Sub-Micron Digital Image Analysis (BIBSEM-DIA), Pore Geometries, and Electrical Resistivity in Carbonate Rocks*

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Abstract

Assessment of electrical flow properties in heterogeneous carbonate rocks with pore sizes spanning several orders of magnitude requires a multi-scale investigation of the pore system. A new technique using Digital Image Analysis (DIA), ranging from millimeter to nanometer scale, allows for imaging and quantification of the sub-micron pore space in unprecedented detail. To capture the nanometer-scale pores, a new method of Broad-Ion-Beam (BIB) milling is used that produces true 2-D cross-sections for subsequent SEM image mosaic acquisition (BIBSEM).

Four samples were chosen from different depositional and diagenetic environments to compare their distinct microstructures. All samples have similar porosity (15%) for the sake of comparability. Electrical resistivity was measured on all samples; pore size distribution was analyzed with mercury injection capillary pressure (MICP) methods, and samples were investigated for their macropore structure with DIA from Optical Light Microscopy (OLM) on epoxy-impregnated thin-sections. For imaging micropores, the sample surfaces were milled down to nanometer-precision flatness with a JEOL SM-09010 BIB cross-section polisher. The large BIB surfaces (up to 2 square mm) are investigated at 5000x and 15000x magnification (resolution: 58.6 nm/pixel and 18.5 nm/pixel, respectively), and acquired BIBSEM mosaics are composed of up to 570 images each. Combining results from BIBSEM-DIA with OLM-DIA yields a multi-scale analysis.

The ultra-high-resolution BIBSEM image mosaics reveal the diverse microarchitectures of the different rock types, allowing for qualitative estimation of flow properties. The most interesting finding from quantitative DIA is that Pore Size Density Distribution (PSDD) follows a power law when pore sizes are normalized to bin width and area and plotted on log-log scale. This implies that pore densities at all scales as well as the Total Pore Density (TPD) can be modeled from DIA at a single resolution. Furthermore, combining conventional image analysis with spatial analysis using GIS software quantifies pore network connectivity (Nearest Neighbor Connectivity Factor; NNCF). The hypothesis is that the closer the next pore, the more likely a connection exists. The results of the multi-scale DIA display a good correlation between
calculated values for TPD and NNCF and electrical flow properties of the rock, corroborating earlier studies that electrical flow properties are strongly influenced by pore density and connectivity.

References Cited


Presenter’s notes: We present a new way to quantify microporosity in carbonate rocks—the BIBSEM method! The results from this method are fed into the Sub-micron DIA and the pore geometries are related to electrical resistivity in order to find the controlling factors.

The black box here represents a missing image in this large mosaic, where the automated raster acquisition failed to take the picture. It has a side length of only 20 microns, yet it contains about 1 million pixels. This should give us an idea of the size and resolution of the image mosaic in the background, which consists of 441 images. The lower part of the image in the background is not blurred out for artistic reasons, but it shows side by side the increase in resolution from previous techniques to the new BIBSEM method.
Presenter’s notes: Presentation starts with background on electrical resistivity and the motivations for this study.  
1st part--BIB-SEM method and resulting mosaics.  
2nd part--the multiscale DIA and results about pore sizes and distributions.  
3rd part--pore throat sizes from Mercury Injection,  
4th part--BT-ratio, which we get from a combination of MICP with DIA.  
Finish with the conclusion and implications.

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<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td><strong>Key Points, Background and Motivations</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>1</strong> Broad-Ion-Beam milling + SEM (BIB-SEM)</td>
<td>Nanometer-scale mosaics</td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>2</strong> Multiscale Digital Image Analysis (MsDIA)</td>
<td>Pore sizes / density / distribution</td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>3</strong> Mercury Injection Capillary Pressure (MICP)</td>
<td>Throat sizes</td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>4</strong> MsDIA + MICP</td>
<td>Pore body-to-throat ratio (BTR)</td>
<td></td>
</tr>
<tr>
<td></td>
<td><strong>Conclusion and Implications</strong></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Key Points

Pore geometry <-> cemenation factor $m$:

- Multiscale-DIA confirms thin-section DIA
  - Smaller pores = lower $m$
  - More pores = lower $m$

- MICP (Mercury Injection)
  - Smaller throats = lower $m$

- BTR (Body-Throat-Ratio)
  - High BTR $\neq$ high $m$
  - BTR: Small effect on $m$

- PSD (Pore Size Distribution)
  - Follow power law!
  - Predictable pore density!
Electrical resistivity is described independent of porosity by the cementation factor. Higher cementation factor = less electrical flow. For example, if you measure $F_m$ resistivity, and know the pore fluid and the porosity, you can calculate $m$. Also, we know that $m$ is controlled by pore geometry; therefore, if we have good knowledge about these controls, we know what kind of pore geometry is behind which cementation factor, $m$, which can be used to predict the pore geometry.
Presenter’s notes: Here, on the left, the size of the pores (DOMsize) is plotted against the complexity of the pore structure (PoA), showing that large and simple pores have high cementation factors, in red color, whereas small and complex pores have low cementation factors, in blue. Similarly, on the right, samples with a high number of pores (on the y-axis) have a lower cementation factor. This means, that because microporosity largely controls pore size and density, it has strong control on cementation factor m! Electrical-flow properties of carbonate rocks are directly related to the amount of pores and size of the pores, both being closely linked to the amount of intrinsic microporosity.
Presenter’s notes: That brings us to the motivation for this specific work, because conventional imaging methods cannot resolve microporosity! We want to determine, if the results obtained from OLM and Micro-CT hold true, when incorporating the smallest pores!!
Presenter’s notes: Quantifying SEM images:
Conventionally prepared surfaces are too rough to quantify pore space; you need a perfectly planar surface to see the pore geometry!
Solution: BIB-milling, with surface topography of only 5 nanometers.
Next we see how BIB-SEM works.
Presenter’s notes: Again, background on electrical resistivity and our motivations is followed by BIBSEM method and resulting mosaics, then combining BIB-section with thin-section images for multiscale analysis, and finally the implications.
Presenter’s notes: Method utilizes this BIB cross-section polisher
Here we show how a sample is mounted inside, underneath this shielding plate, as seen in illustration (a): The sample in blue is sticking out a little underneath the shielding plate so that the ion beam from above takes away the overhanging parts. This results in this very flat surface, in yellow.
Presenter’s notes: One can acquire image rasters of the surfaces, such as shown here.
Presenter’s notes: To show the increase in resolution: This is the polished surfaces shown at magnification similar to optical light microscopy.
Presenter’s notes: If we take a closer look, we can see the dramatic increase in resolution, about 100 times higher!!
Presenter’s notes: The samples here have same porosity, around 15%--from different depositional and diagenetic environments to compare their distinct microstructures, representing “endmember” rock types!
We then acquired large rasters from each type; the results are very large mosaics with very high resolution, illustrated next.
Presenter’s notes: GIGAPIXEL mosaics; note the scales: Well connected micropore network between isopachous, bladed cements, but low number of pores.
Presenter’ notes: Dense pore network between microspar crystals, high number of pores within micritic matrix.
Presenter’s notes: High number of pores, but separated and scattered in dense matrix.
Presenter’s notes: Very few pores, but very well connected between crystalline dolomite rhombs.
Next, what was done with the images.
Presenter’s notes: Now we combine thin-section and BIB-section images for a Ms-DIA and show the results. We want to see if the results obtained from OLM and Micro-CT hold true, when incorporating the smallest pores!! (More pores /smaller pores meaning lower cementation factor)
Presenter’s notes: By combining the high-res BIBSEM mosaics with the thin-section images, we get a multiscale analysis, including pores across 6 orders of magnitude.

It was done by assigning the “dense”, non-macroporous rock areas in the thin-sections with the micropore properties from the BIB-SEM mosaics. Multiscale is especially important in carbonates in order to capture their heterogeneity, which can be seen here in the illustration.
Presenter’s notes: We want to see how pore density and pore size control electrical resistivity and cementation factor $m$, starting with the pore density.

$$TPD = \frac{\text{Amount of pores}}{\text{Area}}$$
Presenter’s notes: The samples here are ordered with increasing cementation factor from left to right.
PoA describes how complex/complicated the enclosure of the pore system is, regardless of the total amount of porosity.

PoA is the ratio of the sum of the perimeter and the sum of the area of all pores identified on a thin section.

\[
PoA = \frac{\sum \text{Perimeter}}{\sum \text{Area}}
\]
Presenter’s notes: We want to see how pore density and pore size control electrical resistivity and cementation factor m.
DOMsize is the maximum size of the pores needed to occupy 50% of the pore space on a given thin section.

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Presenter’s notes: Most interesting finding: power law in PSDs!
Histogram - pores are distributed into logarithmically spaced bins according to their size (area)
Self-similarity can be seen when zooming into the pore space of the Ooid Grainstone, for example. The pore space looks similar at different scales!
And the best thing is shown next.
Presenter’s notes: We found these fractal distributions in ALL 4 SAMPLES, even though there are carbonate heterogeneity and obvious differences in pore structure!!

The kink in the data at the very small sizes is due to sampling error, when reaching resolution limit (at around 50 nm).

Reduce cost of analysis:

For example: estimate micropores from thin-sections, no time-consuming BIB-SEM needed!!

Or, estimate macropores from drill-cuttings; no expensive coring needed!
<table>
<thead>
<tr>
<th></th>
<th>Outline</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Key Points, Background and Motivations</td>
</tr>
<tr>
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</tr>
<tr>
<td></td>
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</tr>
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<tr>
<td></td>
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</tr>
</tbody>
</table>
Presenter’s notes: Here are the MICP curves for the 4 samples, with smaller throats to the right, and as we see, smaller throats mean lower cementation factor m.
All pore throat and pore-size parameters are nicely in order of their cementation factor.
Smaller sizes for Critical diameter $D_c$, DOMthroat and also DOMsize result in lower cementation factor.
For the samples with the highest m, the statistical values for the inflection point is larger than the DOMsize of the pores.
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<thead>
<tr>
<th></th>
<th>Outline</th>
</tr>
</thead>
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<tr>
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</tr>
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</tr>
<tr>
<td></td>
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Presenter’s notes: Results are averaged pore network parameters.
Presenter’s notes: However, our analysis shows that a higher BTR actually does not hinder flow.
## Conclusion + Implications

Results from Multiscale Analysis confirm trends from thin-section DIA

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<th>DOMsize: best correlation with $m$</th>
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</thead>
</table>

Smaller pores/throats = Better electrical flow

$\approx$ Permeability

Body-Throat-Ratio = Weak impact on $m$

Higher BTR $\approx$ Higher $m$

Fractal pore size distributions in carbonates

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<th>Estimate pore density from subsamples</th>
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</thead>
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| Simplify analysis! |