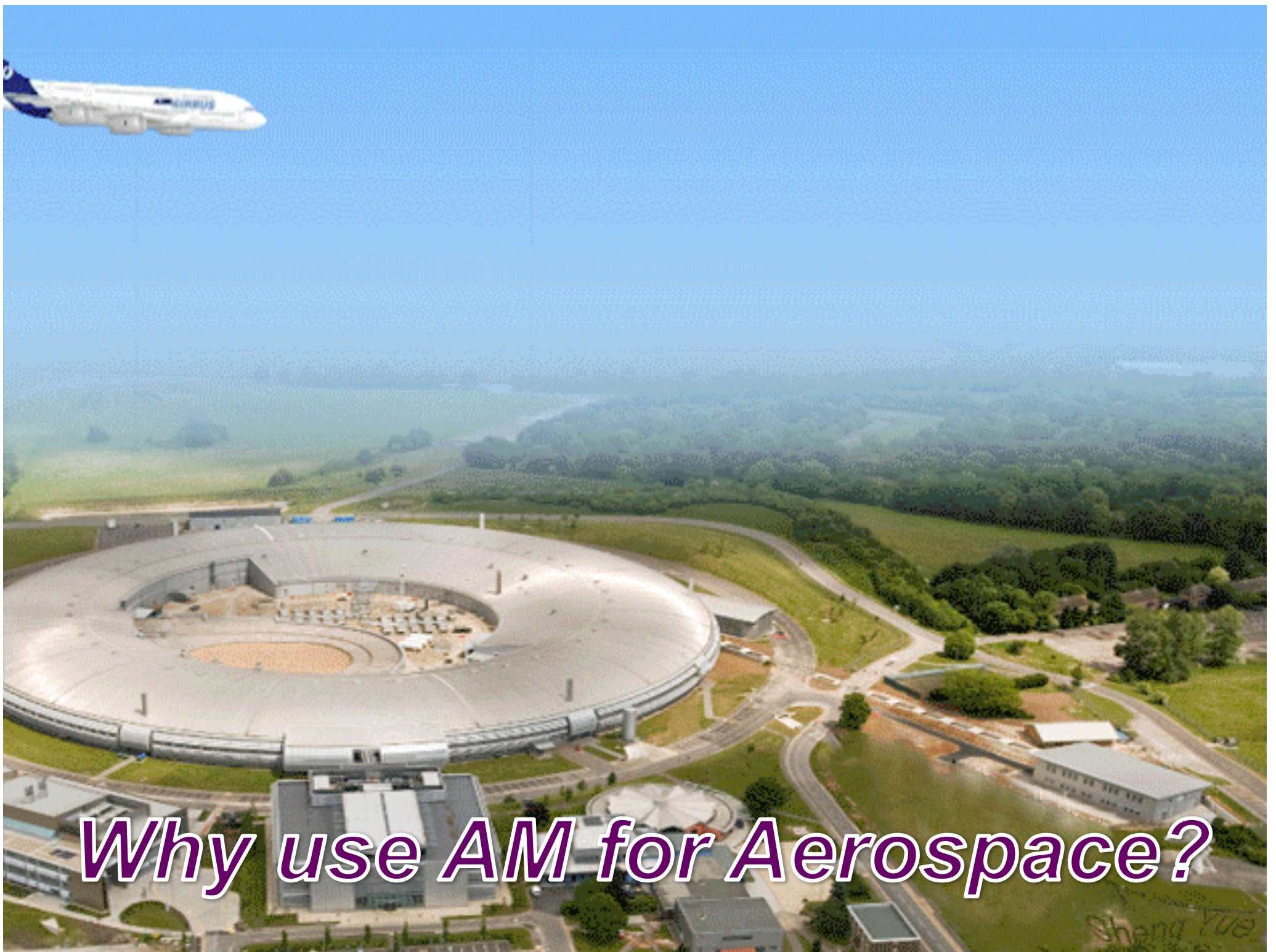




# The Research Complex at Harwell (RCaH)





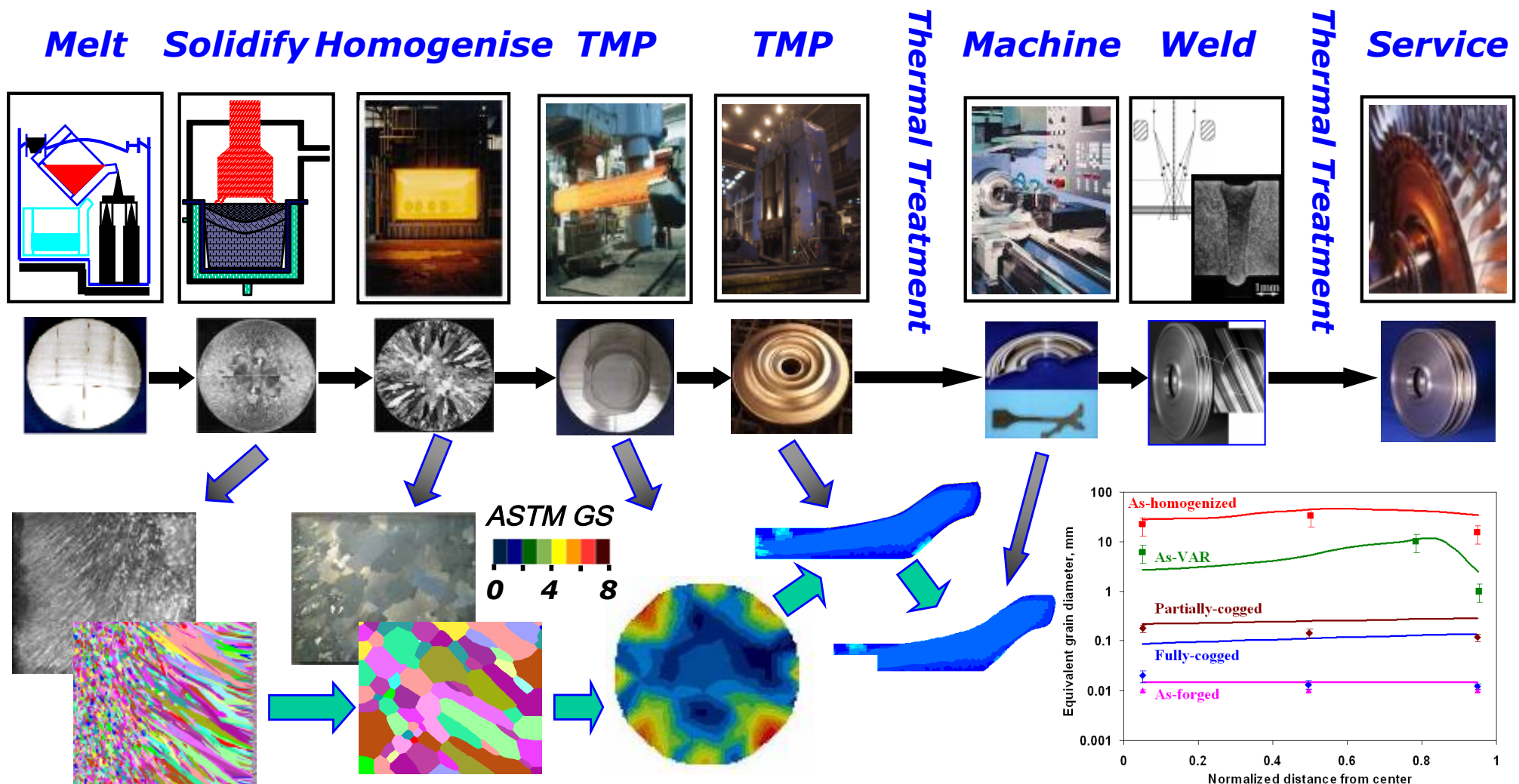


*Why use AM for Aerospace?*



# Why Advanced Powder Processes?

## Traditional Routes Involve many Process Steps ...



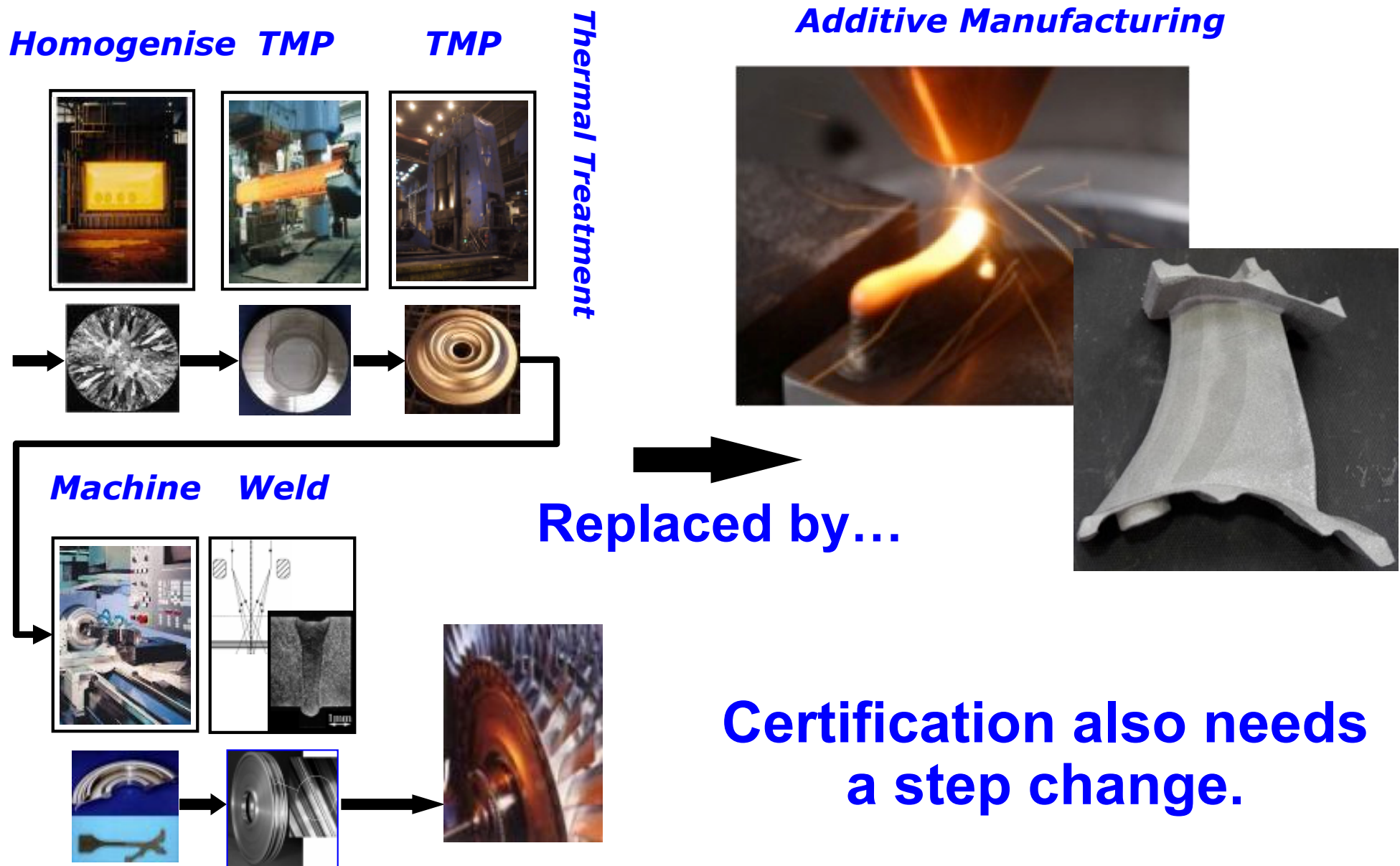
**For Aerospace, investment cost/time for manufacturing & certification is huge, whilst the number of components is low = *high cost per component***

With Rolls-Royce; Special Metals & Wyman-Gordon, Cambridge (Tin) and Birmingham (Ward)  
Kermanpur, Tin, Lee, JOM 56(3) 2004, 72-78. or Tin, Lee, et al Met. Trans. A., 2005.



# Why Advanced Powder Processing?

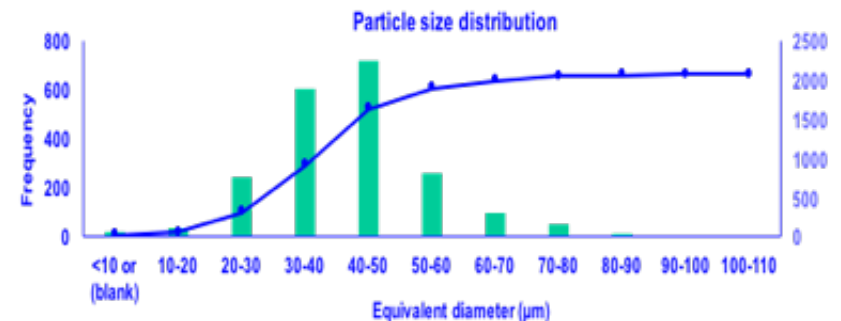
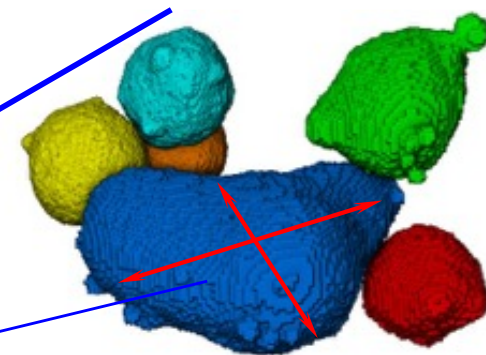
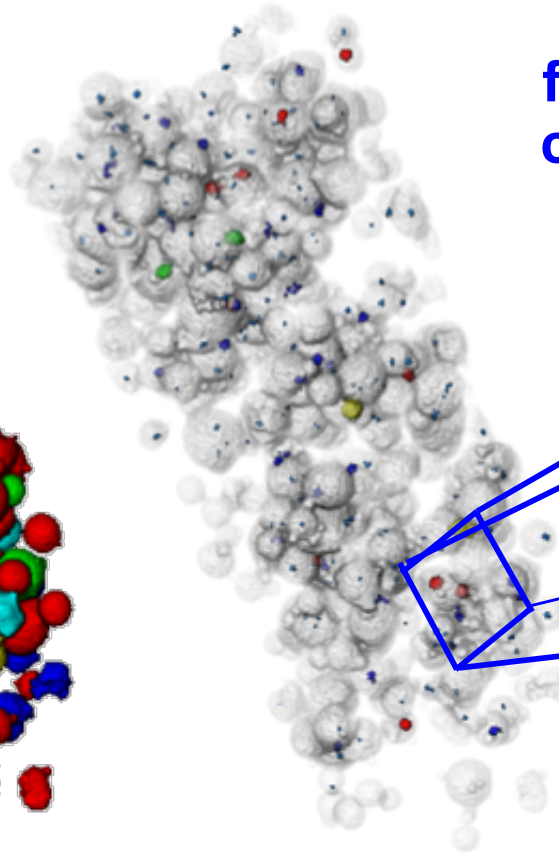
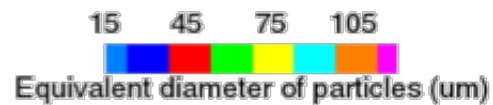
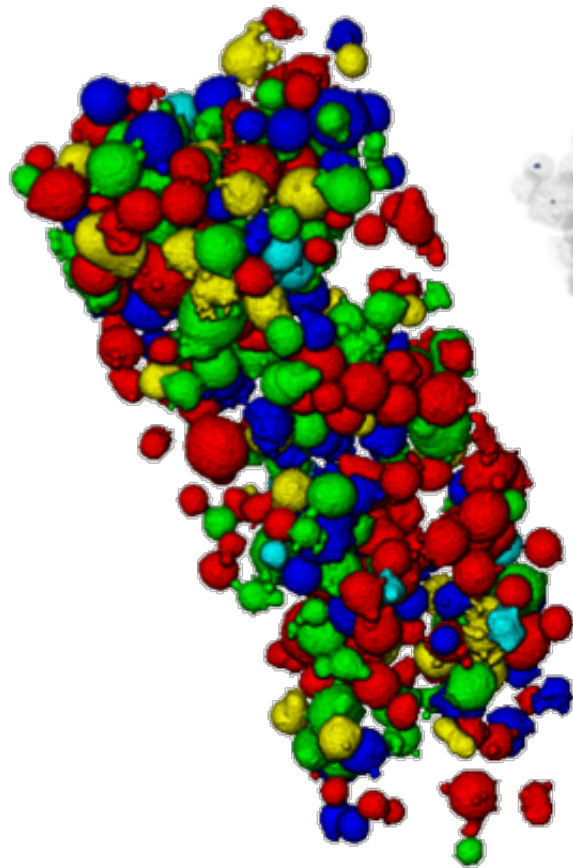
## Promise of faster/lower cost development & manufacturing





# Eg. 1: Characterising powders and their production

Using diffraction, spectroscopy, fluorescence we can characterise chemistry, crystal structure, etc...





## Example 2: Characterising powder processes *in operando* (SLM)

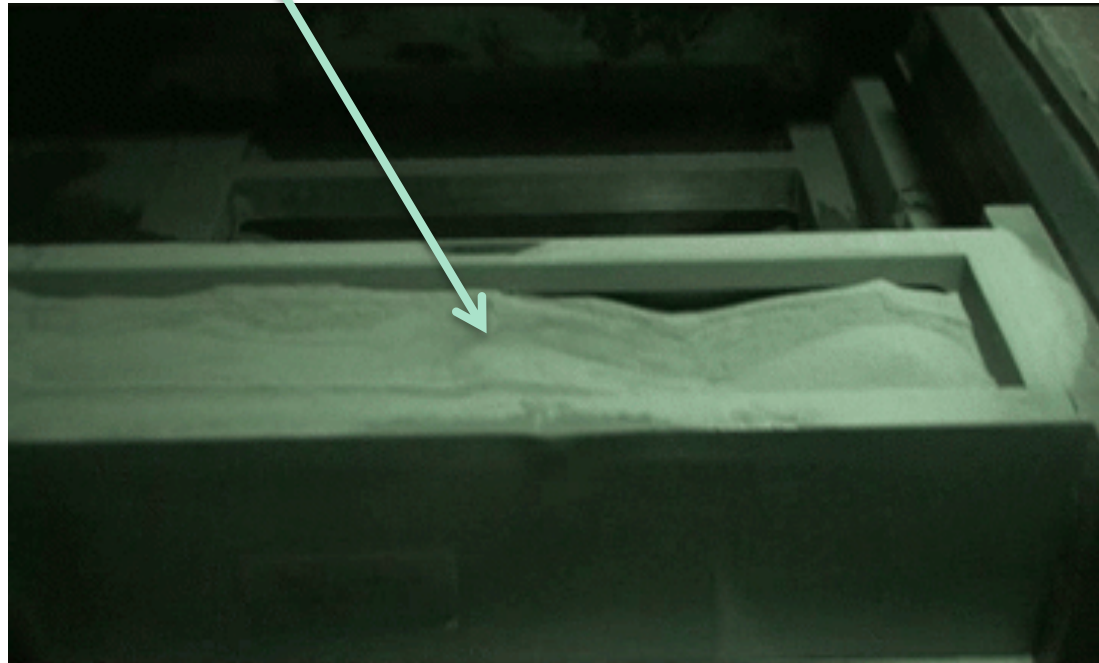
Design



Selective laser melting  
• Layer-wise fashion

Sinter

Ti selectively melted via scanning laser beam

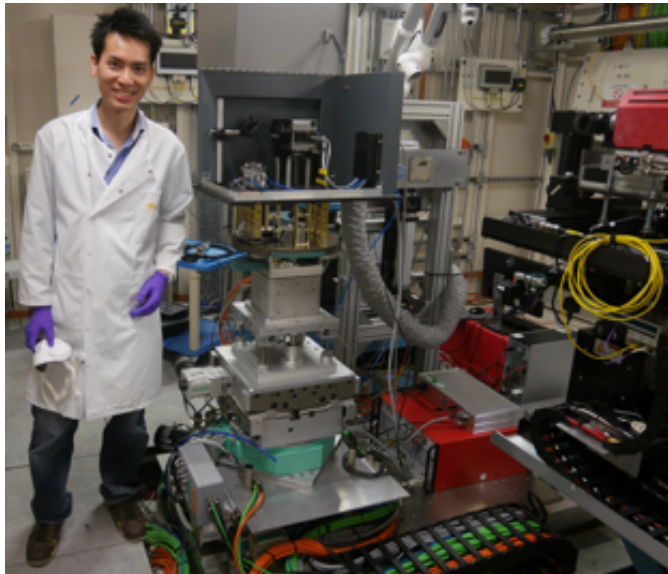


MCP Realizer 2, 250 SLM machine  
(MCP Tooling Technologies, UK)

# *In situ* AM Synchrotron Setup

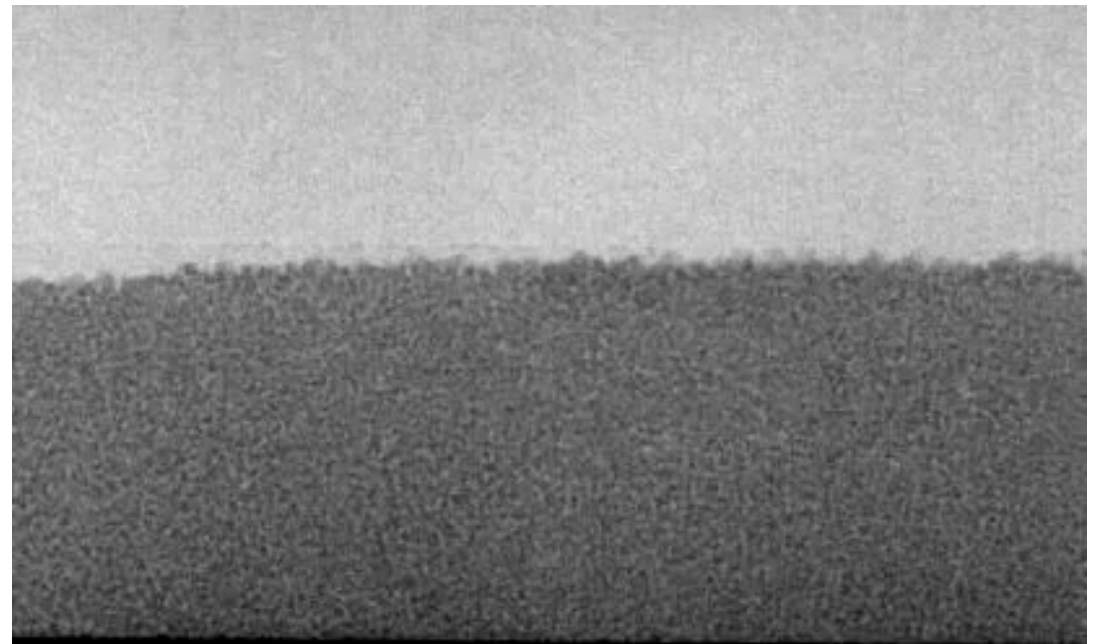
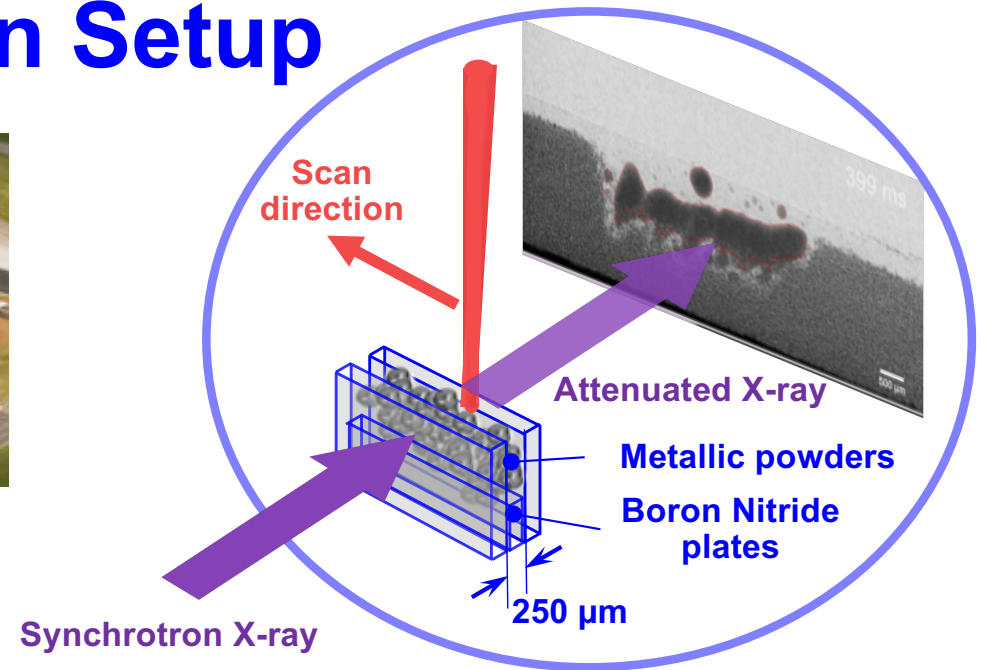


**Diamond Light Source**



**In situ AM on Beamline I12**

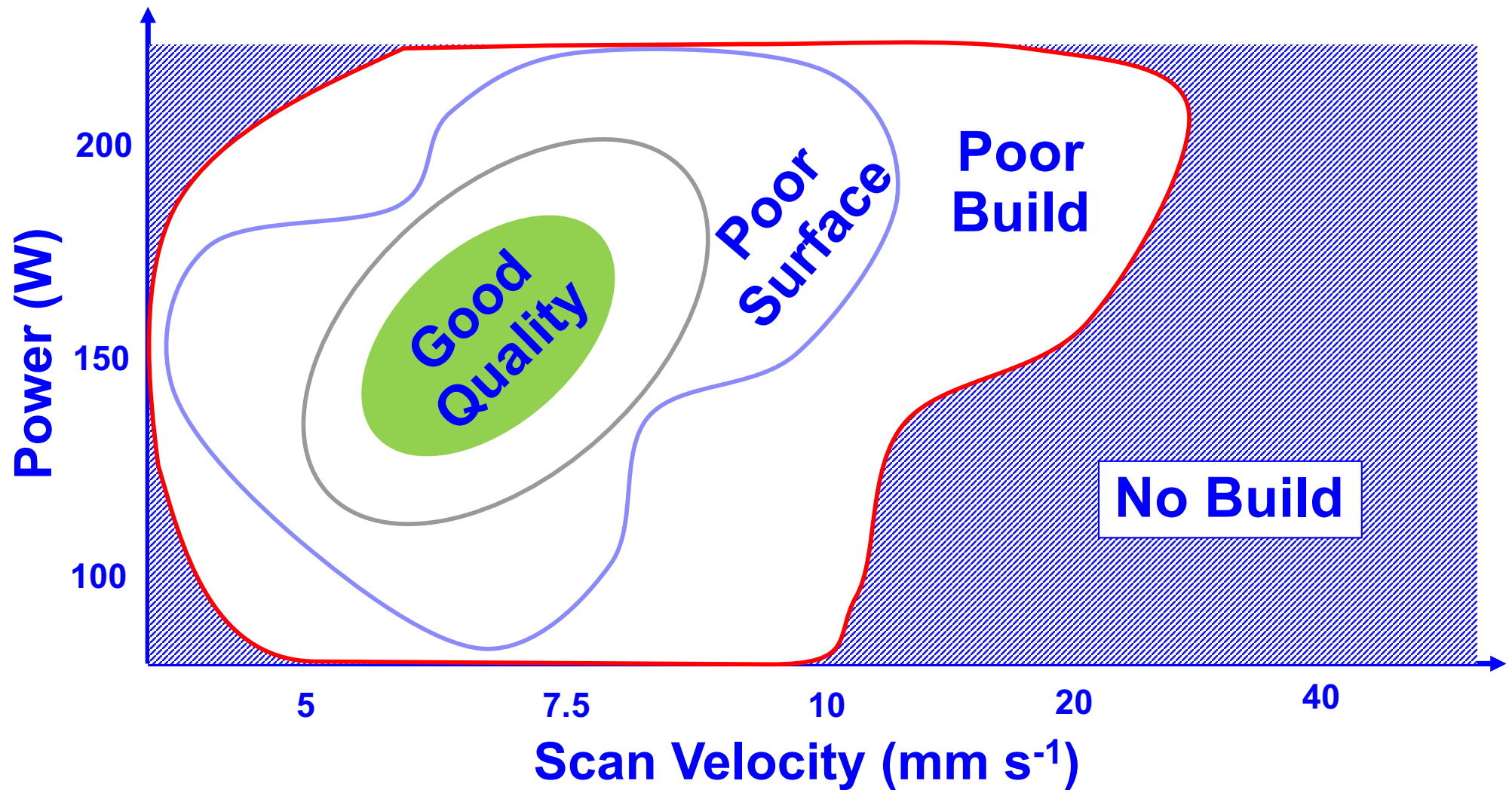
Leung, Lee, Towrie et al,  
Funding EPSRC (RCaH&MAPP), FP7



**SS316, 200W, 7.5mm/s, 5000fps**



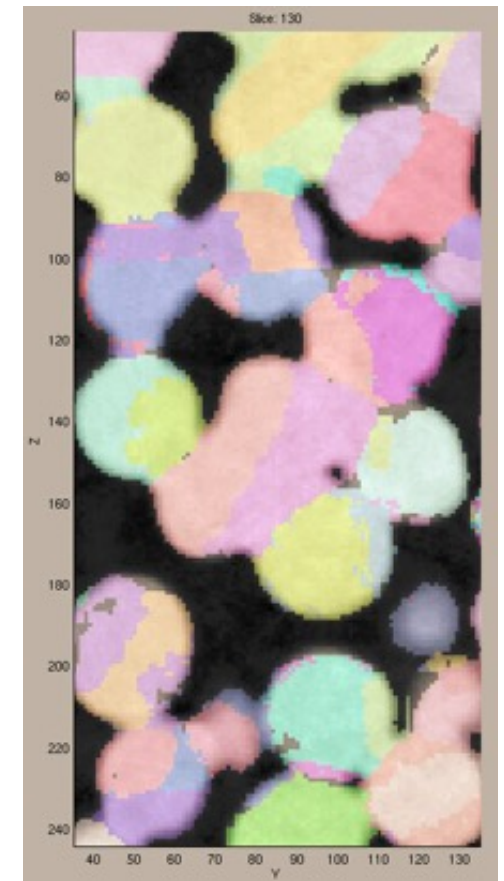
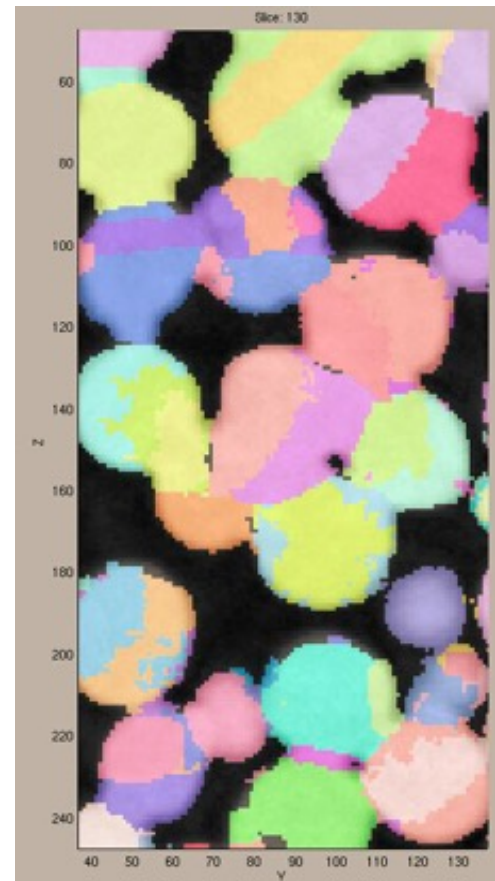
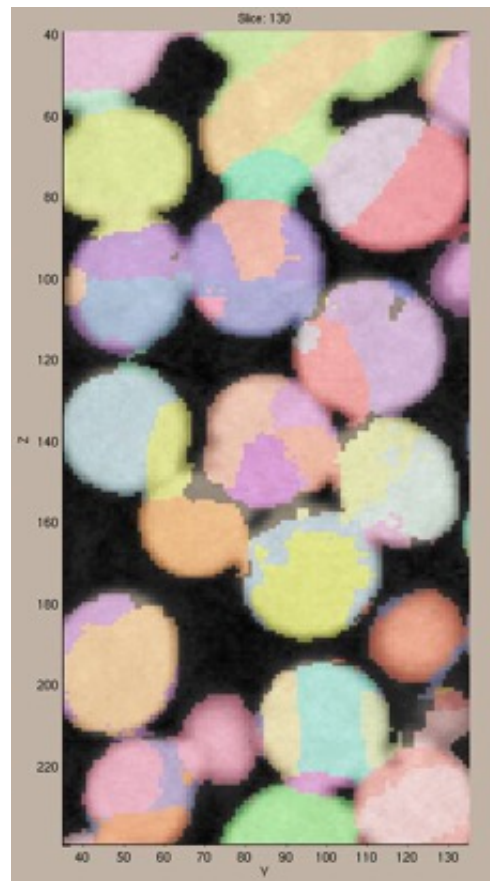
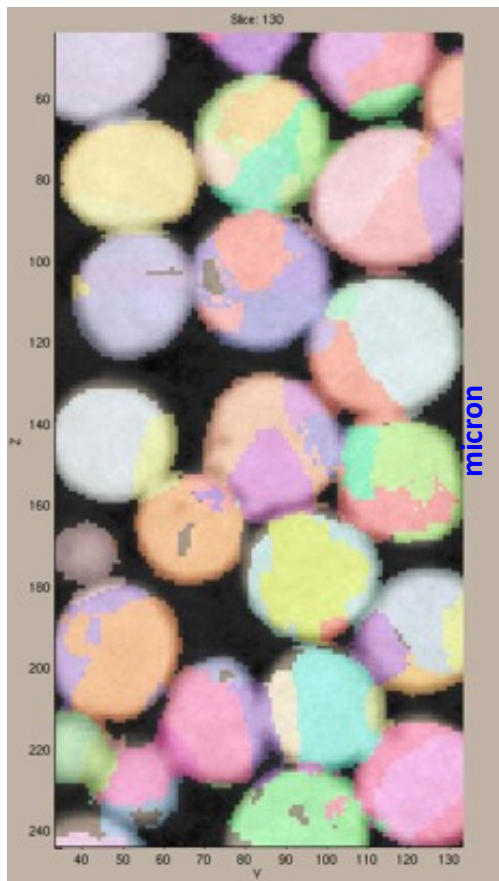
# A Process map for Laser Melting of SS316L Powder



# Example 3:

## Using laboratory Diffraction Contrast Tomography (DCT) to reveal grain evolution during sintering

Time →



— 50 microns

McDonald, Withers et al.



# **Example 4:**

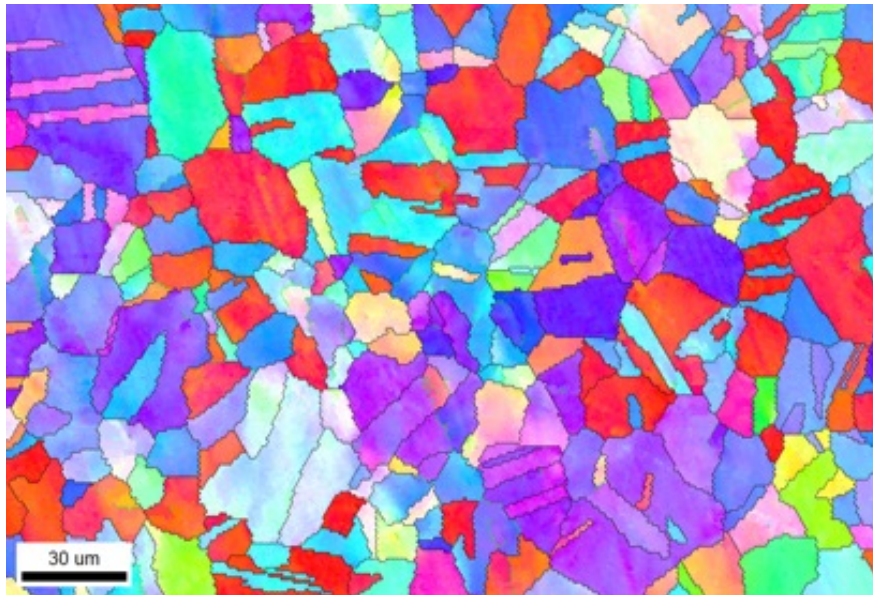
## **Characterising the properties of novel PM alloys using in situ Neutron Diffraction and TEM**



**ISIS Neutron Source, Harwell Campus**

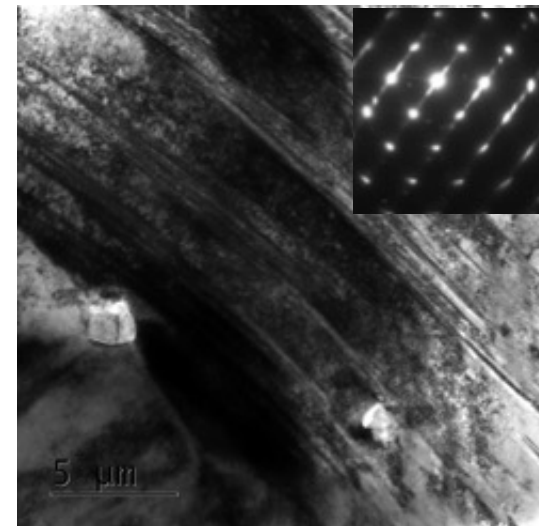
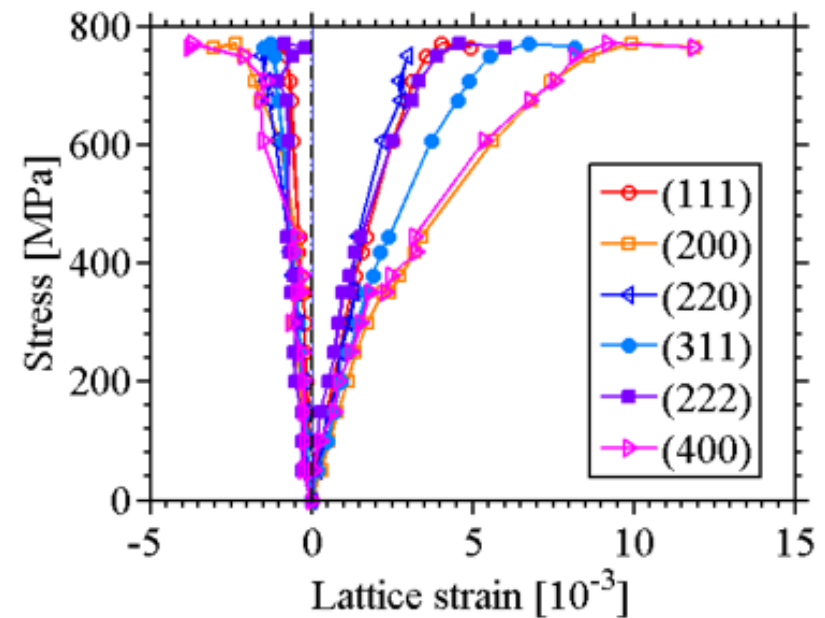
# Revealing how PM can improve properties

- PM significantly increased high entropy alloy properties
- *In situ* neutron diffraction & EM revealed why



grain structure

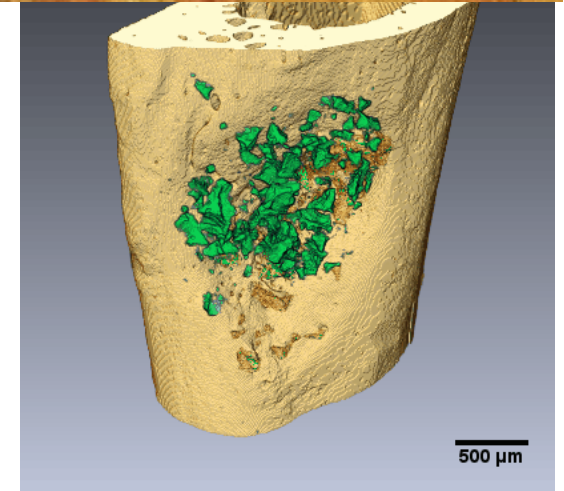
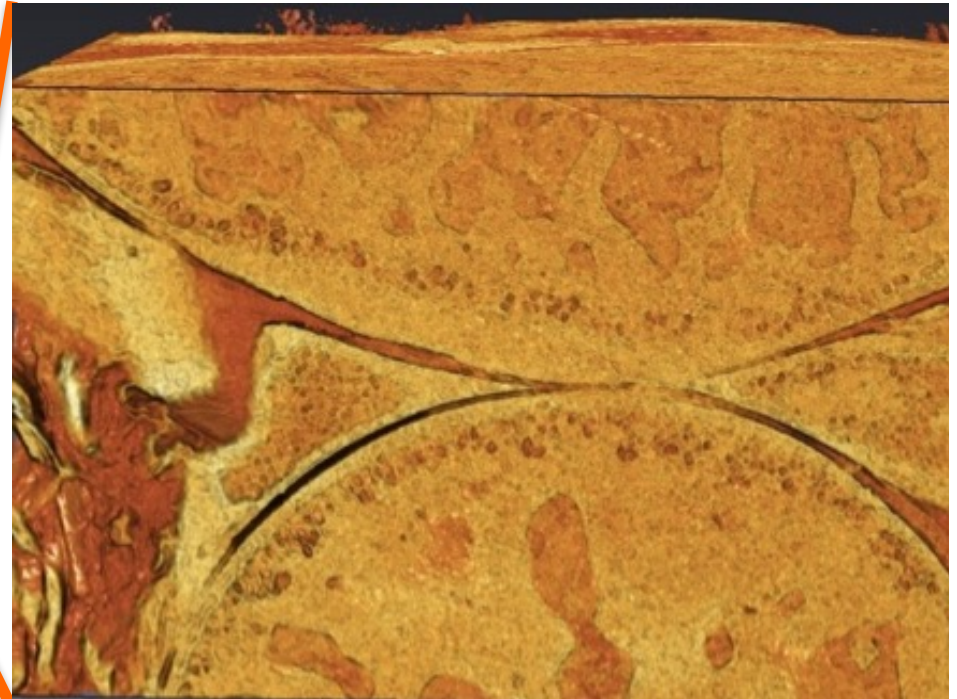
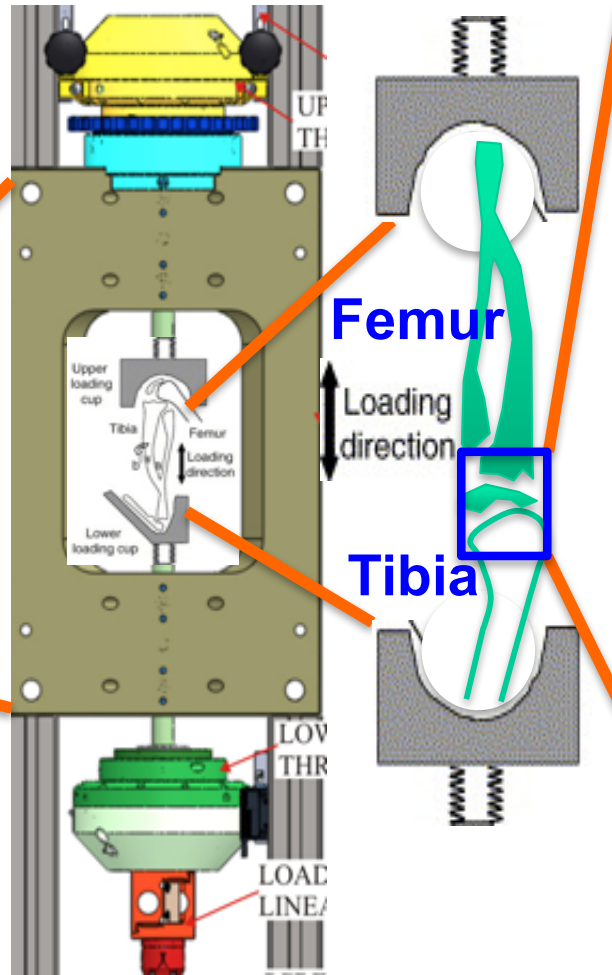
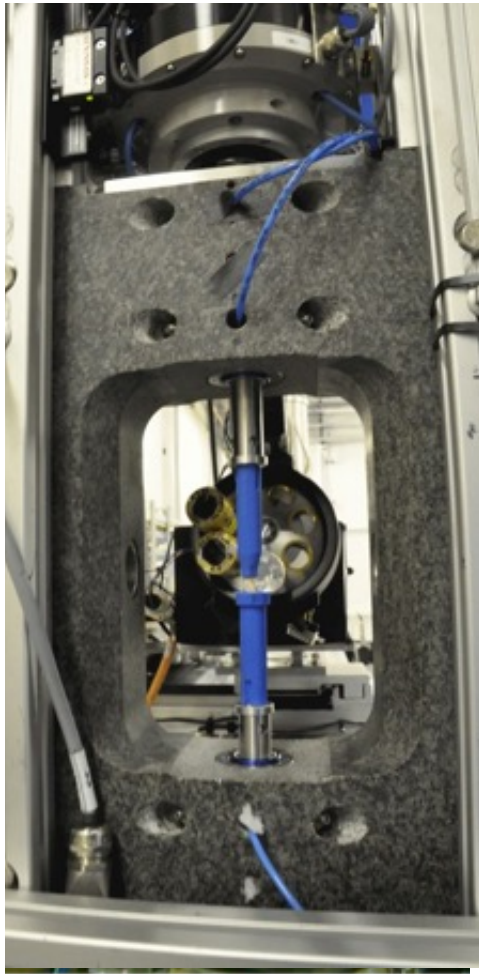
Cai et al, Acta Mat. 2017



deformation nano-twins  
formed due to tensile straining

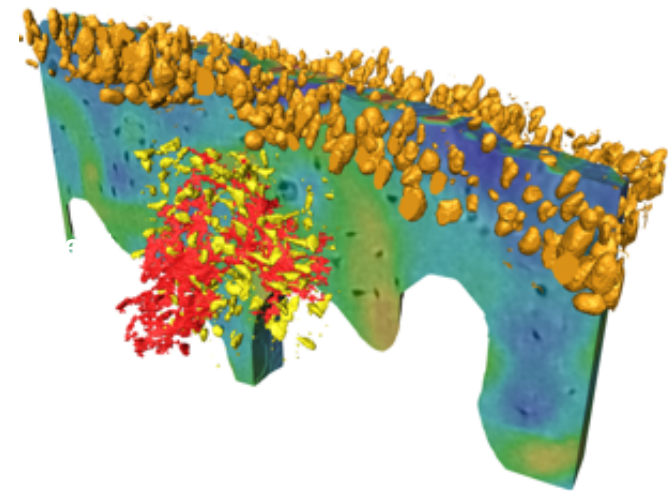
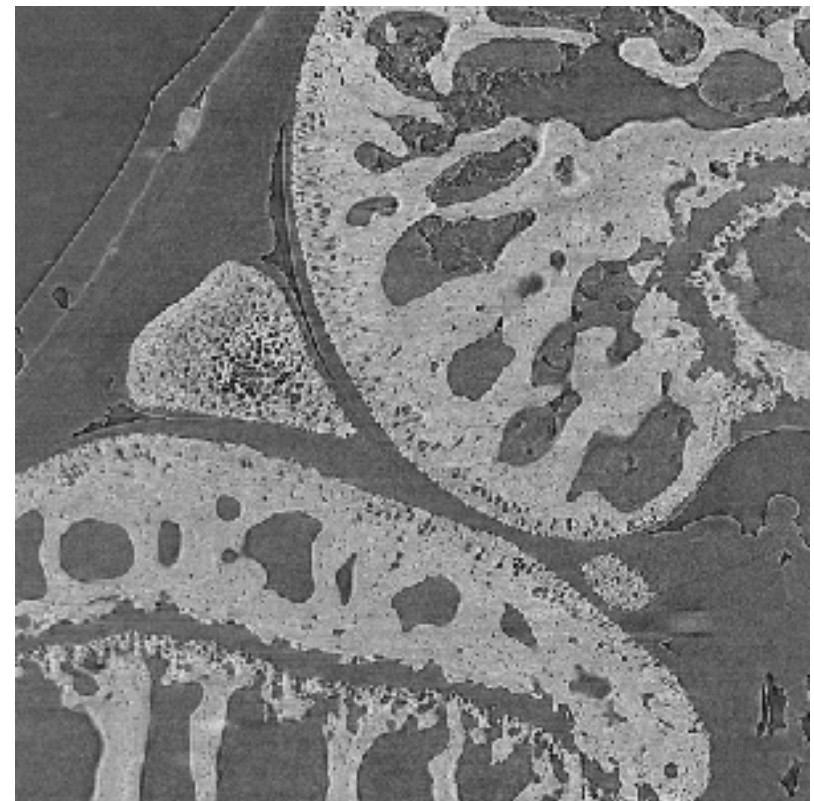
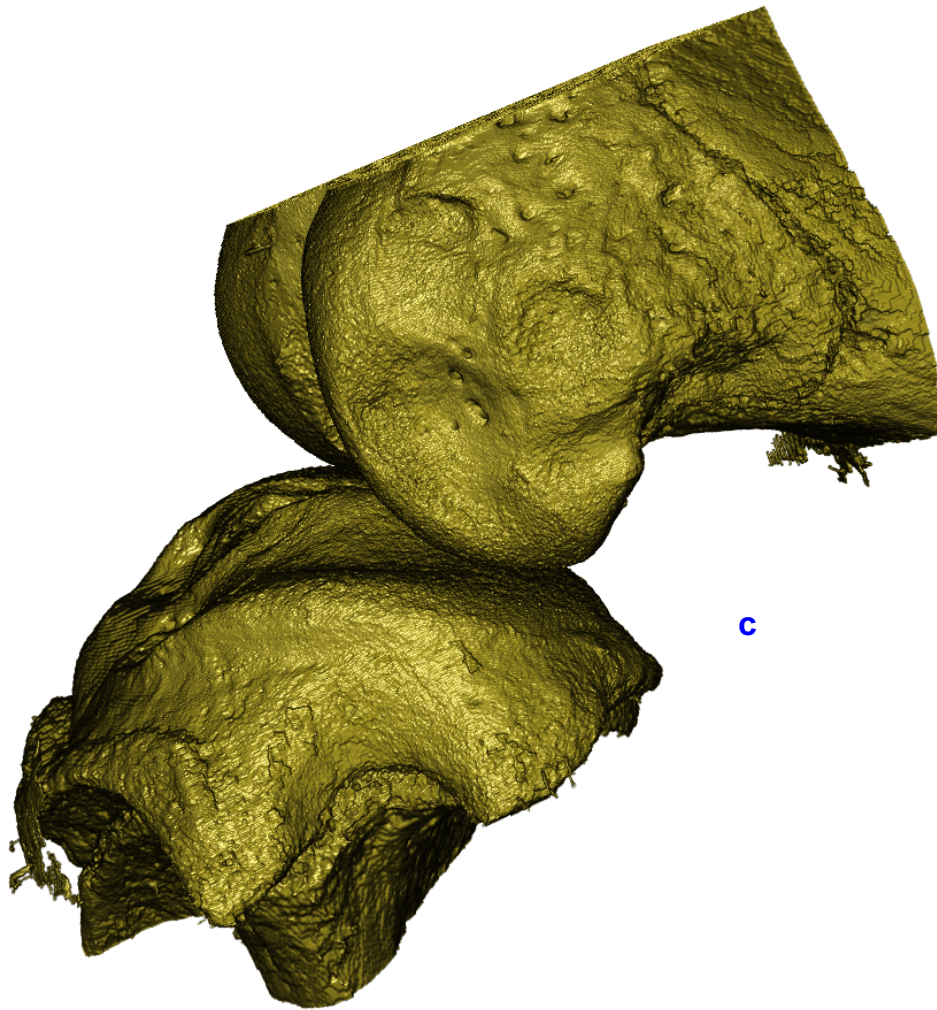


# In situ bio-mechanics simulations allow in service QA









w. RVC, OSU

*In situ 4D imaging and strain quantification*

## w. Liverpool, Ulster & ICL



*Helping develop 3D laser printed Titanium  
joint replacements, now in over 100,000 people*

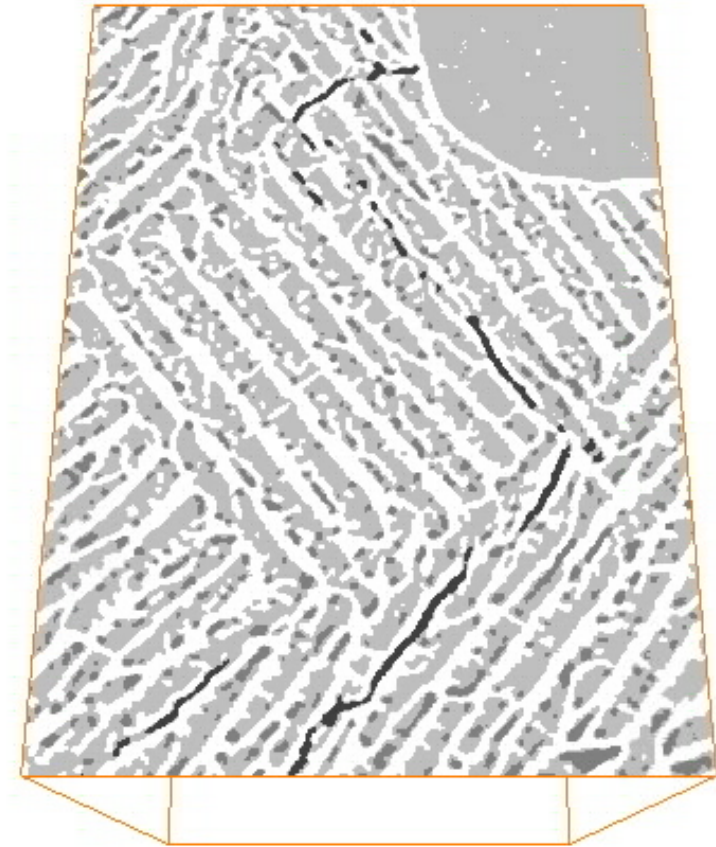
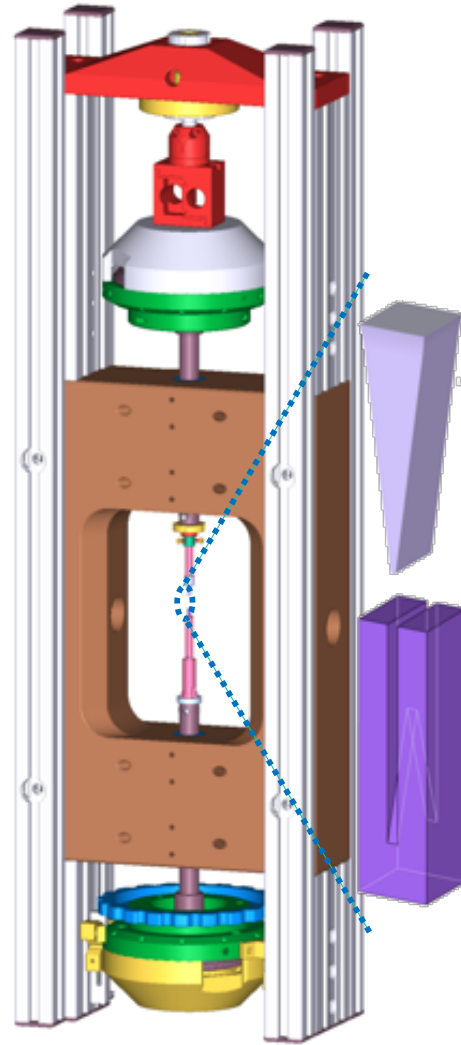
Singh, Lee et al, Acta Bio 2010, Kim et al, J. Mat. Pro. Tech. 2014





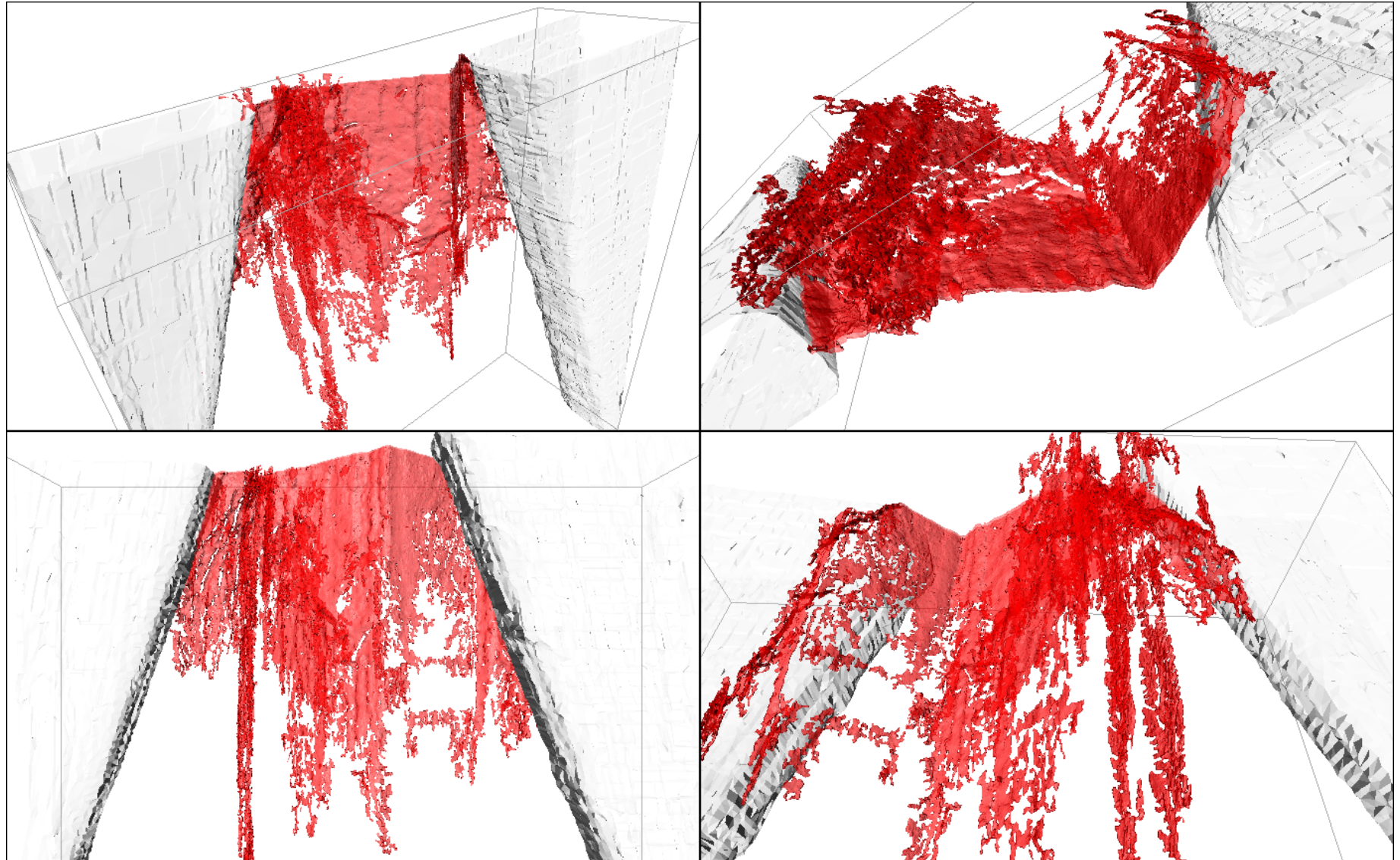
**Example 5:  
Quantifying In Service  
Performance of  
freeze cast ceramic powder -metal  
matrix composites**

# Tracking crack propagation in freeze cast ceramic-metal matrix composites



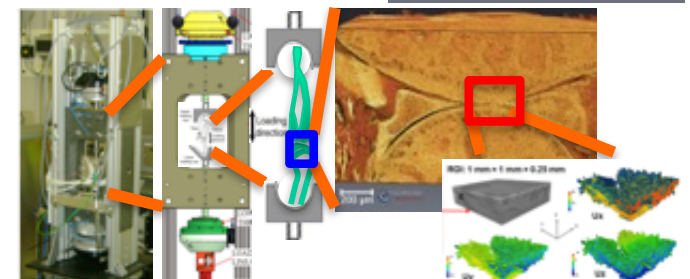
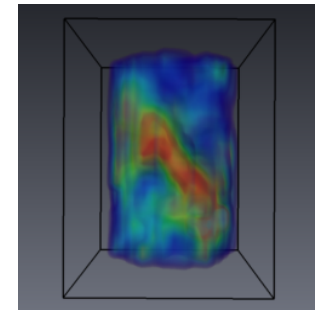
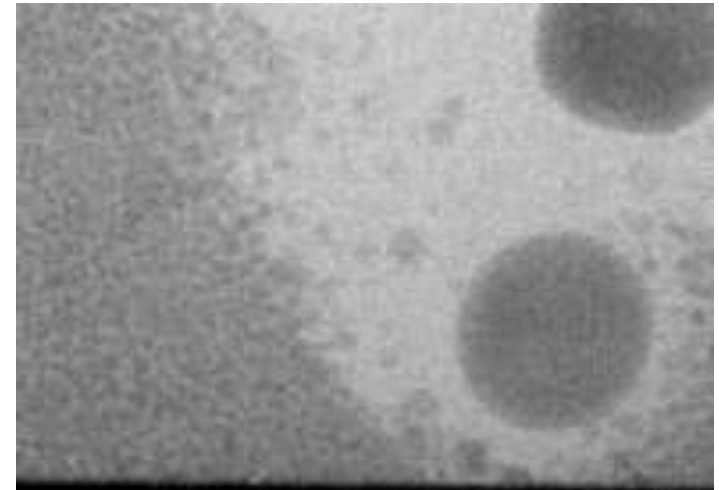
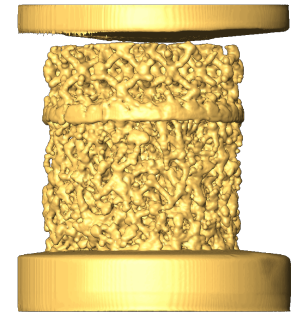


# 3D crack propagation



# Summary

- **Advanced characterisation impacts on:**
  - **Quality Assurance,**
  - **Quality Control**
  - **Performance evaluation/ improvement**
- **In situ synchrotron radiography and tomography impact on:**
  - **Process optimisation**
  - **In operando behaviour**
  - **Informing/validating ICME modelling**





# Acknowledgements

- PhD students, PDRA's, and support staff
- Research Complex at Harwell, STFC, ISIS, CLF
- EU-FP7 AMAZE (FP7-2012-NMP-ICT-FoF-313781)
- EPSRC (EP/I02249X/1, EP/P006566/1)
- Diamond Light Source, especially I12&13 Teams
- Funders and Collaborating Industry

## Questions?

