PORE SCALE CHARACTERIZATION OF CARBONATES AT MULTIPLE SCALES: INTEGRATION OF MICRO-CT, BSEM AND FIBSEM

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This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Noordwijk, The Netherlands 27-30 September, 2009

ABSTRACT
The understanding of petrophysical and multiphase flow properties is essential for the assessment and exploitation of hydrocarbon reserves; these properties in turn are dependent on the 3D geometric and connectivity properties of the pore space. The determination of the pore size distribution in carbonate rocks remains challenging; extreme variability in carbonate depositional environments and susceptibility to a range of post-depositional processes results in complex pore structures comprising length scales from tens of nanometers to several centimeters. To increase understanding of the role of pore structure on connectivity, conductivity, permeability and recoveries requires one to probe the pore scale structure in carbonates in a continuous range across over seven decades of length scales (from 10 nm to 10 cm) and to *integrate* information at these different scales.

In this paper experimental techniques including micro-computed tomography, backscattered scanning electron microscopy (BSEM), and Focussed ion beam SEM (FIBSEM) are used to probe the pore scale structure in carbonates across many decades of scale. Registration techniques are then used to couple information at different length scales. First an image of a 3D plug (4 cm, 20 micron voxel size) is correlated to a sample at macroporous resolutions (8mm diameter, 4 micron voxel size). We then focus on coupling SEM and FIBSEM data at submicron resolutions to micro-CT data at ~3-5 micron resolution. For pixel perfect registration of SEM images, an accurate template has been developed to remove warp artefacts introduced by the SEM scanning procedure and we have successfully mapped the sub-resolution porosity and pore sizes visible in the SEM image to gray scale levels in the 3D image. FIBSEM also allows one to investigate the 3D structure in samples down to tens of nanometers. We briefly discuss how this multiscale information can be used as a method for enhanced analysis of petrophysical properties of carbonates.

INTRODUCTION
The structure and dynamics of carbonate reservoirs are of crucial importance to the oil and gas industry as carbonate reservoirs are believed to contain more than half the world’s oil. Predicting petrophysical and multiphase flow properties in carbonates is challenging due to the nature of the deposition and diagenesis which results in pore scale features and heterogeneity at multiple length scales. To better understand the properties of carbonates information on the range of pore scales must be obtained; in principle, at scales from tens of nanometers to metres (Biswal et al., 2007).

In this paper we describe the ongoing development and integration of a range of experimental and computational tools which can assist one in probing the pore scale structure of carbonates across many decades of scale in an integrated fashion. We focus on integrating geological heterogeneity from the 4 centimetre (plug) scale to the 100 nanometer scale.

METHODS

This section describes the 3D tomographic, the 2D BSEM and the 3D FIBSEM experiments, as well as the computational procedures used to analyse the data.

Experimental procedure

Plug samples of carbonate core were received in the laboratory. The full plugs (usually 1 ½ inch or 38 mm diameter) were initially imaged at a resolution of ~20 microns. Subsample(s) of the plug were then cored with diameters ranging from 3 to 5mm. These were imaged at voxel sizes of 1.5-2.5 microns. After 3D imaging the subsamples were cut and a thin section was prepared from within the field of view of the 3D images. The thin sections were then imaged using a backscattered scanning electron (BSEM) microscope. This allowed the resulting 2D SEM image to be positioned (registered) into the 3D tomogram. This allows a direct comparison of the corresponding slice from the 3D tomographic image and the higher resolution SEM images. FIBSEM images were also obtained from the thin section at voxel resolutions of ~50 nm.

Micro-CT: Most 3D images of the cores were obtained using the ANU micro-CT facility [Sakellariou, 2004] which with sample sizes of 4 cm – 3 mm and a detector size of 2048^2 resulted in images with voxel sizes of ~20 - 1.5 microns respectively.

2D Backscattered Scanning Electron Microscopy: All 2D BSEM images were taken on the Hitachi 4300 SE/N (Schottky Field Emission SEM, 2006).

Focused Ion Beam Scanning Electron Microscopy (FIBSEM): FIBSEM Tomography datasets [Tomutsa, 2003] are produced using both the ion and electron beams to perform serial slicing and imaging of material cross-sections respectively at submicron resolutions. The experiments were performed on a Nova Nanolab located at the University of New South Wales. The samples were taken from the thin sections previously used to generate the SEM images. For sample preparation and operational details, see [Ghous 2009].

Computational Methods

Filtering, Segmentation and Microporosity Assignment in μ-CT images: The 2000^3 tomographic images, obtained at ~2 micron voxel size (Fig 1a), were filtered and segmented into three phases using the technique of converging active contours [Sheppard, 2004]. The three resulting phases represent the resolvable voidspace, the meso/microporosity that is at scales below the resolution of the CT image and the solid region respectively (Fig 1b and e). The resolvable areas are designated as macroporous (resulting in a macropore size cut-off of around 5-10 microns) and the unresolvable porosity is labelled here as *microporosity.* The microporous phase was subsequently divided into a number of separate phases based on their relative attenuation (Fig 1c and 1f). Voxels defined as microporous are assigned a microporosity value (ϕ_{micro}) between 100% and 0% based on a linear interpolation of the attenuation between the maximal

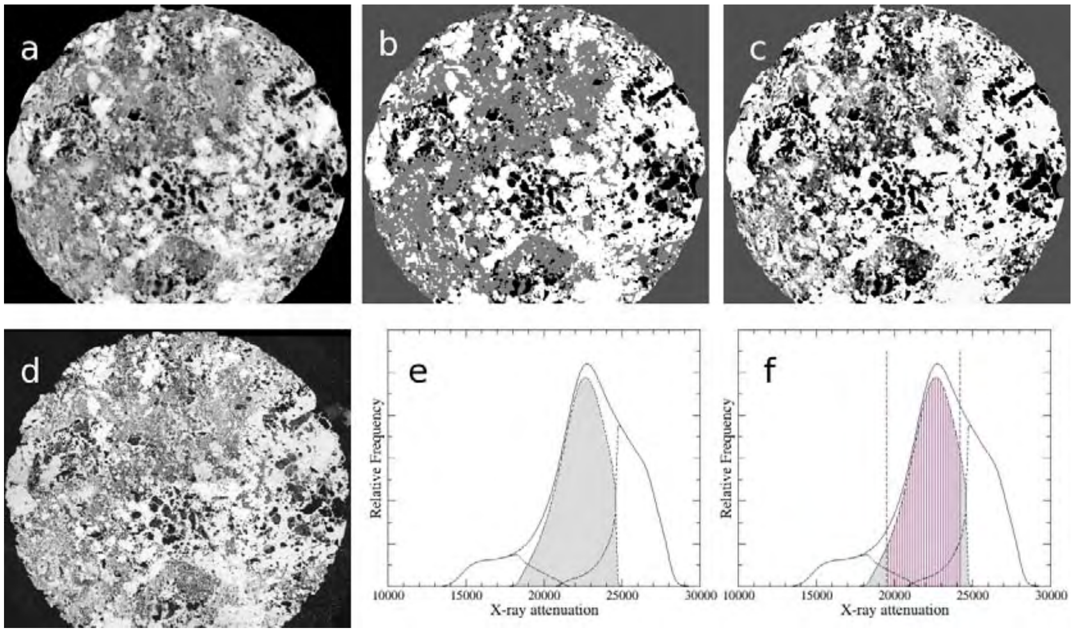

Figure 1. (a) Tomogram of a typical carbonate. (b) Three phase segmentation of the tomogram, black is macroporosity, grey is microporous phase, white is solid. (c) Microporosity assignment, the microporous regions is divided into 100 bins and assigned a microporosity value between 0 and 100%, indicated by greyscale. (d) An SEM image of the same sample (images a-c are the registered tomographic equivalents). (e) X-ray attenuation histogram of the three phase segmentation. (f) X-ray attenuation histogram with microporous regions indicated. The voxels in the red region are given microporosity values ϕ_{micro} based on attenuation.

value of the identified macroporous phase and minimum value of the solid phase; in Fig. 1(c) these cutoffs are 19500 and 24150 respectively.

Segmentation of the 2D FIBSEM samples: FIBSEM images were segmented into two phases: pore and solid. These were done either using the previous method or in some cases, due to local intensity variations in the different slices because of charge accumulation, a semi automatic segmentation procedure was followed in which the operator had to pick seed regions for local thresholding of pore and solid phases (Ghous, 2009).

Dewarping of SEM images: Some microscopes (particularly when scanning fields of view of mm to cm) introduce geometric distortion into an image; this is problematic when creating a mosaic of images, or in this case, when wanting to register or align a mosaic with a slice of a micro-CT image. This geometric distortion needs to be removed before accurate quantitative information can be inferred; before two images can be correctly registered in order to properly fuse their information. This process of removing such distortions from the images is referred to as de-warping [Varslot, in preparation]. The de-warping process consists of two stages. Initially, a warp transformation is determined which is associated with the observed distortions in the instrument. This involves taking an image of a regular array; in our case we use a 5 x 5 mm matrix of 10 micron chromium disks on an optically flat soda glass wafer, spaced 50 microns apart. The disk centres are correct to 25 nm precision. Then, the inverse of the warp transformation is applied to the image to produce a geometrically corrected

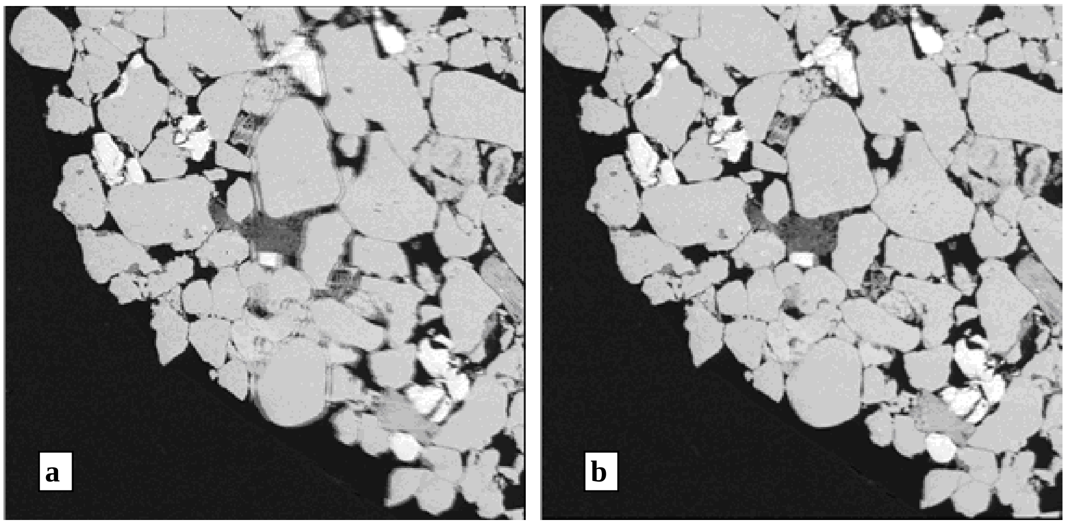

Fig. 2: Two SEMs stitched together. (a) registered without dewarping (b) registered after being dewarped.

image. In Fig. 2 we illustrate the effect that applying the process of dewarping has on the quality of an integrated image. Fig. 2(a) shows the direct stitching of two 2D SEM images at high resolution (~1 micron); the field of view is ~3mm x 3mm. Fig. 2(b) shows the merged image after applying dewarping. The quality of the image is significantly improved in (b).

Registration procedure: The registration procedure is documented in [Latham et al., 2008] but in brief the principles are as follows. We optimise the 'similarity' of the two images (two 2D images, one 2D and one 3D image, or two 3D images), as a function of the translation, rotation and scaling parameters. The similarity measure used depends on the type of data that is to be registered.

RESULTS

Plug to pore scale (~2 micron) registration in 3D

In this first results section we describe the example of correlating pore structure in a heterogeneous sample at a plug scale to the intergranular macropore scale. A 3D image represented by the slice in Fig. 3(a) is obtained at a field of view of 4 cm diameter at 20 micron voxel size. This allows features of the material between 20 microns and 4 cm to be discerned. However to probe features at higher resolutions, one or more subsets of the sample originally imaged at the scale of 4 cm have to be reimaged at a smaller sample volume. Fig. 3(b) shows one such example and the spatial position of the image within the original plug. This image, a subset of the original image, has been obtained at a resolution of 2.5 microns. Features can now be identified at an improved resolution; approximately 8 times better than the original resolution. The information obtained from these two images can be integrated by directly comparing a slice from the 3D image data at the two resolutions; see Fig. 3(c) and (d). The grey scale attenuation information in the lower resolution image can be directly correlated into the higher resolution information. Mapping of attenuation at the larger scale to both pore size and porosity from the higher resolution image is undertaken.

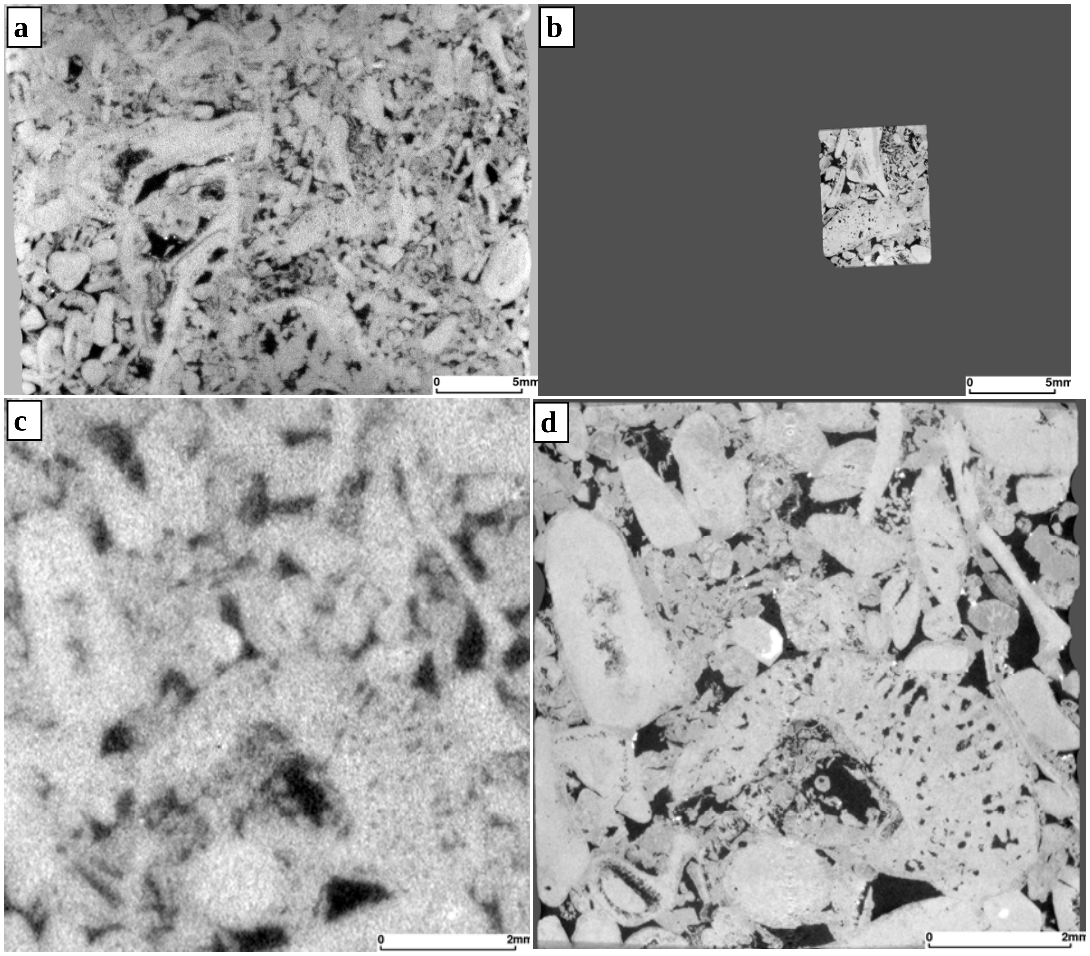

Fig. 3: Example of the alignment of two 3D images at different resolutions. (a) shows an image of a slice from a tomogram of a carbonate plug obtained at the full plug scale (20 microns per voxel). (b) shows the spatial position of the subset of the 5mm subset of the plug now imaged at 2.5 microns per voxel. Bottom row shows slices from the same region of the carbonate imaged at (c) low and (d) higher resolutions.

Pore scale correlation to submicron scale: Registration of 3D to 2D SEM images

In Fig. 4(a) we show a schematic concept of the registration of a 2D SEM slice to a 3D micro-CT image described previously by Latham et al., 2008. In registering a slice one undertakes all the translation, rotation, warping and scaling transformations required to couple the SEM data to the slice of a 3D tomographic data set. In Fig. 4(b) we illustrate the concept of mapping the image data from a high resolution 2D SEM to a slice of the tomogram. Generally tomographic data has an inherent resolution of ~1 micron while microscopy allows one to probe scales down to nanometers. Figure 4(b) illustrates the coupling of 250 nm resolution SEM data to 2.85 micron micro-CT slice from a 3D data set. Microporous features (e.g., porosity at submicron scales, pore size) from the higher resolution SEM can be mapped onto the corresponding slice of the tomogram. From this mapping features one can then stochastically propagate this higher resolution information throughout the 3D volumetric tomographic data.

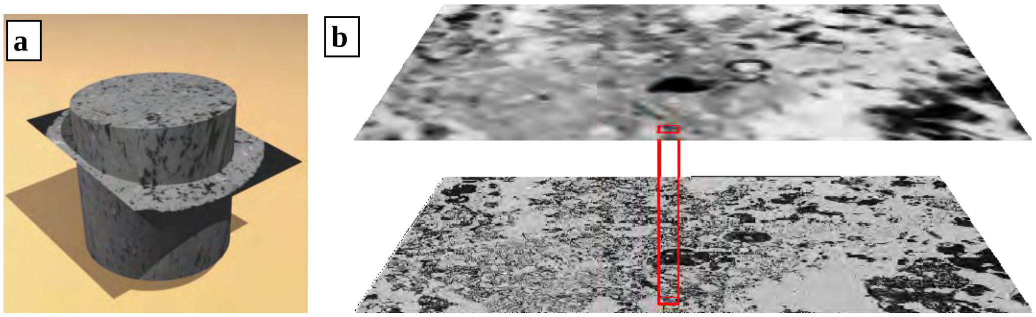

Fig. 4: (a) Schematic of 2D SEM to 3D micro-CT registration; (b) example of the mapping of the microporosity from a registered slice of a 3D CT image (2.85 microns voxels; upper region) to the higher resolution information (0.25 micron pixels) in the SEM image (lower region).

Correlation of microporosity to attenuation: In Fig. 5 we show close up images of the sample shown in Fig. 1 and 4(b). The slice of the x-ray CT image obtained at 2.85 microns per voxel correlates well to the SEM image obtained at 1.2 micron pixel size. Upon closer inspection we see two small problems that are cause by the preparation of the thin section. There are areas where small bits of rock have been removed by the polishing process (plucking; given by square in Fig. 5) and some areas where the pores have been filled in with milling debris (circled in Fig.5). Due to this disturbance at the polished image surfaces we have started to take SEMs from polished stubs instead of polishing down to thin sections.

In Fig. 6 we compare the tomographic X-ray attenuation to the intensity of the SEM image (at both lower (pixel size 1.24 micron) and higher (pixel size 0.25 micron) resolution). In the region of the intermediate attenuation, the "microporous" phase, indicated in the image as shaded grey, we see a good linear correlation. For the higher and lower X-ray attenuations we see a deviation and this may related to the higher dynamic range of the micro-CT detector and the problems mentioned previously regarding the polishing process. In Fig. 7 we show another example of mapping between a carbonate slice in 3D and an SEM; here the resolutions are 2.1 microns per voxel and 0.25 microns per pixel respectively. In this case little disturbance of the sample was noted and the correlation between x-ray attenuation and porosity in the SEM is good.

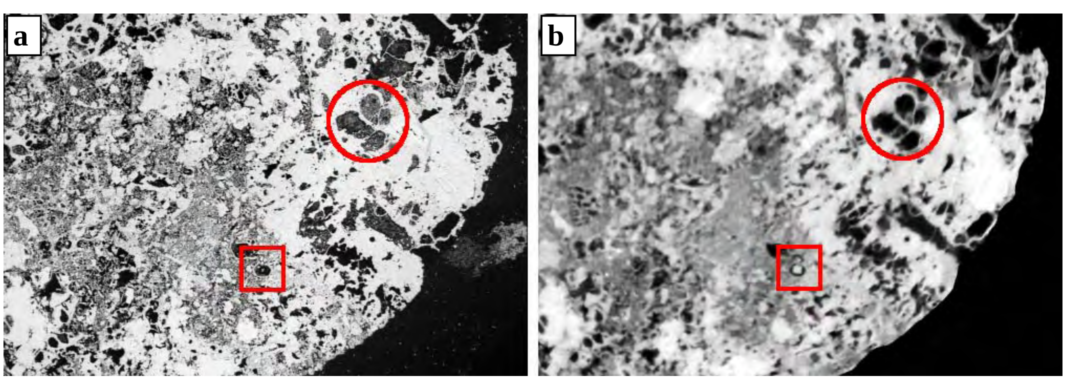

Fig. 5: (a) SEM image at (1.24 micron pixel size). (b) The registered slice of micro-CT data. The circle in the images show a region where milling debris has accumulated in the macro pores after polishing and the square shows a region where a dolomite crystal was plucked from the thin section. The overall image size is 3.1 mm x 2.4 mm for both images.

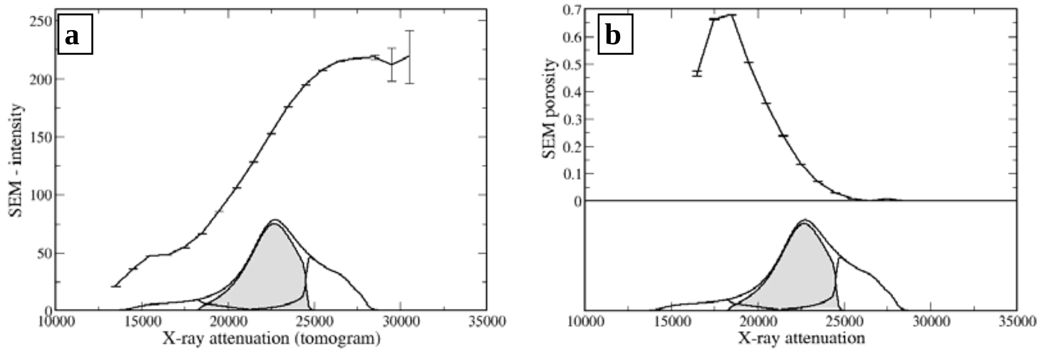

Fig. 6: (a) SEM intensity (low resolution image,1.24 micron pixel size) as a function of X-ray attenuation of the tomogram. (b) SEM average porosity (higher resolution image, 0.25 micron pixel size) as a function of the X-ray attenuation of the tomogram. The x-ray attenuation histogram in Fig. 1(e-f) is also shown in the image. In both images we have also shown the X-ray intensity distribution. The region defined as microporous in the 3D tomographic image correlates to regions of porosity in the SEM.

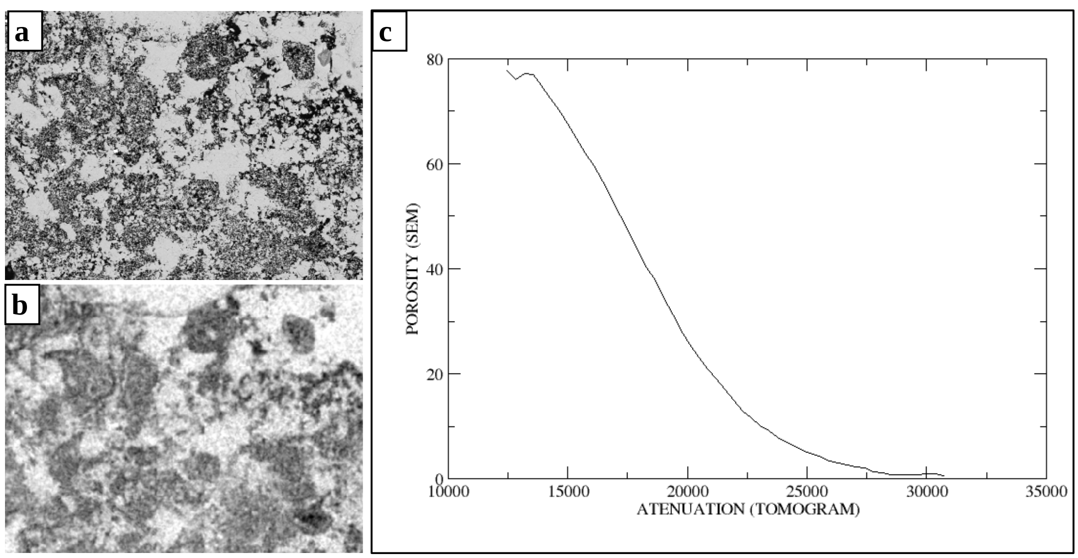

Fig. 7: (a) SEM and (b) registered CT images: Image size = 1.2 mm x 0.9 mm. (c) Correlation between x-ray attenuation and porosity.

Correlation of pore size to attenuation: Other properties obtained at high resolutions from the 2D SEM images can also be quantitatively integrated with registered 3D slices from tomographic images. In the example shown in Fig. 8, the SEM image (a) is obtained at a pixel resolution of 0.25 microns. Fig. 8(c) is a registered slice from the X-ray CT image obtained at a voxel size of 2.85 microns. The X-ray attenuation can be correlated to the pore size data in the Fig. 8(a). Fig. 8(b) represents mapping of the local pore size distribution on the SEM image data. The grey scale in the central image corresponds to pore size from the SEM slice with the brighter regions corresponding to larger pore sizes and darker greys to smaller pore sizes. Visually the correlation between (b) and (c) is reasonable. Fig. 9 shows a plot of the correlation between the local pore size distribution (y-axis) and the microporosity mapping from the X-ray attenuation (x-axis). This correlation allows mapping of the pore sizes throughout the entire 3D image.

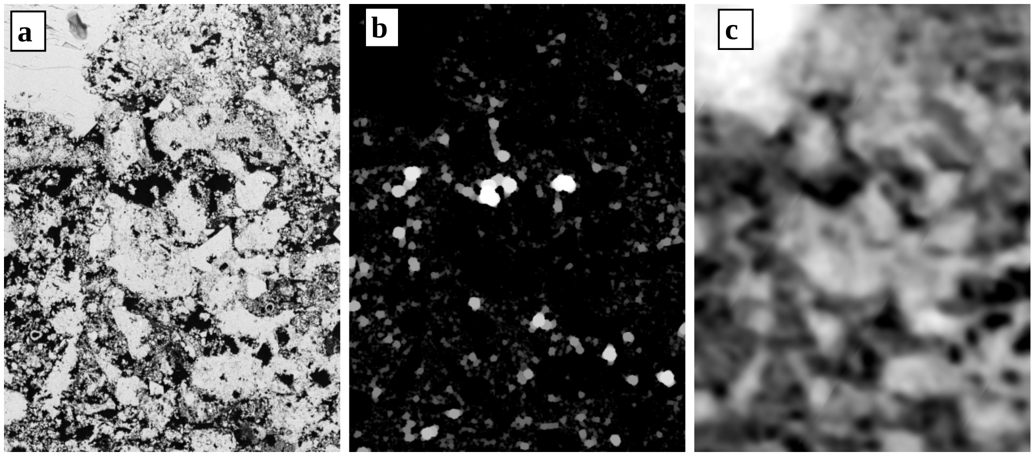

Fig. 8: (a) SEM image obtained at a pixel resolution of 0.25 microns. (b) Mapping of the local pore size distribution on the image data; the grey scale in this plot corresponds to pore size from SEM slice. The brighter regions correspond to larger pore sizes. (c) Registered slice from the x-ray CT image obtained at a voxel size of 2.5 microns. The xray attenuation on this image can be correlated to the pore size mapping in the central image.

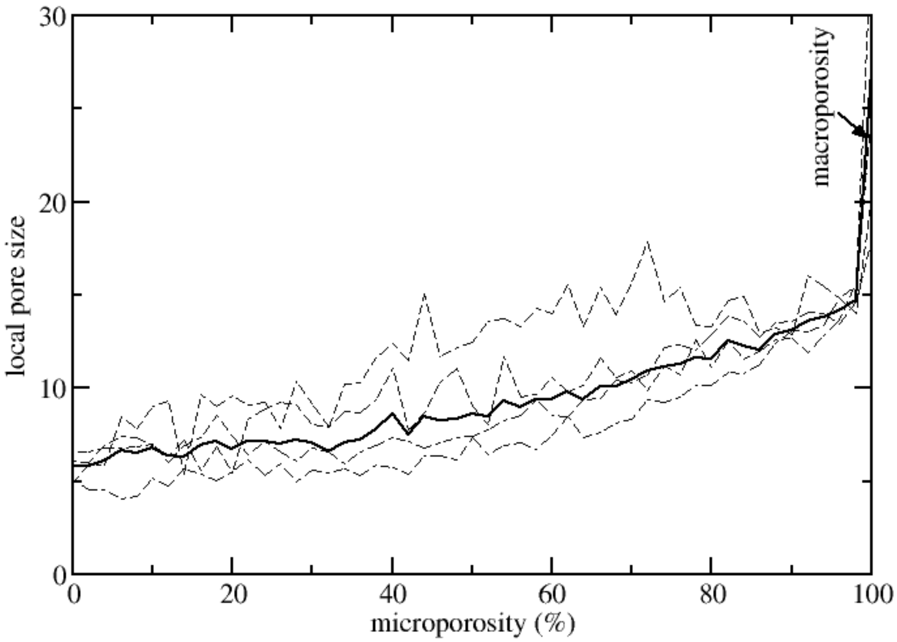

Fig. 9: Correlation between the local pore size distribution (y-axis, pixel units, pixel size is 0.25 micron) and the microporosity mapping from the x-ray attenuation (x-axis). The four dashed lines show the correlation of four separate regions and the solid line is the average correlation. This correlation allows one to map the pore sizes throughout the 3D image.

Integration of FIBSEM data with micro-CT data

While many carbonates exhibit extreme geological heterogeneity, very fine-grained mudstones may be extremely uniform in a reservoir at a scale much smaller than a plug. Here measurements at small scales could be an appropriate scale of investigation. Unfortunately the pore sizes in many mudstones lie below the resolution of conventional micro-CT analysis, and it remains difficult to gather any 3D information about the pore space with image data at the >1-2 micron resolution. Fig. 10(a-b) shows the MICP porosimetry curve and a slice from a micro-CT image of a mudstone sample. From the MICP data we note that the pore throats in the sample are of the order of 0.2-1 micron and the image obtained at a voxel size of 2.1 microns shows most of the pore features are below the resolution of micro-CT. While one can undertake 2D to 3D registration of slices from the image and attempt to map porosity and pore size, it would be difficult to capture the topology and pore connectivity of this sample utilizing these methods alone.

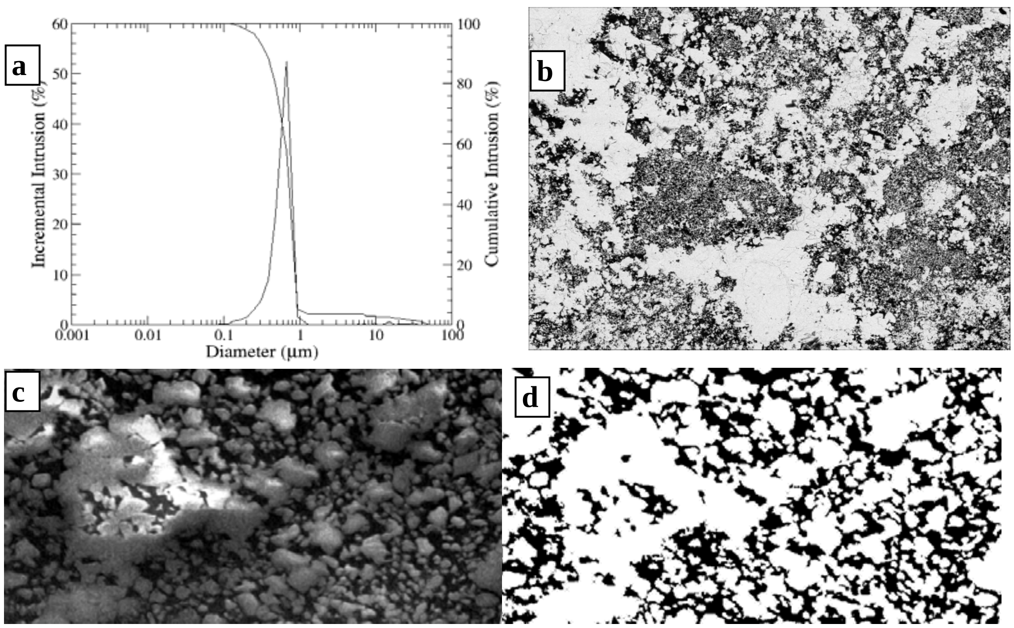

Fig. 10: (a) MICP data for a mudstone. (b) 1.2 x 0.9 mm subset of a slice from the tomogram obtained at 2.1 microns per voxel. No clear porosity is visible at this resolution. Large connected regions of microporosity are evident throughout the volume. (c) raw image of a slice from the image (80 x 45 microns) and (d) the slice phase separated into pore and solid phases.

In order to estimate reservoir properties of this rock effectively we must use a high resolution 3D imaging tool with finer scale resolution. We utilize FIBSEM to undertake an imaging study of the mudstone at a submicron scale. The total image slice size was 1024 x 884 pixels; the region of the sample imaged was 80x45x65 microns and the resolution in plane was 105 nm. Raw and segmented images from a slice of the tomographic data is shown in Fig. 10(c) and (d). The porosity obtained from the segmented image ϕ=33.8% is slightly higher than that measured on the plug 29.3%. However, given the small sample size this variation is not unexpected. Permeability was calculated on the imaged sample in three orthogonal directions gave k=2-3 mD. This is in good agreement with plug scale permeability of k=1 mD.

We have made initial attempts to co-register the 3D micro-CT data (2.1 microns per voxel) to the 3D FIBSEM data (105 nm per voxel). Given the limited sample size of the FIBSEM data set this has been quite difficult. A longer term goal of this work is to undertake complete multiscale registration from the whole core and/or plug scale through the micro-CT length scale and down to the submicron (SEM or FIBSEM) scale. This work is ongoing.

DISCUSSION

Carbonates contain 3D structural features of vastly different dimensions (from nanometers to centimeters). One single 3D image cannot capture the full information at all multiple scales (such an image would require 10,000,000 cubed or 2 x 10^9 terabytes).

Due to the exact and quantitative nature of the image registration methodology described here one can use features identified at a higher resolution image to populate a larger image captured at poorer resolutions. This can potentially address an important problem in understanding carbonate reservoirs—the need to find the appropriate data across multiple scales of investigation for different properties. In this discussion section we give an example of one method to use this multiscale information for enhanced analysis of petrophysical properties.

Here we consider the imaging and characterization of a carbonate sample exhibiting a broad pore size distribution. The MICP data, SEM image and registered 3D slice of the sample is shown in Fig. 11. This sample clearly exhibits significant regions of the pore space that are >100 micron in extent. Many of these pores are not accessible via throats at this scale; the MICP data indicates a small fraction of pores accessible at throat sizes >5 microns. The images therefore indicate a complex multiscale pore space. Segmentation of the tomographic image leads to estimates of macroporosity ϕ_{macro}=11%, microporosity ϕ_{micro}=24% and therefore total porosity=35%. This is in reasonable agreement with MICP data which indicates ϕ=36%. As the macropores do not connect macroscopically a pore network from the micro-CT is only made up of isolated clusters of pores (Fig. 12(a)). Describing the interconnected pore space requires imaging at higher resolutions. FIBSEM analysis was undertaken on a subset of this core to probe the pore size and connectivity of the extensive microporous regions of the sample (Fig. 11(d)). A pore network can be extracted from the FIBSEM data (shown in Fig. 12 (b)). This pore network is well connected in 3D. One can then utilize a stochastic method to embed the network obtained from the FIBSEM data onto the larger sample volume (Figs. 12 c-d). This integrated network spans in 3D. Network representations allow one to quantify the geometrical and topological properties of the pore space and give good representations of the pore space in the context of capillary dominated flow processes. One can therefore use information from this integrated network to undertake multiphase flow simulations which incorporate information on pore structural at multiple scales in a carbonate core.

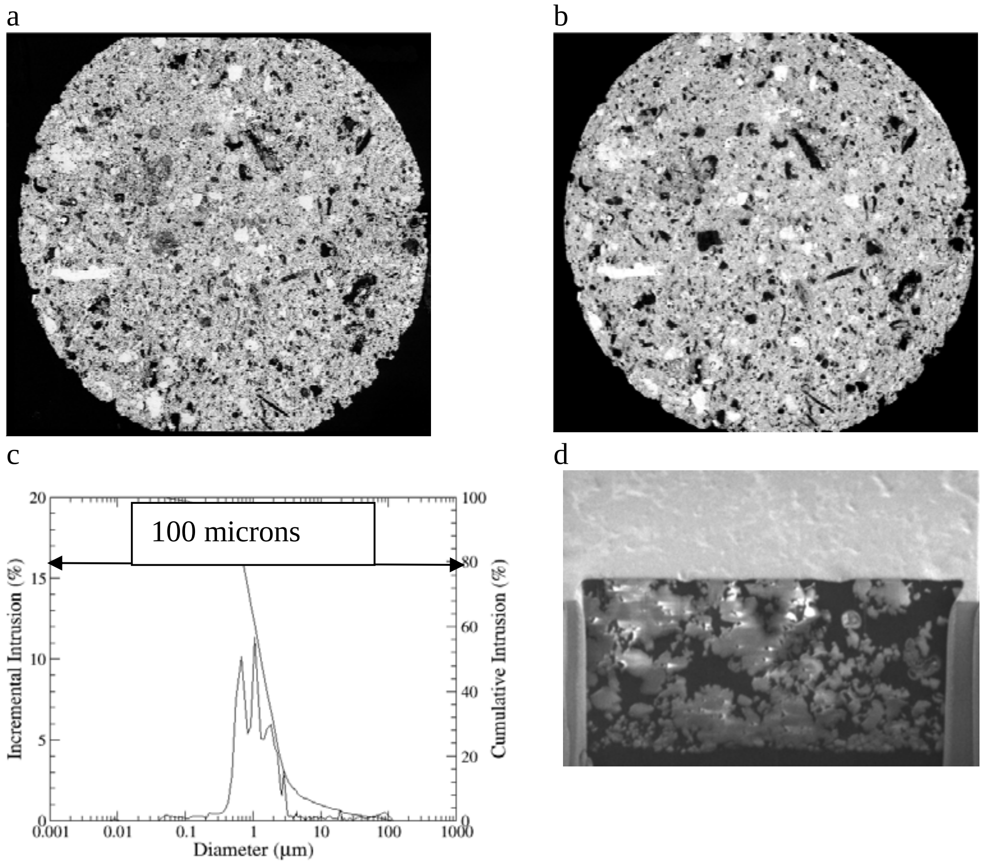

Fig. 11: (a) SEM and (b) registered microtomographic slice of a multimodal carbonate sample (field of view is 5mm diameter). (c) MICP curve for the sample and (d) a slice from the FIBSEM of a subset of this sample.

CONCLUSION

- Image registration is used to probe the 3D pore structure of carbonate cores across several decades of scale. Methods integrated include SEM, FIBSEM and micro-CT. Images from the plug scale (4 cm) to images at 100 nanometer resolution are integrated.
- Dewarping of 2D SEM images is implemented to ensure better alignment of image data across imaging modalities.
- X-ray attenuation data from micro-CT images at 2 micron per voxel are directly correlated to porosity identified via high resolution SEM (250 nanometers per pixel). The correlation enables one to map microporosity throughout the 3D image space.
- In carbonate samples considered here a good correlation was observed between x-ray attenuation from micro-CT data and pore sizes of 500 nm and greater.
- FIBSEM data for monomodal microporous carbonates give good agreement with porosity and permeability obtained from conventional core analysis.

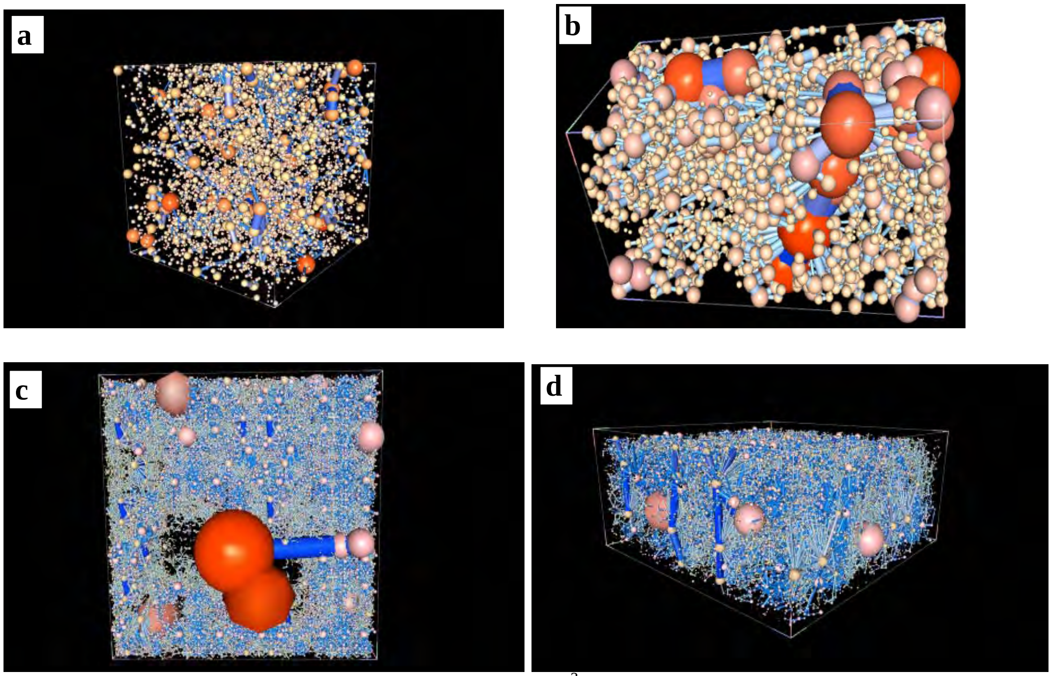

Fig. 12: (a) Topology of the pore network (500 voxels^3) generated from 2 micron resolution micro-CT data. The pores resolved at this scale do not connect in 3D. (b) Network generated from 100nm resolution FIBSEM image (600 x 300 x300 voxels^3).
(c) and (d) Dual scale networks incorporating the macropores from (a) and the microporous network in (b). (c) shows a region of 250 x 250 x 40 microns^3 incorporating > 30,000 pores and 50,000 throats (d) shows a zoomed region of 250 x 125 x 40 microns^3 within (c).

ACKNOWLEDGEMENTS

The authors acknowledge the member companies of the Digital Core Consortium for providing funding support and the Australian Partnership for Advanced Computation for supplying computing resources. We thank Christoph Arns for obtaining the permeability data on the FIBSEM images.

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