# ULTRAHIGH RESOLUTION 3D IMAGING AND CHARACTERISATION OF NANOSCALE PORE STRUCTURE IN SHALES AND ITS CONTROL ON GAS TRANSPORT

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## INTRODUCTION

Shale gas is the fastest growing energy sector globally. The imaging and quantification of size, geometry and distribution of the pores and minerals in shales is highly challenging due to their extremely fine grain size. Nano X-ray computed tomography (Nano-CT) combined with focused Ion Beam-Scanning Electron Microscopy (FIB-SEM) are used to resolve this problem and give us information in 3D from multiple scales in the shale gas extending down to resolutions of 50 nm.

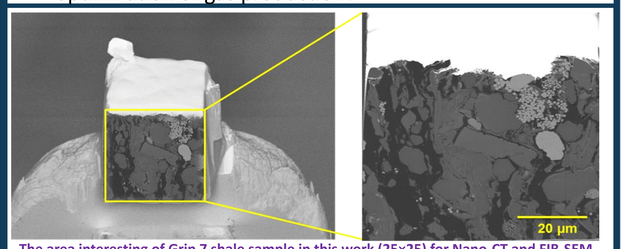
## AIM

To understand the relationship between fluid transport and pore microstructure in gas shales. Specific objectives include :-

- To undertake different measurements using FIB-SEM and Nano-CT and combine these imaging data to examine how the variability of the pore network architecture of shales at different scales affects the physical properties and unconventional reservoir quality.
- To characterise the petrophysical properties on a small rock shale sample (25 µm) on the same location that was used for the Nano-CT and FIB-SEM measurements to judge whatever the same place can represent the whole shale sample.

## SCIENTIFIC QUESTIONS

- How is the hydrocarbon stored in gas shale within the same area measurement by Nano-CT and FIB-SEM ?
- How does the microstructural distribution of porosity and kerogen in gas shale within the same site compare when applied by different measurements that can control the gas transport?
- How can gas shale microstructure be modified for the optimization of gas production?




The area interesting of Grip 7 shale sample in this work (25×25) for Nano-CT and FIB-SEM

## ACKNOWLEDGEMENTS

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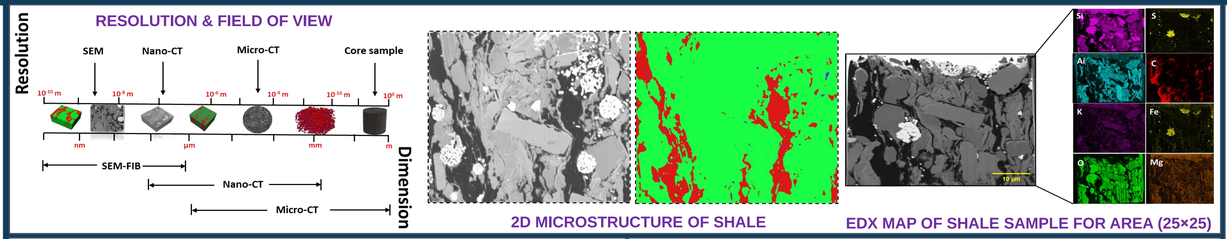
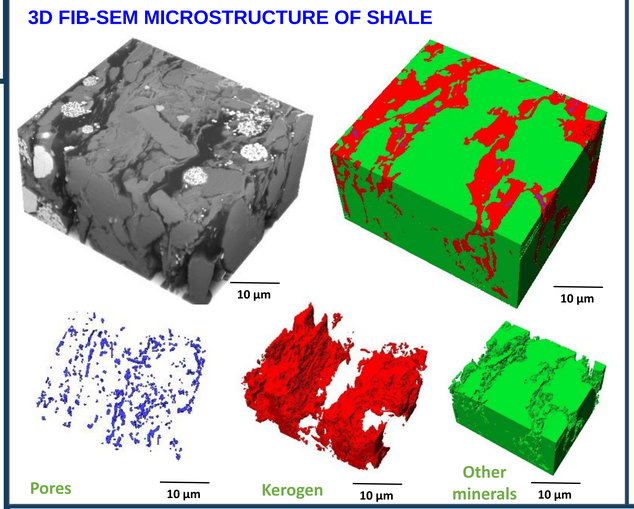
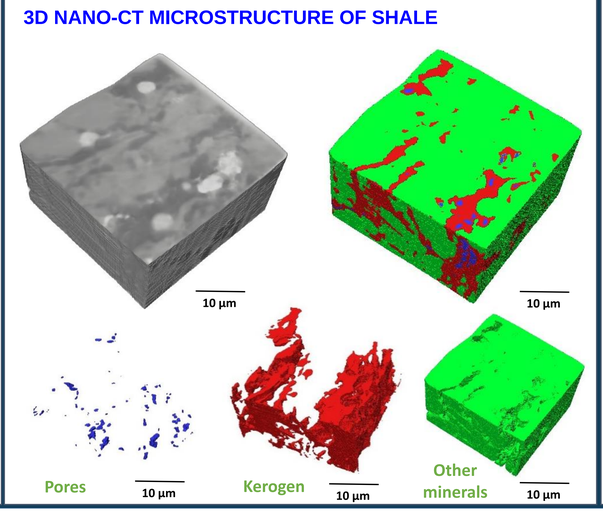


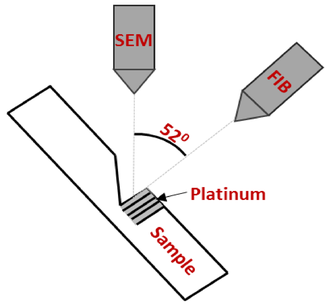



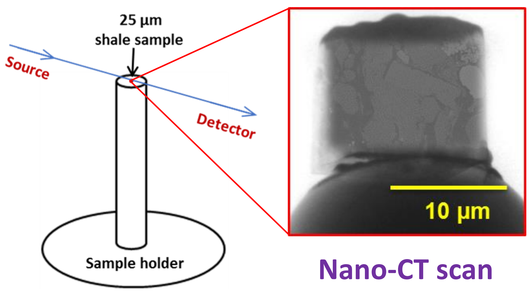

## METHODS

**Nano-CT** is used on the nano-level scale. Thousands of projections are taken as the sample is rotated through 360$^0$ and a series of 2D radiographs are then reconstructed into 3D images to reveal the internal structure of shale.

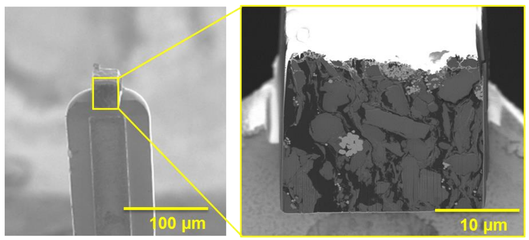

**FIB-SEM** is a destructive technique which uses an electron beam to progress-ively mill or remove material from the face of the sample between high resolution imaging of the exposed surface.




**Sample size: 25 µm**
**Pixel size: 70 nm**
**Non-destructive**




Nano-CT scan




FIB-SEM scan

- At the **micrometer scale** imaging, pores larger than 1 µm can be recognized by using the Nano-CT technique. The porosity and kerogen fraction of the Grip 7 sample are distributed discretely and account for 0.43% and 19.6%, respectively.
- At **nanometer scale** imaging at 50 nm by using FIB-SEM, the porosity for pores and kerogen are 0.7% and 26%, respectively.
- At **low resolutions** the pores show no connectivity. However, kerogen seems to be connected when viewed using the Nano-CT technique.
- At **high resolutions** both open pores and kerogen are clearly highly connected and will contribute to gas flow in the gas shale.

| Parameter | Sample Grip 7 | Resolution (Pixel size) |
|---|---|---|
| FIB-SEM Porosity for pore | 0.7% | 50 nm |
| FIB-SEM Porosity for Kerogen | 26.0% | 50 nm |
| Nano-CT Porosity for pore | 0.43% | 70 nm |
| Nano-CT Porosity for Kerogen | 19.6% | 70 nm |
| FIB-SEM Permeability | 2.55 nD | 50 nm |
| Nano-CT Permeability | 9.92 nD | 70 nm |

## CONCLUSIONS

- FIB-SEM and Nano-CT can be applied in tandem for the quantification of the fine scale pore network architecture characterisation because they interrogate different scales.
- In each case, different shale components can be distinguished.
- The 2D/3D microstructural properties on shale change from sample to sample, for different scales and using different experimental measurements.

## FUTURE WORK

Work is currently being carried out on improvements in the following areas:

- The flow, quantification, type and distribution of kerogen.
- The size distribution and connectivity of pores as a function of scale, distinguishing between key finely connected porosity and larger unconnected porosity.
- The balance between sample representatively and resolution.
- Permeability simulation based on empirical modelling, upscaling and the use of machine learning methods.

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