Mineralogical and Petrophysical Characterisation of a Fine-Grained Sandstone With Significant Clay Coating Using 3D Micro-CT and SEM Imaging From a 5 mm Plug*

Alexandra Golab[1], Alessio Arena[1], Ishtar Barranco[2], Rafael Salazar-Tio[3], Joseph Hamilton[1], Nasiru Idowu[1], Puvaa Rajan[1], Silvano Sommacal[1], Benjamin Young[1], Anna Carnerup[1], Josephina M. Schembre-McCabe[3], and Keith Boyle[2]

Search and Discovery Article #41756 (2015)
Posted January 18, 2016

*Adapted from oral presentation given at AAPG/SEG International Conference & Exhibition, Melbourne, Australia, September 13-16, 2015

[1]Digital Rock Services, FEI Lithicon, Canberra, ACT, Australia
[2]Chevron Australia Pty Ltd, Perth, WA, Australia (irina.borissova@ga.gov.au)
[3]Chevron Energy Technology Company, Houston, TX, United States

Abstract

This project demonstrates how high resolution 3D X-ray microcomputed tomographic (micro-CT) imaging integrated with higher resolution SEM imaging and automated quantitative mineral mapping may be used to digitally determine petrophysical properties, e.g. porosity, permeability, relative permeability, capillary pressure curves, formation resistivity factor, Archie's cementation exponent, grain size distribution, pore and throat size distribution, water saturation, and elastic properties. The workflow was established on a sub-plug (5 mm long by 5 mm diameter) from a core plug of an Australian Jurassic, fine-grained sandstone with significant grain coating. High resolution 3D micro-CT images of the sub-plug were acquired in as-received state and after saturating it with X-ray dense brine. The images were registered in 3D into perfect geometric alignment then a difference map was created of the connected porosity including pores that are below the resolution of the micro-CT images. The sample contains a lot of pore-filling materials and the grains are coated by clay. Segmentation of the 3D tomograms was carried out to map the 3D distribution of the grain coatings and other minerals, and capture resolved as well as sub-resolution porosity. BSEM imaging (at higher resolution than the micro-CT images) of a polished section through the sub-plug was performed to better characterise the porosity of the pore-filling and grain-coating minerals, and automated quantified mineral mapping by SEM-EDS (QEMSCAN™) was performed to identify the minerals and improve the segmentation. A pore network was then extracted from the segmented images and used as input into a quasi-static pore network model for multiphase simulations. The capillary pressure simulation was compared with experimental mercury injection data. There is a good agreement between the simulated and experimental data in the resolved porosity (macroporous) region of the curve.

Mineralogical and Petrophysical Characterisation of a Fine Grained Sandstone with Significant Clay Coating Using 3D Micro-CT and SEM Imaging from a 5mm Plug

Golab, A.[1], Arena, A.[1], Barranco, I.[2], Hamilton, J.[1], Idowu, N.[1], Rajan, P.[1], Sommacal, S.[1], Salazar-Tio, R.[2], Schembre, J.[2], Young, B.[1], and Carnerup, A.[1]

[1] FEI [2] Chevron

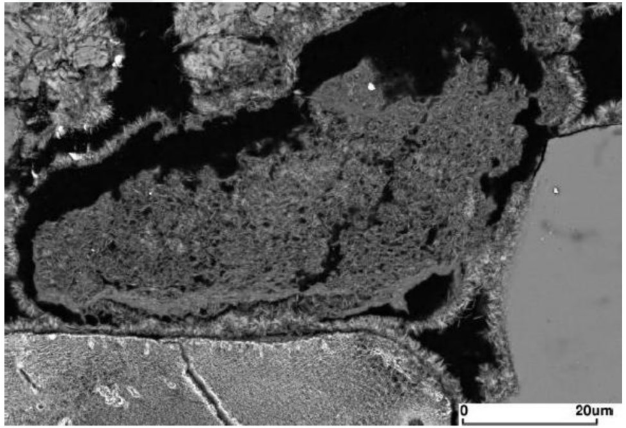
Ishtar Barranco

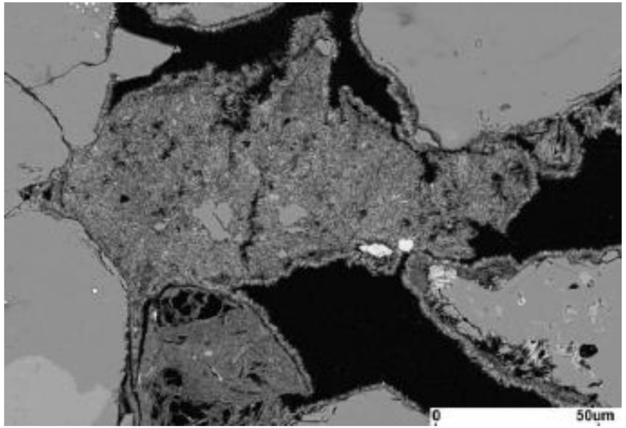
presenting on behalf of Alexandra Golab

Agenda

- Project Overview
- Workflow
- 3D micro-CT images and segmentation
- SEM imaging and QEMSCAN mineral analysis
- Petrophysical properties simulation
- Conclusions

Project Overview

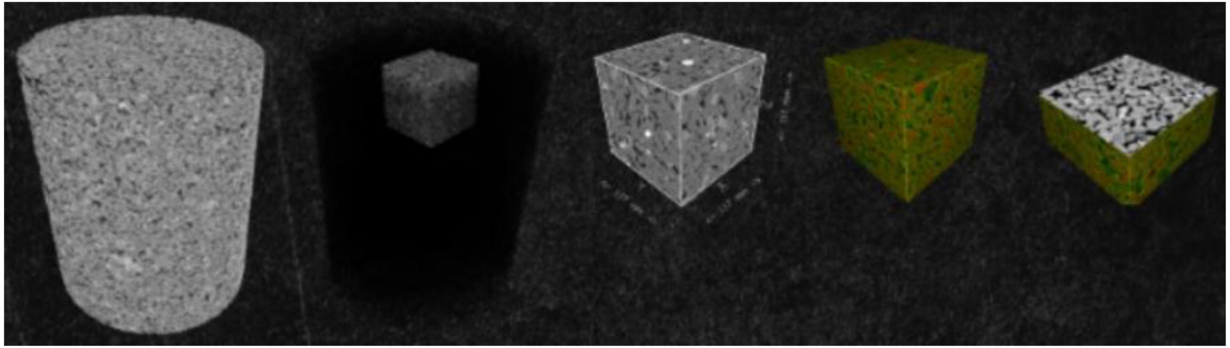




- The purpose of the study was to evaluate if using a mini plug (~5 mm) it was possible to accurately determine petrophysical properties such as Ø, k, kr and Pc curves, F, m, grain size distribution, pore and throat size distribution, Sw, and elastic properties
- These properties were also measured at the lab using a 1.5” plug from conventional core to compare the results
- A fine grained sandstone with significant grain coating was selected for the study because the clay coating thickness was below the micro-CT scan resolution and it has a significant impact on the petrophysical properties

Workflow

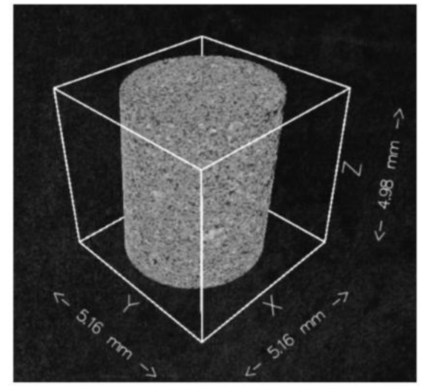
1. High resolution 3D micro-CT (5 mm sub-plug): as-received state and X-ray dense brine saturated
2. 3D images registered in perfect geometric alignment for the as-received and saturated state
3. Difference map of the connected porosity to identify pores below micro-CT images resolution
4. Segmentation of the X-ray distinct minerals in the 3D tomograms
5. BSEM imaging (at higher resolution than the micro-CT images) of a polished section through the sub-plug to better characterise the porosity of the pore-filling and grain-coating minerals
6. Automated quantified mineral mapping (QEMSCAN™) to improve segmentation
7. Pore network model extracted from the segmented images and used as input for multiphase simulations



Plug properties

Plug ~ 7 cm

Sub - plug ~ 5 mm



Properties from digital core analysis:

Porosity: 27.7% Permeability: $k_{x,y}$ = 140 – 145; k_z = 154 mD

$F_{x,y}$ = 8.58 – 8.74; F_z = 8.2; m =1.72

S_{wi} = 0.44; S_{CO2r} = 0.224; Krw @ S_{CO2r} = 0.15

Explore. Discover. Resolve.

Presenter's notes: The core plug shown on this slide was imaged in 3D by X-ray micro-computed tomography (micro-CT) and it was confirmed from the image that the plug is homogeneous in terms of grain size and distribution. The image was used to decide the location and diameter of a sub-plug to be drilled from the core plug. The sub-plug was 5mm in diameter.

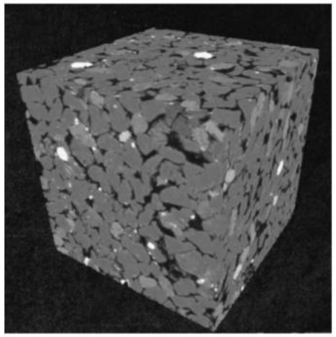
(Presenter's notes continued on next slide)

(Presenter's notes continued from previous slide)

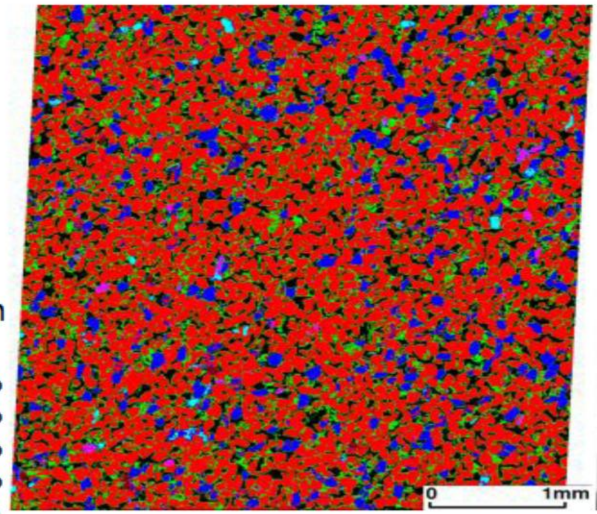
The properties shown on this slide are from digital core analysis, not lab measurements.
Swi= Initial water saturation
Sco2r= residual CO_2 saturation
Krw = relative permeability of water

HR Scan (5mm): As-received state Tomogram

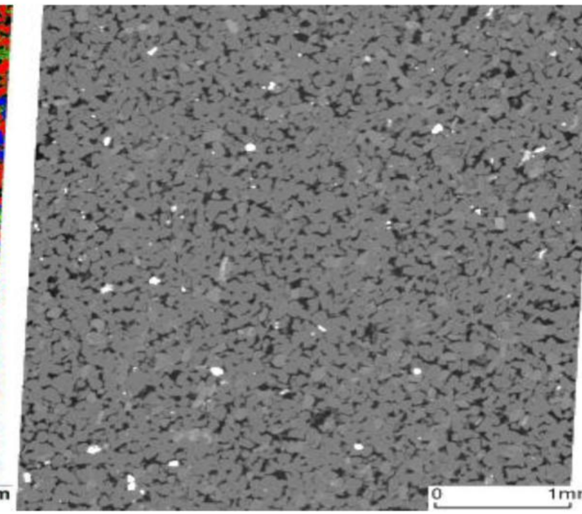
Voxel Size: 2.93µm
X-Slice



Mineral Segmentation



As-received state Tomogram



Phase volume fraction calculation - results for 3D image:

Resolved Porosity	:	16.8 %
Microporous Phases	:	17.3 %
Quartz	:	56.0 %
K feldspar	:	8.3 %
High Density Phase 1	:	0.8 %
High Density Phase 2	:	0.5 %
Zircon	:	0.2 %

Presenter’s notes: The sub-plug was imaged in 3D by helical micro-CT.
In micro-CT imaging, X-rays pass through the rock sample and the different minerals and pore space attenuate the X-rays in different ways. It is possible to separate the different minerals from one another in 3D and quantify their content.
Note here that the ‘resolved’ porosity refers to voxels (cubes that here are 2.93 micron in size) that are entirely filled by pore space and do not contain any mineral matter. ‘microporous’ phases refers to voxels that have a partial contribution from pores and mineral matter.

HR Scan (5mm): Saturated state Tomogram

Voxel Size: 2.93µm

X-Slice

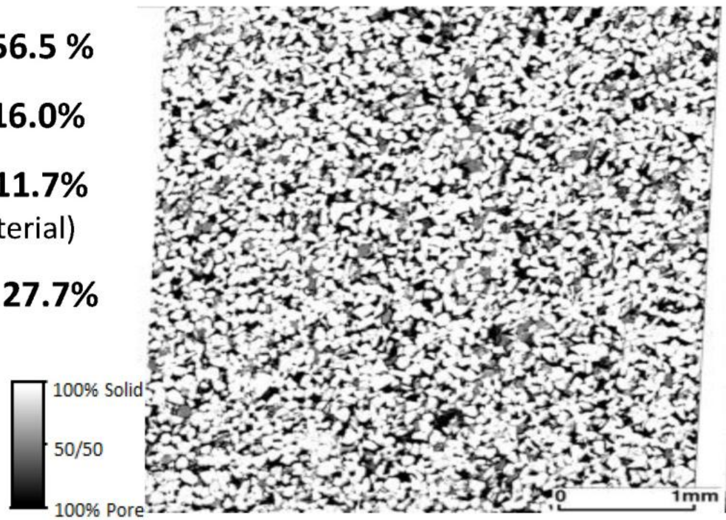
Solid fraction : **56.5 %**

Resolved porosity : **16.0%**

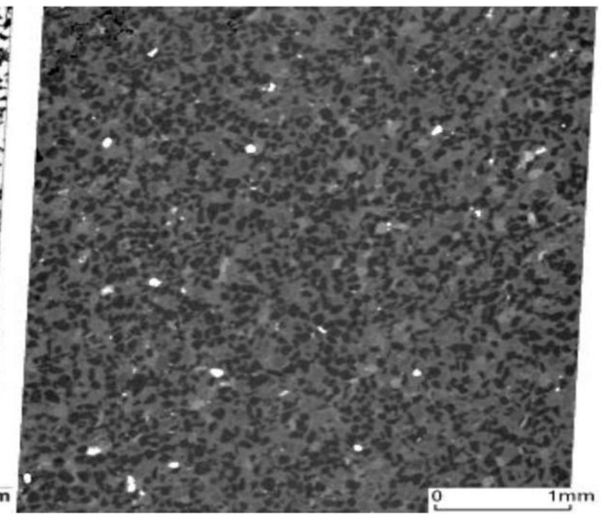
Microporosity : **11.7%**
(from 27.52% porous material)

Total porosity : **27.7%**

Porosity Map



Saturated state Tomogram



Saturation brine: 1M KCl/0.6M NaI

Explore. Discover. Resolve.

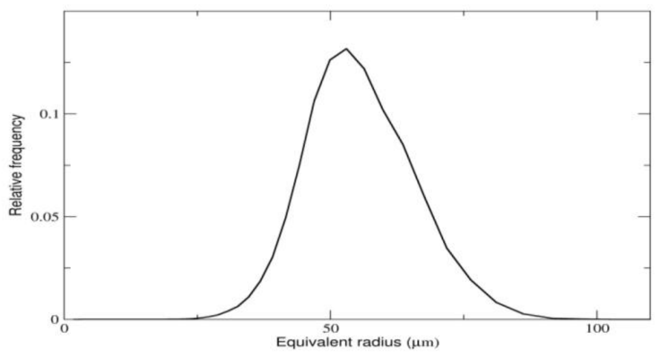
Presenter's notes: In order to accurately capture the porosity of the microporous minerals in this sample, the sub-plug was saturated with an X-ray dense brine with composition shown on the slide. The brine fully enters all pores and porous material and is bright to X-rays. The sub-plug was then re-imaged. The as-received state and saturated-state tomograms were then registered into perfect geometric alignment and one was subtracted from the other, thus removing the signal from mineral matter and leaving a difference tomogram with the only signal being from the brine-invaded pore space. This difference tomogram was then segmented in 3D to create an accurate porosity map with a total effective porosity of 27.7%.

HR Scan (5mm): Grain Clusters

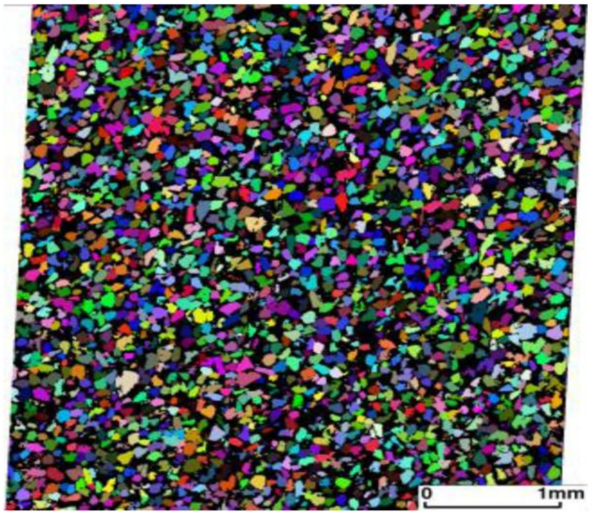
Voxel Size: 2.93µm

X-Slice

Grain Size Distribution

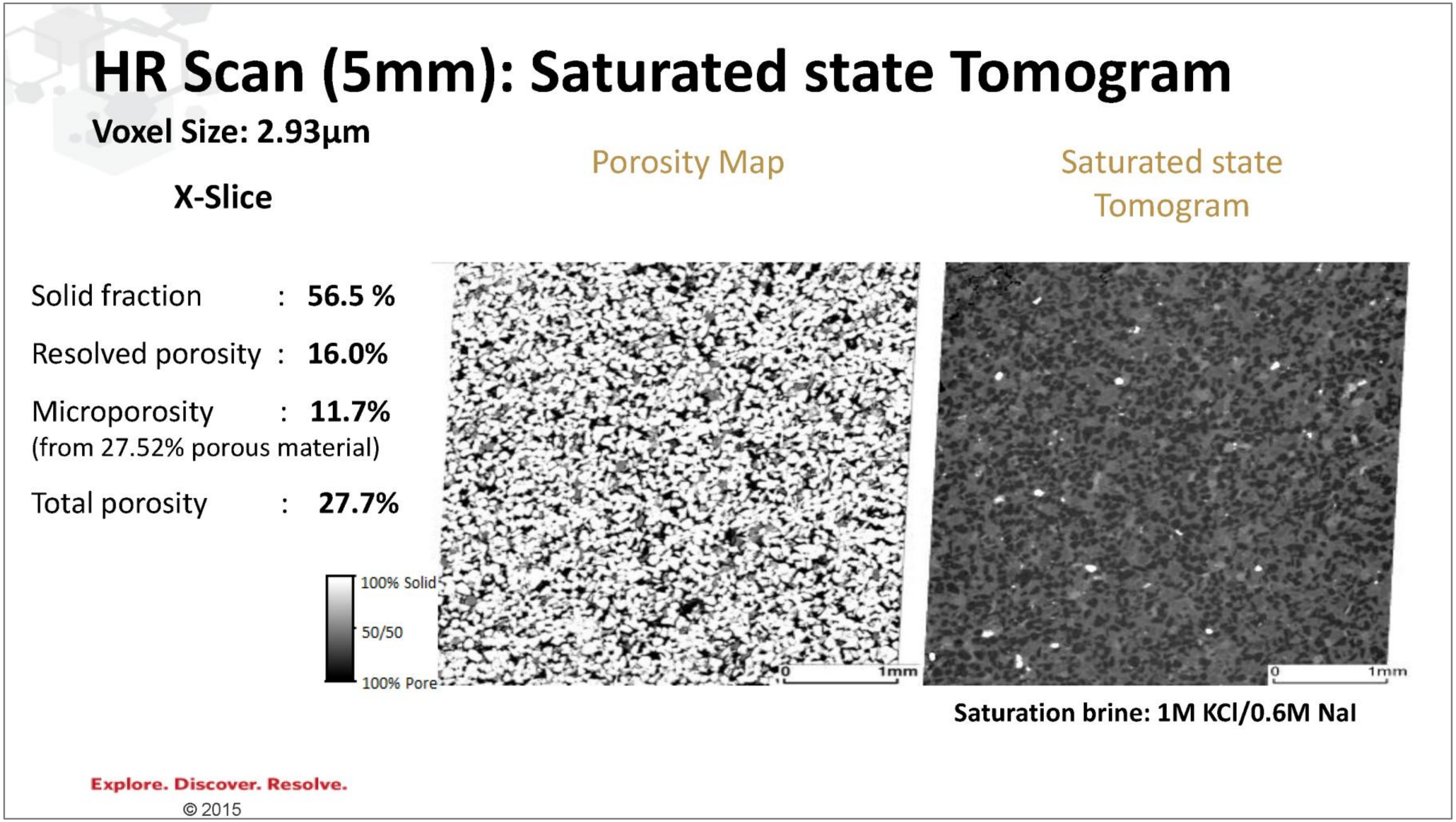


Grain Clusters



The sample is homogeneous in terms of grain size.

Presenter's notes: The individual grains were separated from one another in 3D (each grain has a different colour to each of it's neighbours) and the radius of each grain was calculated and is shown here as a plot of frequency v radius.



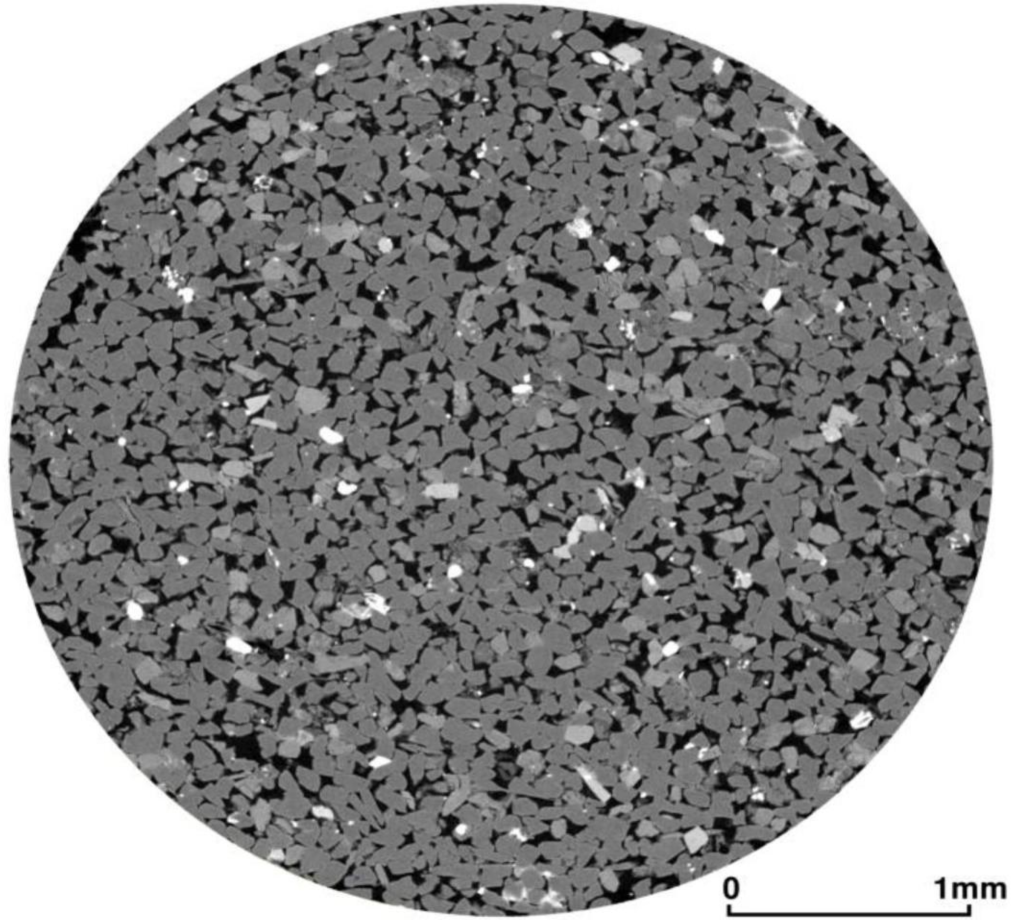
Presenter's notes: The variation in porosity in the sub-plug was calculated with distance down the sub-plug. The red line represents resolved pores, i.e. completely filling voxels, while the black curve represents total effective porosity. The lack of variation down the length of the sub-plug shows that the sample is homogenous in terms of pore distribution.

HR Scan (5mm)

Voxel Size: 2.93µm

Z-Slice

As-received
state
Tomogram



Explore. Discover. Resolve.

Presenter's notes: The sub-plug was cut along the red line, resin embedded a polished section was created for imaging by Scanning Electron Microscope (SEM) to investigate textures and microporosity.

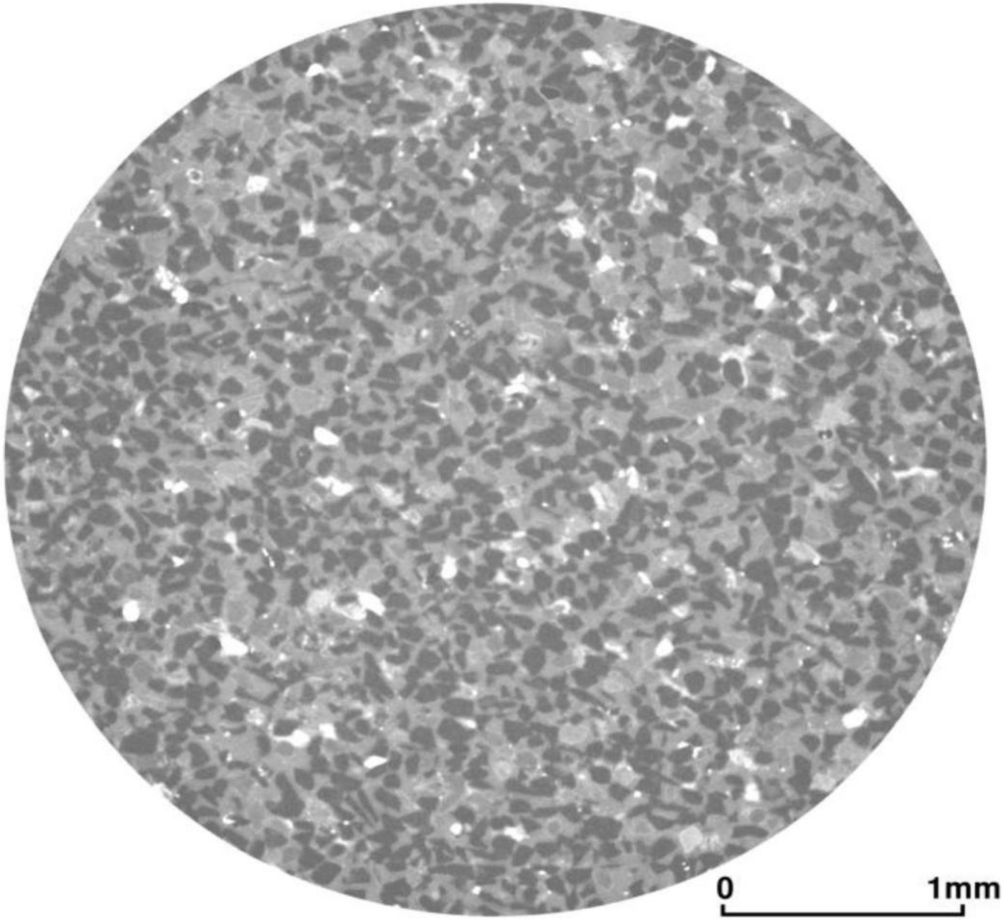
A series of images was acquired and then stitched into a mosaic and the mosaic was registered into perfect geometric alignment with the 3D tomograms.

HR Scan (5mm)

Voxel Size: 2.93μm

Z-Slice

Saturated
state
Tomogram



Explore. Discover. Resolve.

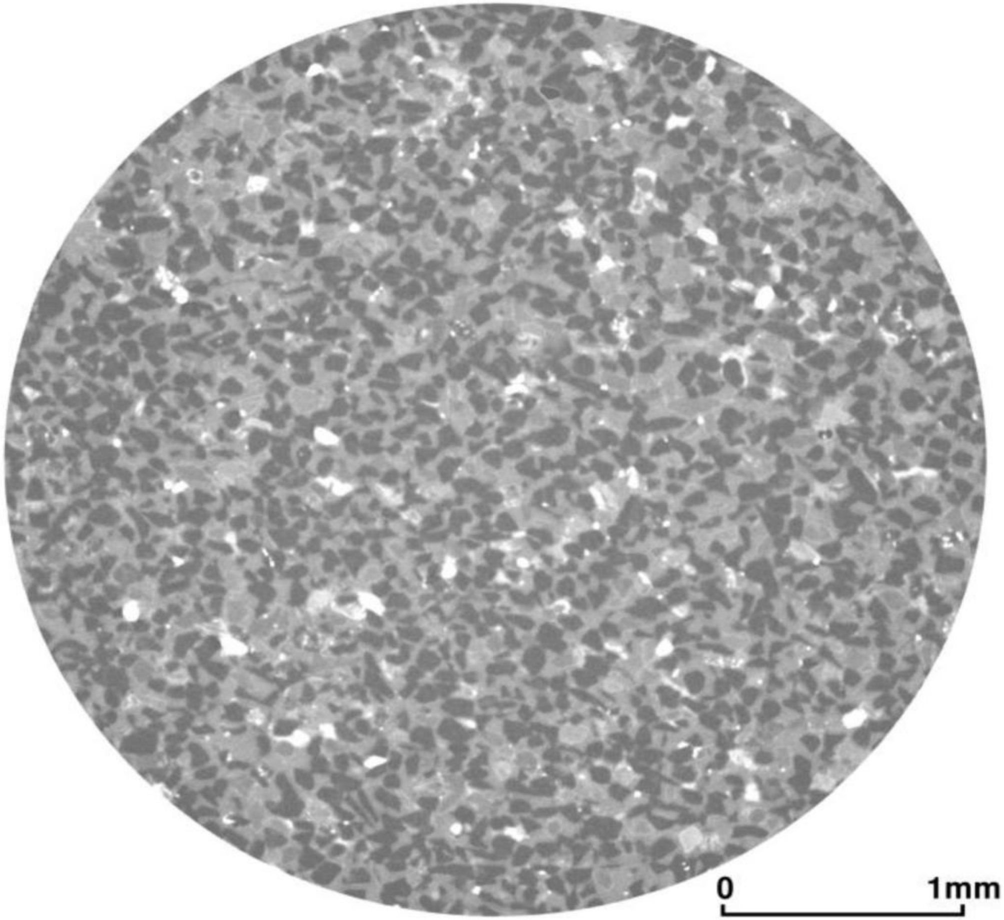
Presenter's notes: This shows one horizontal plane through the 3D tomogram in as-received state

HR Scan (5mm)

Voxel Size: 2.93µm

Z-Slice

Saturated
state
Tomogram



Explore. Discover. Resolve.

Presenter’s notes: This shows the same horizontal plane from the saturated state tomogram after 3D registration

HR Scan (5mm)

Voxel Size: 2.93µm

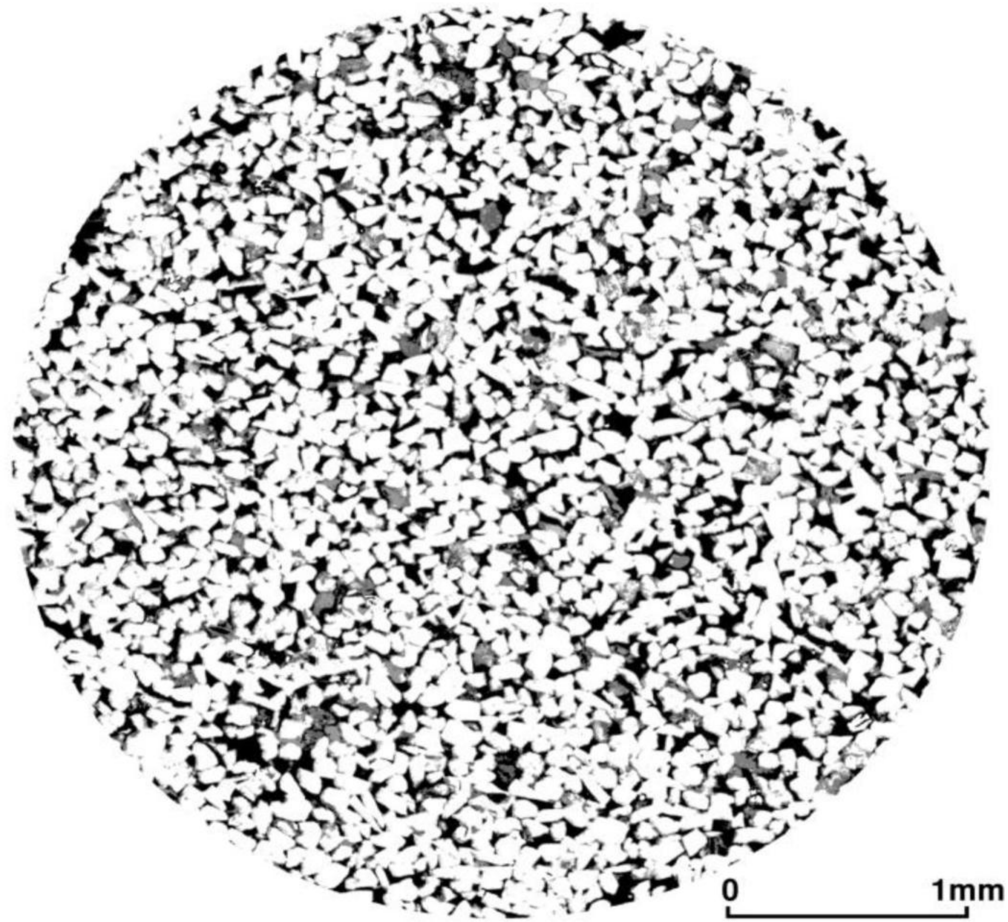
Z-Slice

Porosity Map

100% Solid

50/50

100% Pore



Solid fraction	:	**43.5 %**
Resolved porosity	:	**16.0%**
Microporosity (from 37.52% porous material)	:	**11.7%**
Total porosity	:	**27.7%**

Explore. Discover. Resolve.

Presenter’s notes: This shows the same plane from the porosity map.

HR Scan (5mm)

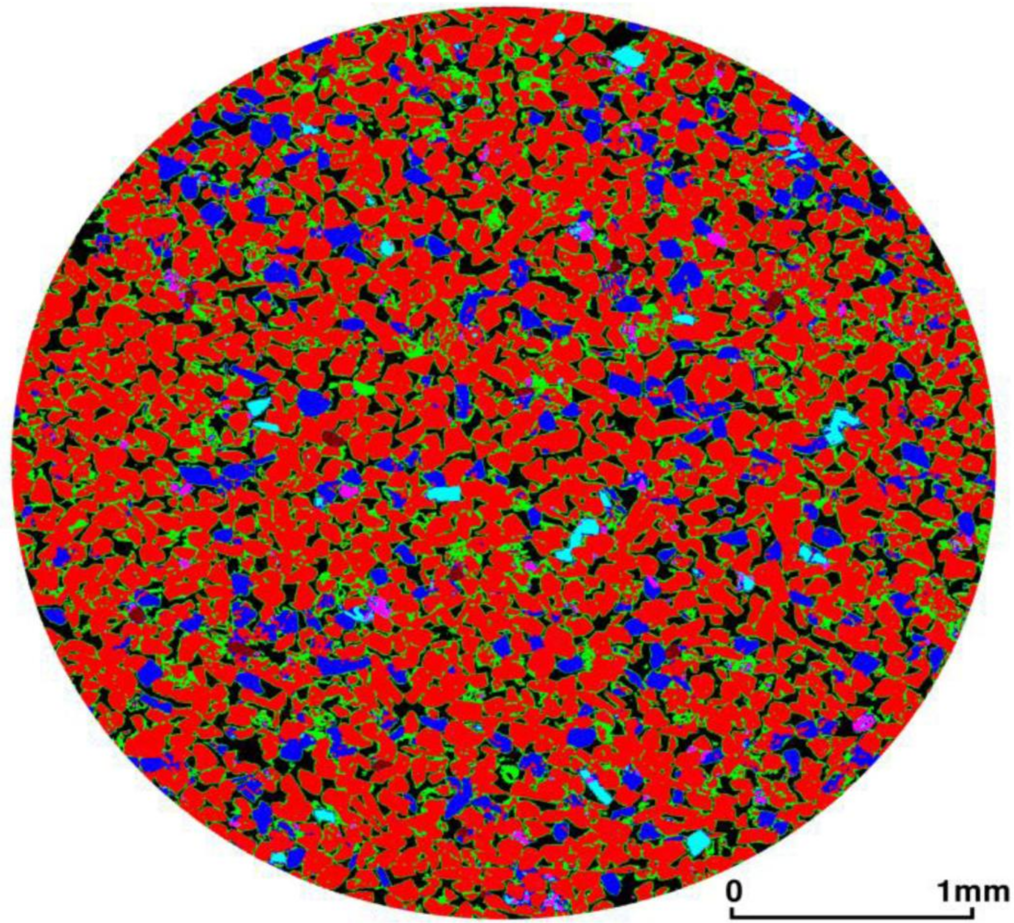
Voxel Size: 2.93µm

Z-Slice

Mineral Segmentation

Phase volume fraction calculation
- results for 3D image:

(■) Resolved Porosity	:	16.8 %
(■) Microporous Phases	:	17.3 %
(■) Quartz	:	56.0 %
(■) K feldspar	:	8.3 %
(■) High Density Phase 1	:	0.8 %
(■) High Density Phase 2	:	0.5 %
(■) Zircon	:	0.2 %



Explore. Discover. Resolve.

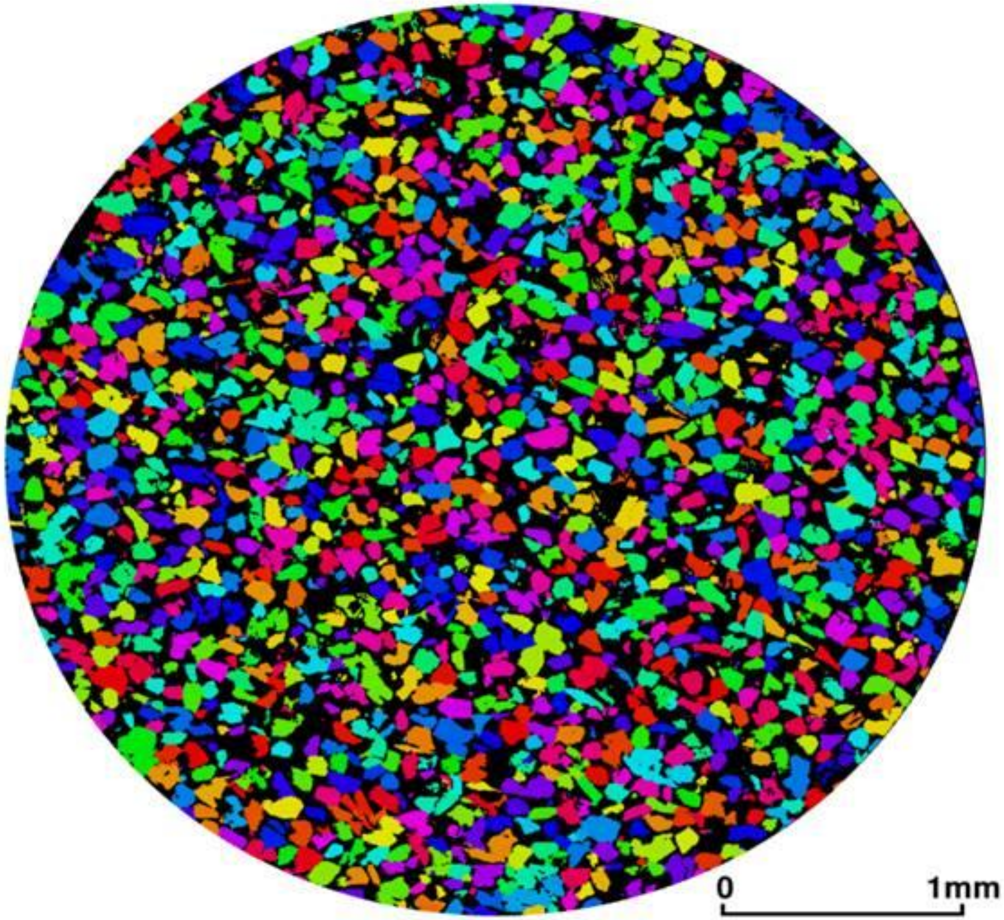
Presenter’s notes: This shows the mineral segmentation, note the grain coatings in green.

HR Scan (5mm)

Voxel Size: 2.93µm

Z-Slice

Grain clusters

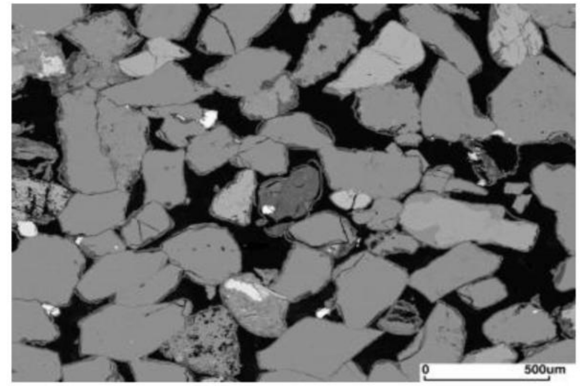


Explore. Discover. Resolve.

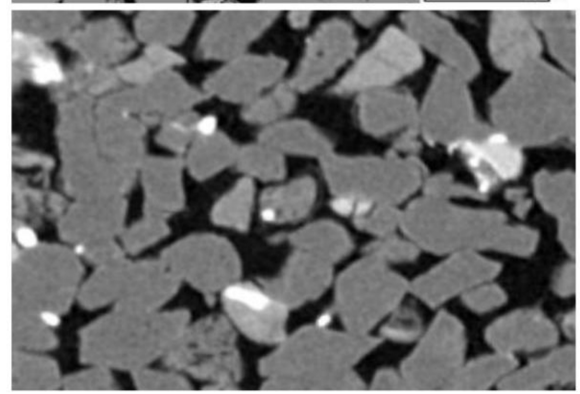
Presenter's notes: This shows the individual grains separated from one another

Extensive Coating of Framework Grains

SEM Image:
Pixel size: 245 nm



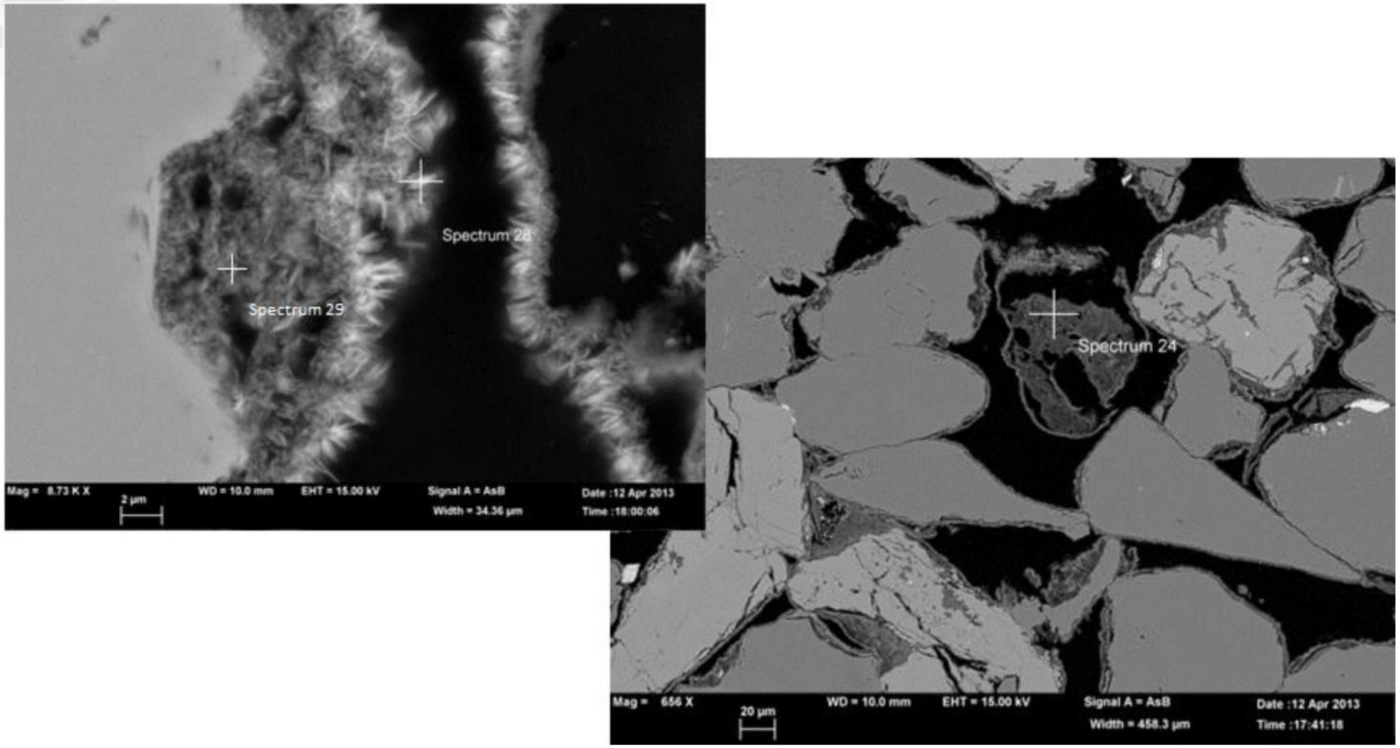
As-received state Tomogram:
Voxel size: 2.93µm



Explore. Discover. Resolve.

Presenter's notes: In the micro-CT images we thought that we could detect grain coatings and by imaging at much higher resolution using BSEM (245nm compared to 2,930nm by micro-CT), we were able to see the grain coatings in great detail and confirm that they are both real and ubiquitous.

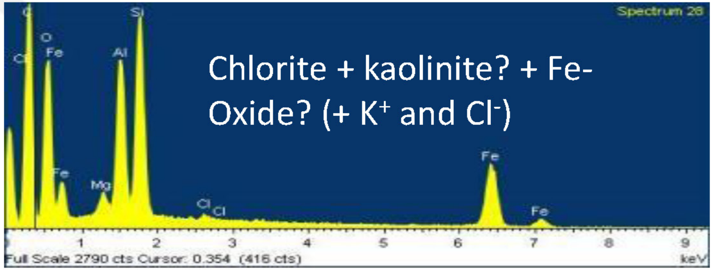
SEM-EDS Analysis on grain coatings and clay pellet

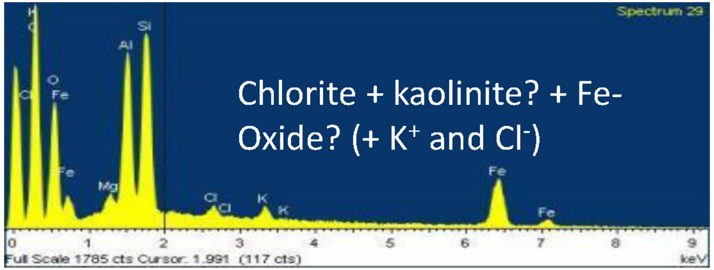


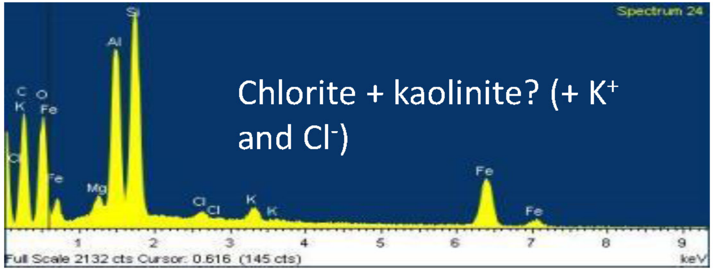
Explore. Discover. Resolve.

Presenter's notes: We performed spot SEM-EDS analysis on the grain coatings to identify them.

SEM-ED Spectra and Analysis







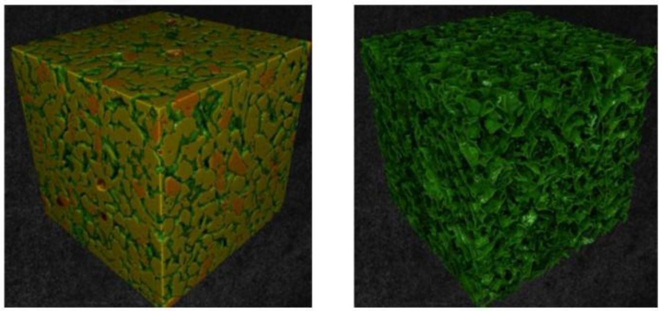
Interpretation:

-The grain coatings have a mixed mineralogy and are highly porous, as indicated by high resolution SEM images

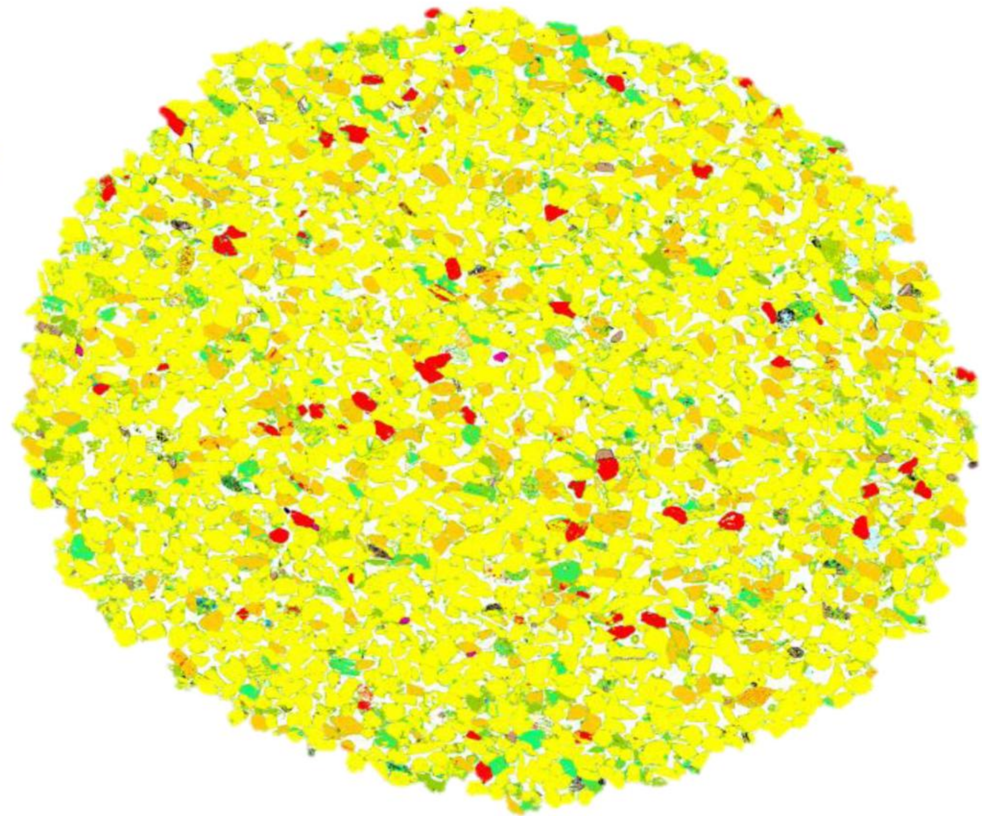
-The fine-grained coatings on the framework grains have inhibited formation of quartz overgrowths and hence allowed preservation of significant intergranular porosity.

HR Scan (5mm)

Voxel Size: 2.93µm Z-Slice



Mineral Map from QEMSCAN



Mineral Name	Area %
Background	22.1
Quartz	66.6
K Feldspar	10.2
Plagioclase	2.3
Siderite	0.1
Calcite	0.2
Kaolinite	0.0
Chlorite_&_ChloriteCoatin	15.8
Illite/Smectite	2.7
Illite/Muscovite	0.6
Biotite	0.0
Pyrite	0.0
Zircon	0.1
Rutile	0.5
Unclassified+traces	0.9

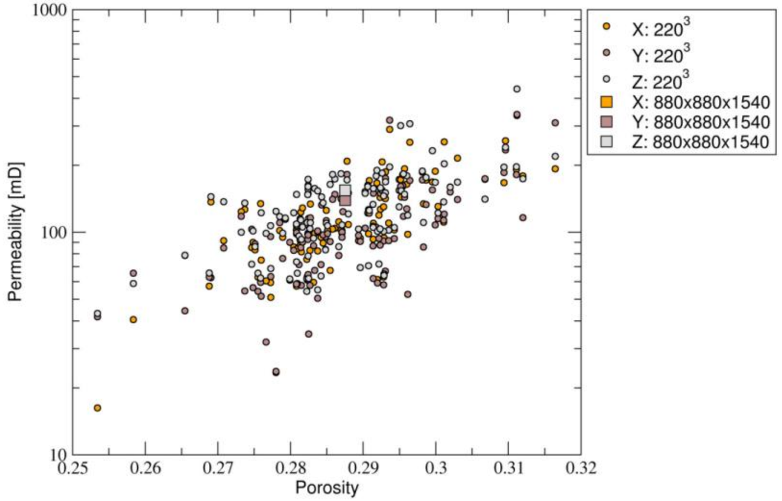
Explore. Discover. Resolve.

Presenter's notes: An automated, quantified SEM-EDS analysis was performed on the same polished section to identify and quantify the mineral composition. QEMSCAN gives quantified mineral distribution with valuable information about the textural relationships between the minerals and pores. This cannot be gained from a bulk mineral analysis like XRD.

Petrophysical Properties

Porosity/Permeability: Simulated v measured

	Simulation				Experiment			
	H_1	H_2	V	Abs.	MICP	Air	Brine	He
ϕ				0.287				0.289
K, mD	144.56	139.55	154.25		151.34	166.00	118.42	



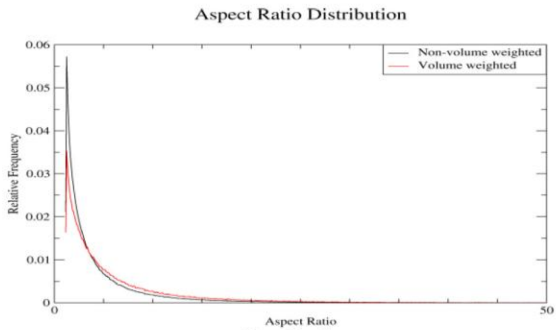
Explore. Discover. Resolve.

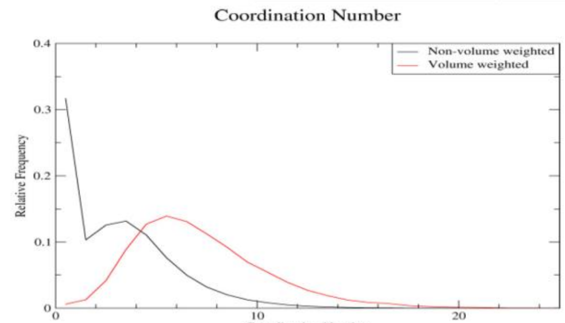
Presenter's notes: The segmented tomogram was used to calculate the porosity, absolute permeability in 3 orthogonal directions, FRF and m. These are shown with RCA data from the same plug.

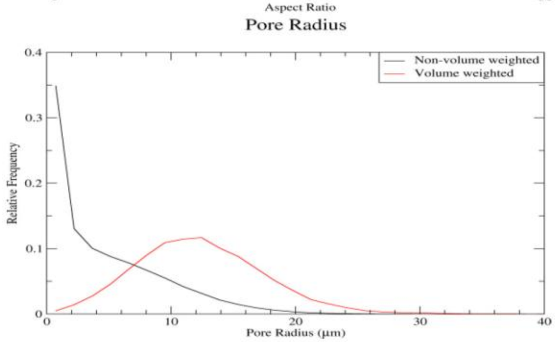
Plot of porosity versus permeability. The Z direction applies to the axis along the sub-plug axis and will usually correspond to the vertical orientation. X and Y directions are orthogonal to Z and will often correspond to horizontal orientations. The values indicated in the legends refer to the number of voxels in the volume of data/rock represented by that data point.

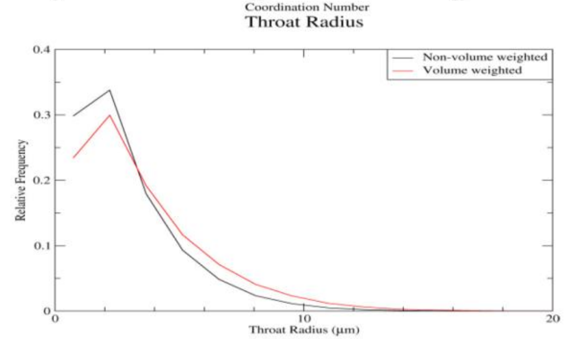
Pore and throat statistics

	Non-Volume weighted mean ± s.d.	Volume weighted mean ± s.d.
Aspect ratio	4.6 ± 4.8	6.1 ± 6.6
Coordination number	2.6 ± 2.7	6.7 ± 3.4
Pore radius	4.7 ± 4.6	12.4 ± 5.3
Throat radius	2.8 ± 2.1	3.4 ± 2.6





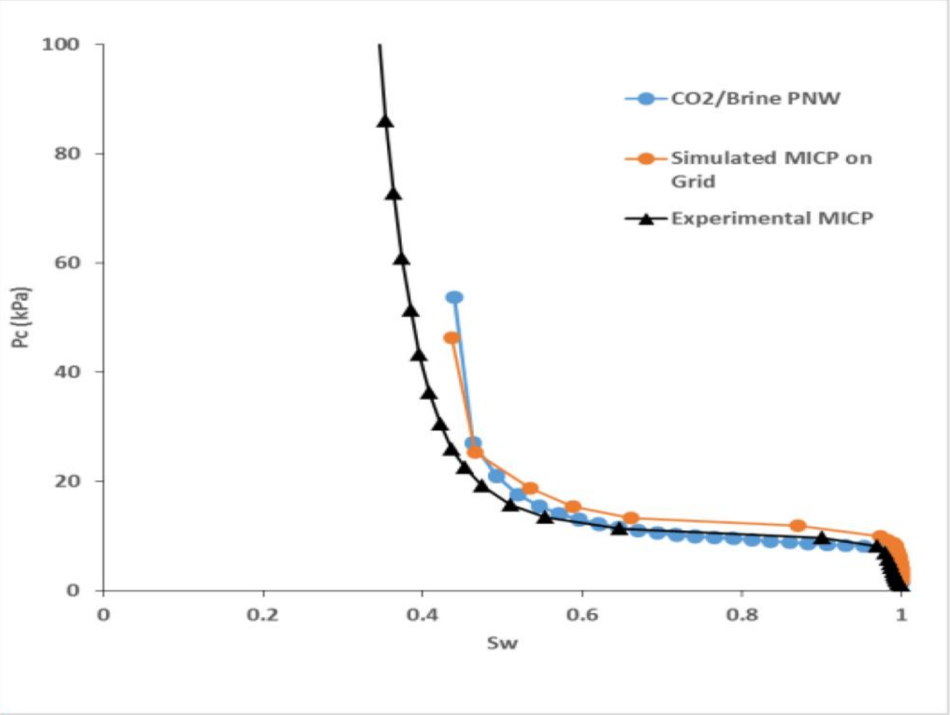




Explore. Discover. Resolve.

Presenter's notes: The pore network was extracted from the 3D tomogram and the pore and throat statistics were calculated. These are shown in volume weighted and non-volume weighted distributions.

Pc (Sw) curves from MICP experiment and two different models



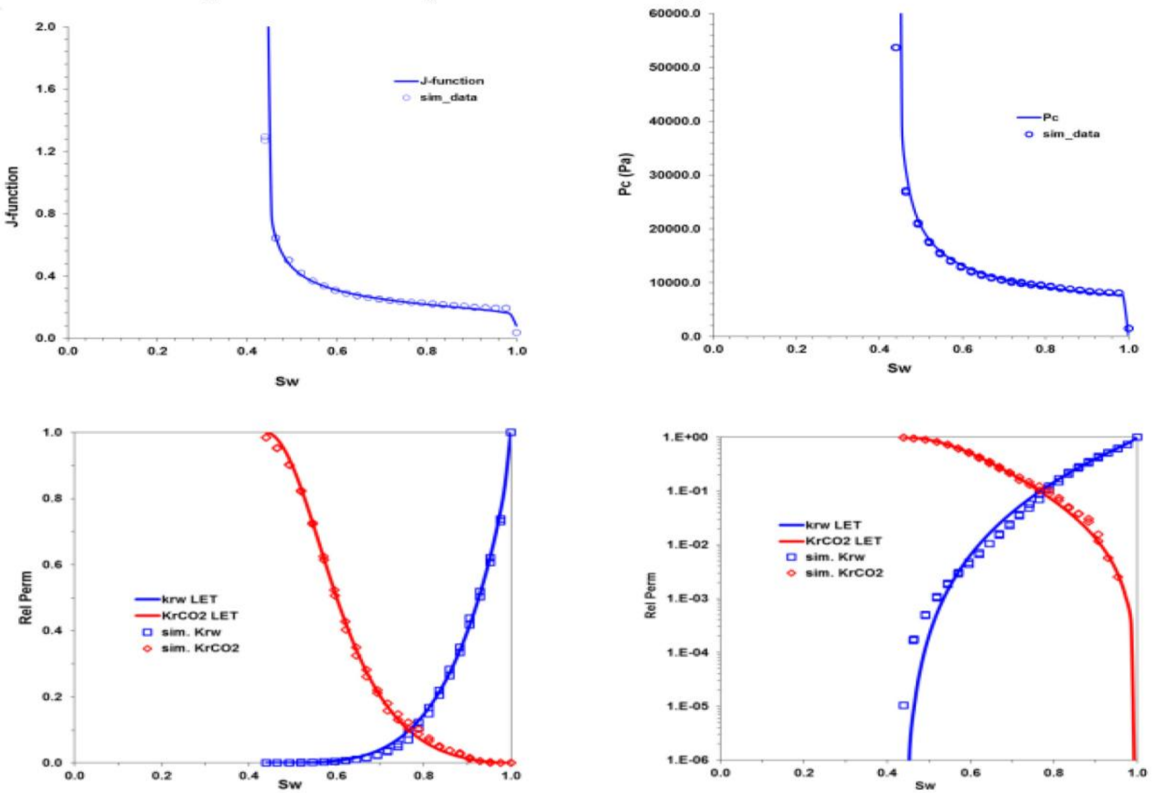
Explore. Discover. Resolve.

Presenter's notes: A grid was created on the 3D micro-CT tomogram and MICP was simulated on that grid (orange curve). A pore-network model was extracted from the image and CO2/brine drainage was simulated then imbibition. The blue curve shows the Pc v Sw behaviour. The simulation was not run to as high pressure as the experimental MICP (black curve), hence the black curve extends higher on the plot. *(Presenter's notes continued on next slide)*

(Presenter's notes continued from previous slide)

CO2/Brine PNW – Pore Network with Drainage CO2/Brine (Density and IFT represents those two fluids)
The numerical model is capturing the pore structure in the mid-high water saturation region.
The mismatch in the low water saturation indicates that we have some challenges with capturing/allocating the clay portion

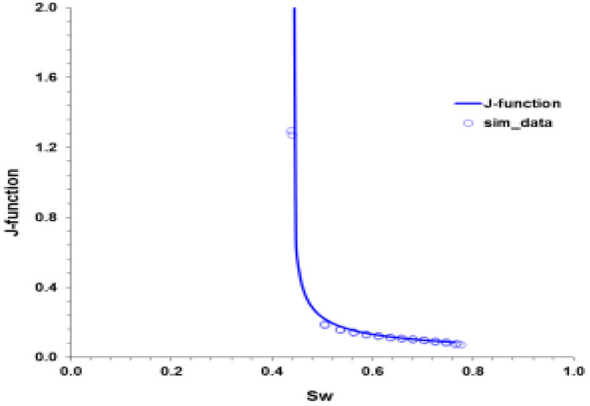
CO_2/brine primary drainage

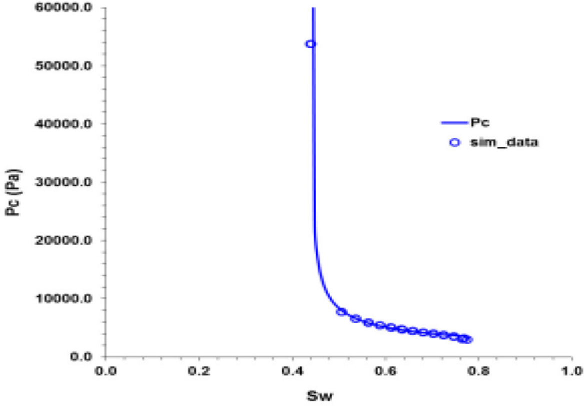


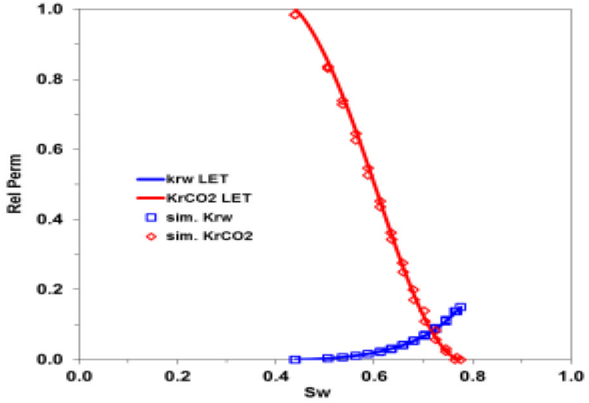
Explore. Discover. Resolve.

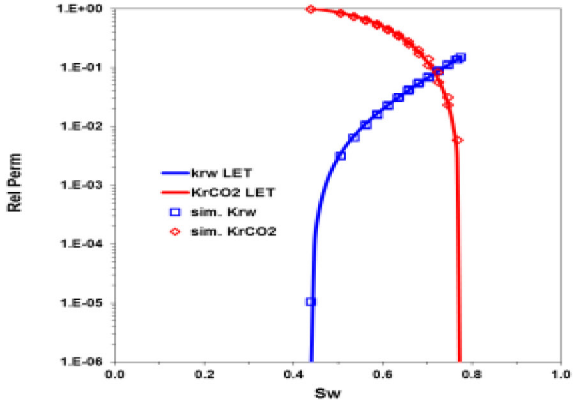
Presenter's notes: The squares and diamonds are points from the simulation while the curves are from fitting with the LET method. The trapping of CO2 is in the range with what is observed in the literature / experiments.

CO_2/brine imbibition









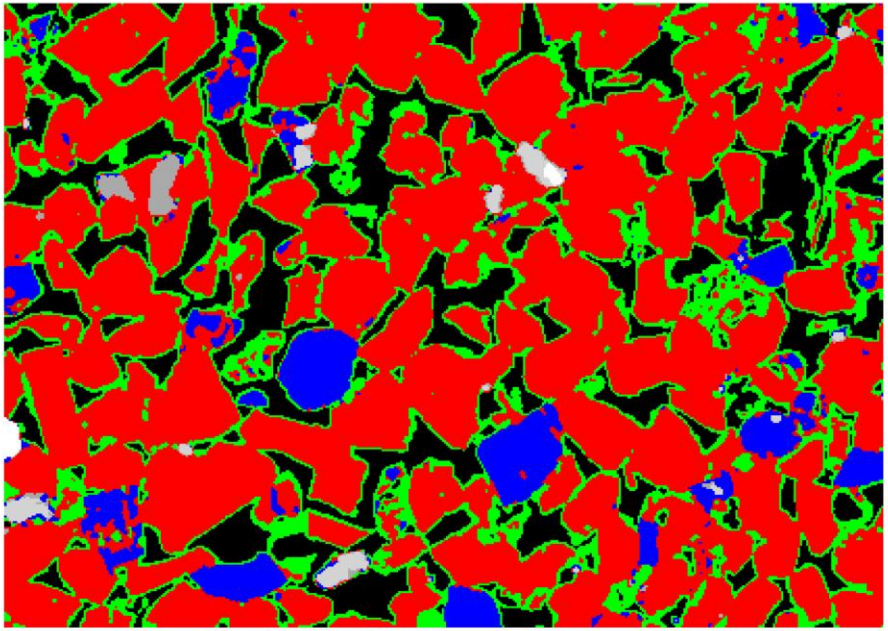
Elastic response

HR Scan (5mm)

Voxel Size: 2.93μm

X-Slice

Mineral Segmentation



Phase volume fraction calculation
- results for 3D image:

( ) Resolved Porosity	:	16.8 %
( ) Microporous Phases	:	17.3 %
( ) Quartz	:	56.0 %
( ) K feldspar	:	8.3 %
( ) High Density Phase 1	:	0.8 %
( ) High Density Phase 2	:	0.5 %
( ) Zircon	:	0.2 %

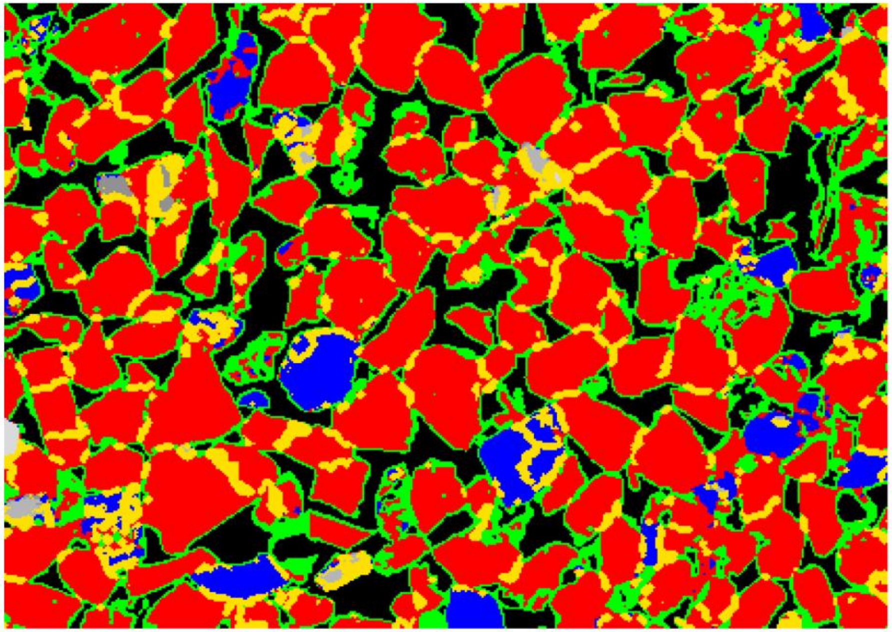
Presenter's notes: The elastic properties from the lab indicate that the sandstone is slightly weakened by the grain coatings

HR Scan (5mm)

Voxel Size: 2.93µm

X-Slice

Mineral Segmentation with Grain Contacts



( ) Resolved Porosity
( ) Microporous Phases
( ) Quartz
( ) K feldspar
( ) High Density Phase 1
( ) High Density Phase 2
( ) High Density Phase 3
( ) Grain Contact

Explore. Discover. Resolve.

Presenter's notes: For the simulation of elastic properties it is essential to accurately characterize the grain contacts and the minerals. Moduli for each mineral are then applied to the model before running the simulation.

HR Scan (5mm)

Voxel Size: 2.93µm

	Hard grain contact	Soft grain contact (2 voxels thick)
Bulk Modulus	9.63 GPa	8.93 GPa
Shear Modulus	8.26 GPa	6.81 GPa
Young Modulus	19.27 GPa	16.29 GPa
Poisson ratio	0.166	0.1959
Vp	3.20 Km/s	2.85 Km/s
Vs	2.02 Km/s	1.75 Km/s
Vp/Vs	1.5814	1.6262

Measured Vp : 2.818 Km/s

Explore. Discover. Resolve.

Presenter's notes: Two separate simulations were run, one with hard grain contacts (left) and one with soft grain contacts, to emphasize the impact of the grain coatings on the elastic response. The experimentally measured Vp is shown at the bottom of the slide and it can be seen that with hard GC the simulated Vp is too high but with soft GC the simulated Vp is similar to the experimentally measured Vp.

NMR response

NMR T_2

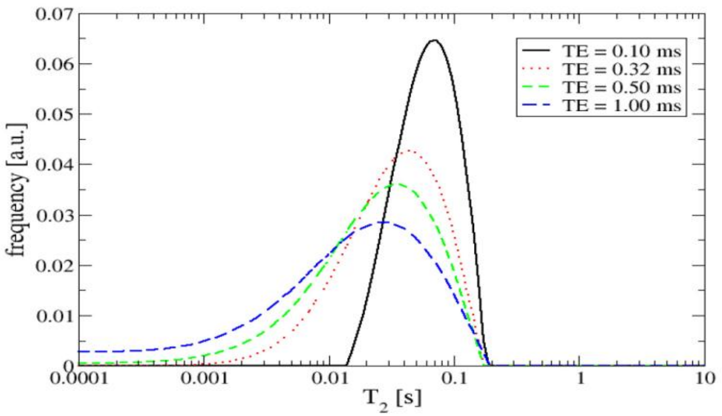
T_2 relaxation

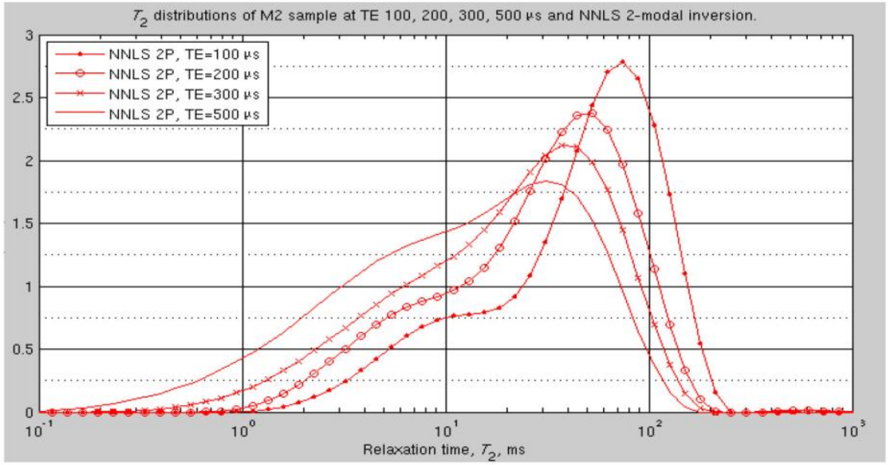
- Since the presence of iron in the coating of the framework grains causes considerable uncertainty in the interpretation of NMR response, a number of different scenarios were considered
- To understand the sensitivity of the transverse relaxation response to the iron coating, the response was modelled with a set of different echo spacings, TE = 0.1ms, TE = 0.32ms (NMR logging), TE = 0.5ms, TE = 1.0ms.
- The magnetic susceptibility of the iron-chlorite coating was varied within values consistent with literature, ranging from low values (χ = 40mSi) to high values (χ = 4000mSi).

Explore. Discover. Resolve.

Presenter's notes: NMR T2 response was simulated at different echo spacings to understand the impact of the Fe-bearing chlorite in the grain coatings. Different values of magnetic susceptibility were also tested for the Fe-chlorite.

NMR T_2: Simulation v Experimental

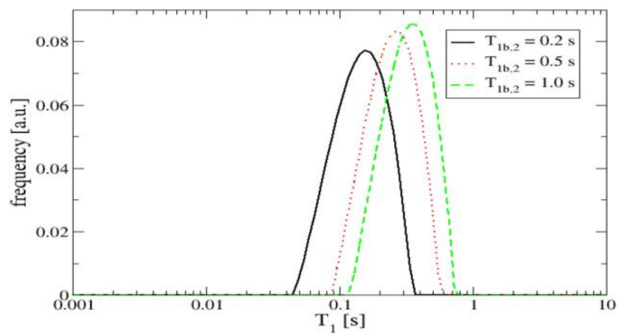


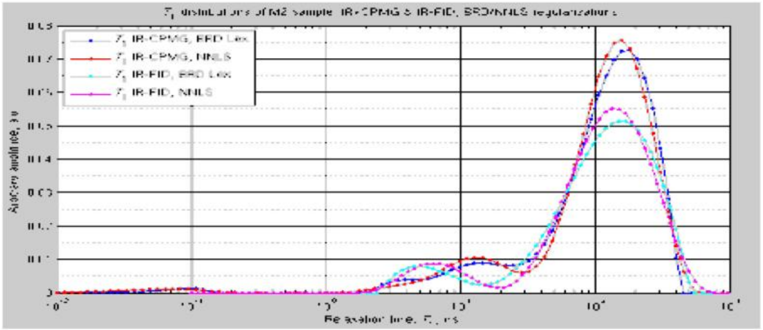


Explore. Discover. Resolve.

Presenter's notes: On the right is a plot data from NMR T2 experiments at different echo spacings. On the left is a plot of simulated NMR T2 response showing a good match to the experiments.
The experimental curves have second peaks that were not characterized in the simulation due to the limitations of image resolution not capturing all sub-micron features.

NMR T_1: Simulation v Experimental





Presenter's notes: NMR T1 response was also measured (right) and simulated (left) and shows a good match.
The experimental curves have long tails that were not characterized in the simulation due to the limitations of image resolution not capturing all sub-micron features.

The simulations(left) of the T1 response reveals little structure due to the choice of simulation parameters and the discretization/segmentation of the sample. In particular, it is assumed that the surface relaxivity is constant, while the microstructure of the chloritic phase is described by an effective T1 relaxation rate. The use of a T1 relaxation time of 0.5s in the simulations seems to be consistent with the main features of the experimentally(right) observed T1 NMR response.

Conclusions

- The digital technique successfully simulated the petrophysical properties including primary drainage and imbibition to CO_2/brine, elastic response and NMR response and in all cases the simulations match the experimental results for the same plug
- The use of digital rock analysis revealed unique insights into the grain coatings on this Jurassic sandstone and their contribution to porosity and effect on properties such as elastic and NMR responses.