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

Jan H. Norbisrath[1], Ben Laurich[2], Janos Urai[2], Guillaume Desbois[2], Gregor P. Eberli[1], Klaas Verwer[3], and Ralf J. Weger[1]

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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

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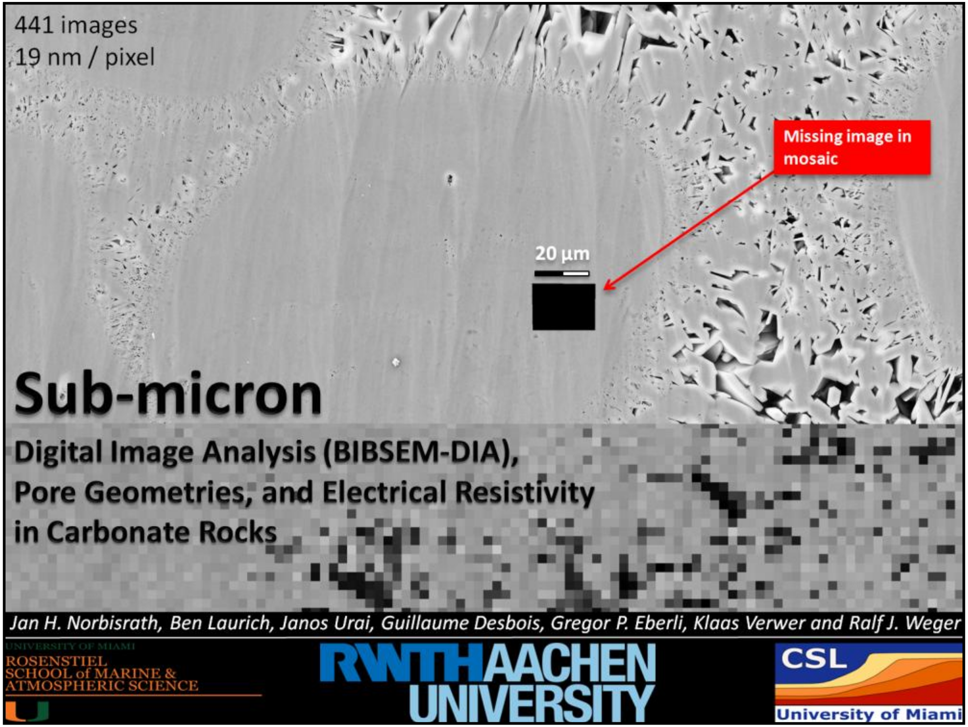

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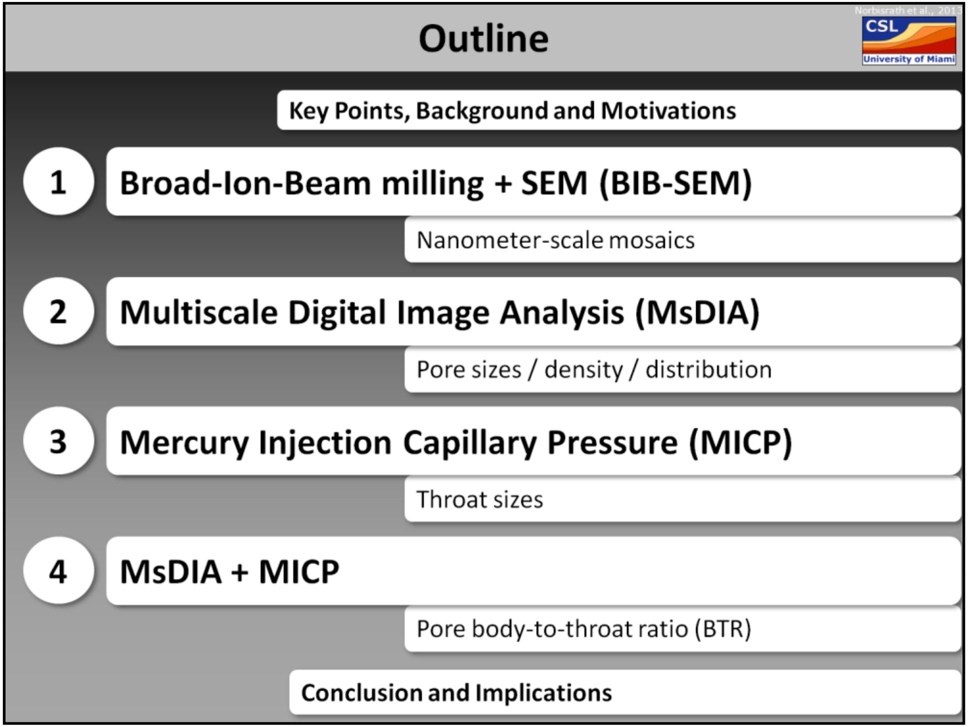
UNIVERSITY

CSL
University of Miami

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 arer 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.**



Norbisrath et al., 2013

Outline

Key Points, Background and Motivations

1 **Broad-Ion-Beam milling + SEM (BIB-SEM)**

Nanometer-scale mosaics

2 **Multiscale Digital Image Analysis (MsDIA)**

Pore sizes / density / distribution

3 **Mercury Injection Capillary Pressure (MICP)**

Throat sizes

4 **MsDIA + MICP**

Pore body-to-throat ratio (BTR)

Conclusion and Implications

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.

Key Points

Pore geometry <> *cementation factor m:*

Multiscale-DIA confirms thin-section DIA

- Smaller pores = lower ***m***
- More pores = lower ***m***

MICP (Mercury Injection)

- Smaller throats = lower ***m***

Microporosity: Lowers *m*

BTR (Body-Throat-Ratio)

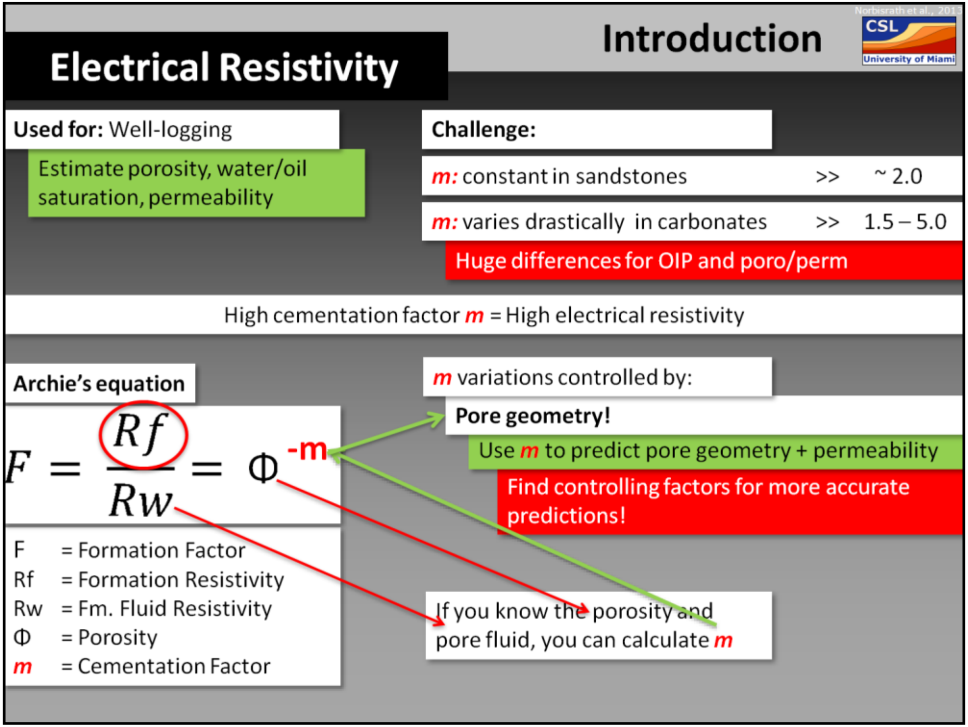
- High BTR =/= high ***m***

BTR: Small effect on *m*

PSD (Pore Size Distribution)

- Follow power law!

Predictable pore density!



Norbisrath et al., 2013

Introduction

Electrical Resistivity

Used for: Well-logging

Estimate porosity, water/oil saturation, permeability

Challenge:

m: constant in sandstones >> ~ 2.0

m: varies drastically in carbonates >> 1.5 – 5.0

Huge differences for OIP and poro/perm

High cementation factor *m* = High electrical resistivity

Archie's equation

$$F = \frac{Rf}{Rw} = \Phi^{-m}$$

F = Formation Factor
Rf = Formation Resistivity
Rw = Fm. Fluid Resistivity
Φ = Porosity
m = Cementation Factor

m variations controlled by:

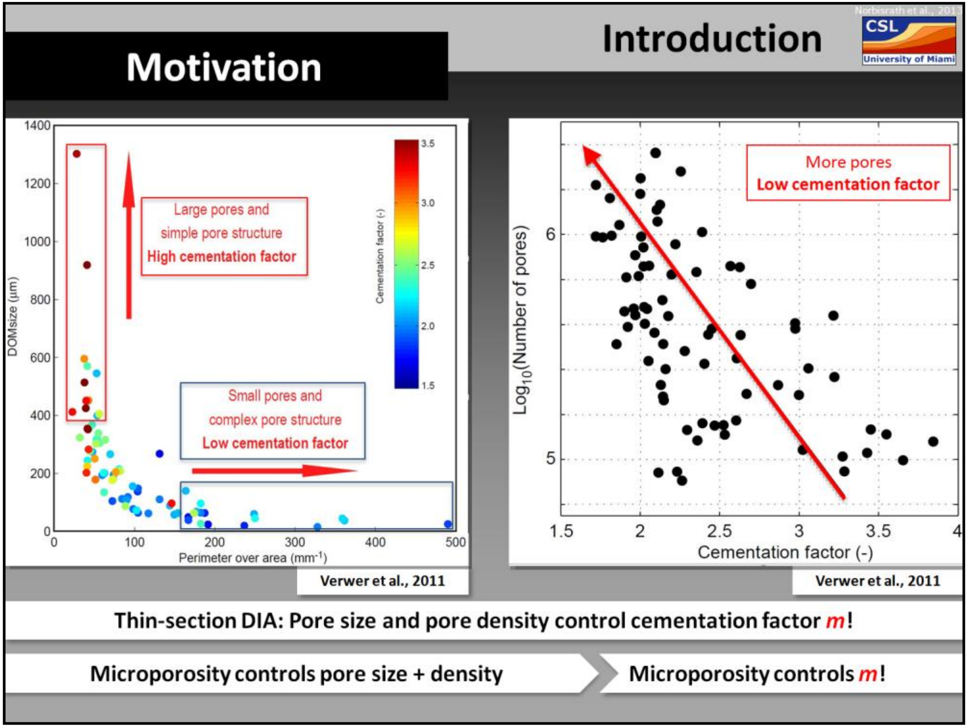
Pore geometry!

Use *m* to predict pore geometry + permeability

Find controlling factors for more accurate predictions!

If you know the porosity and pore fluid, you can calculate *m*

Presenter's notes: Electrical resistivity is described independent of porosity by the cementation factor. Higher cementation factor = less electrical flow. For example, if you measure Fm 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.



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Introduction

Motivation

Verwer et al., 2011

Verwer et al., 2011

Thin-section DIA: Pore size and pore density control cementation factor *m*!

Microporosity controls pore size + density

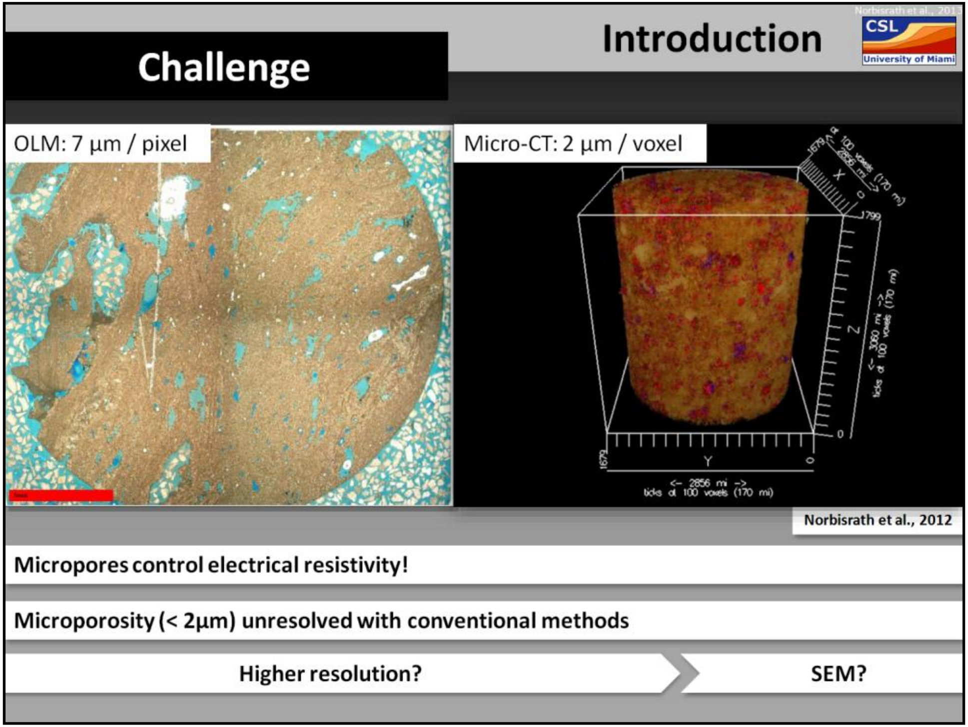
Microporosity controls *m*!

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.



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Introduction

Challenge

OLM: 7 µm / pixel

Micro-CT: 2 µm / voxel

Norbisrath et al., 2012

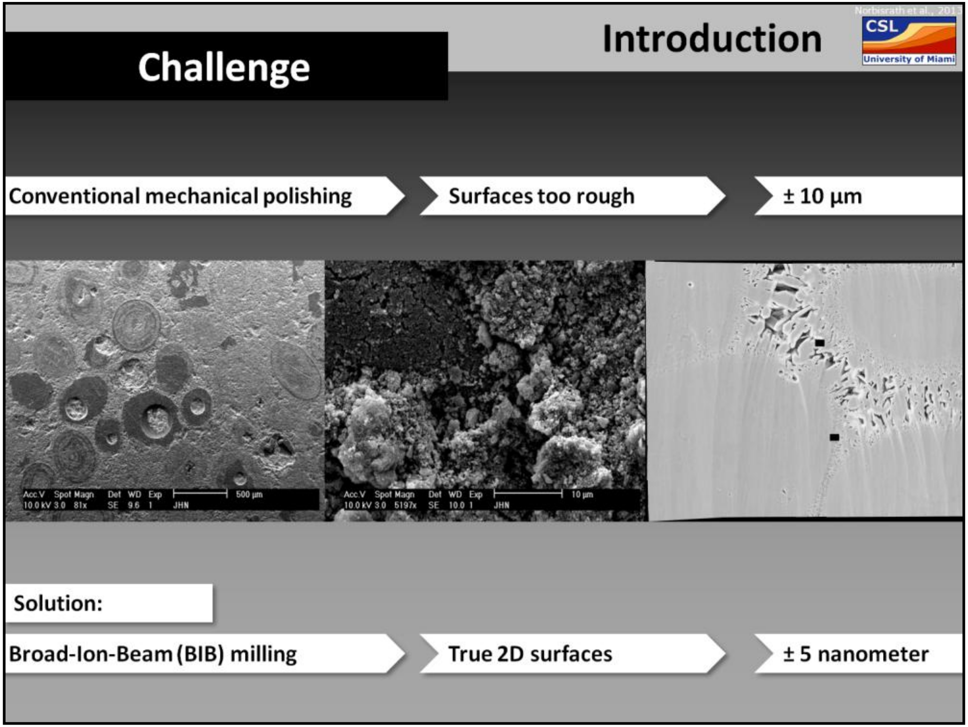
Micropores control electrical resistivity!

Microporosity (< 2µm) unresolved with conventional methods

Higher resolution? → **SEM?**

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!!**

Norbisrath et al., 2013



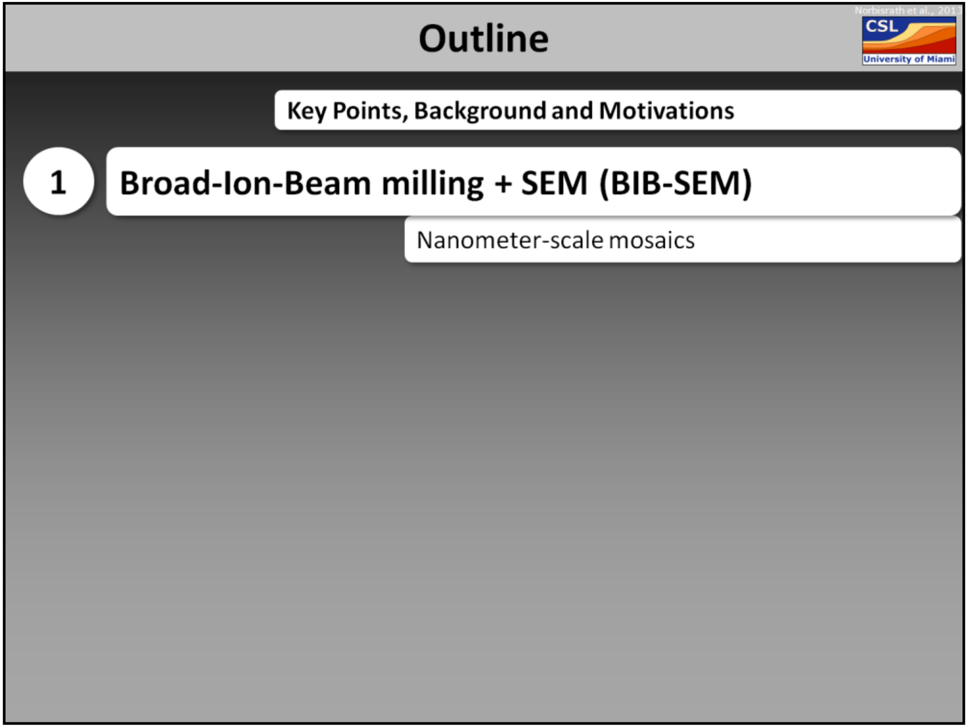
Challenge

Conventional mechanical polishing → Surfaces too rough → ± 10 µm

Solution:

Broad-Ion-Beam (BIB) milling → True 2D surfaces → ± 5 nanometer

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.



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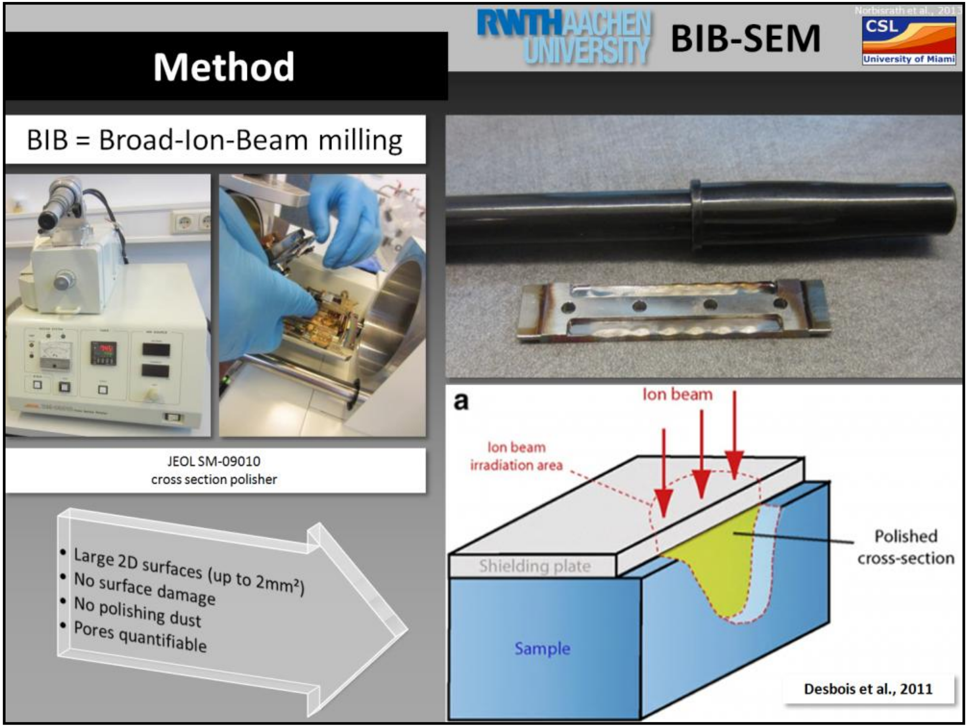
Outline

Key Points, Background and Motivations

1 **Broad-Ion-Beam milling + SEM (BIB-SEM)**

Nanometer-scale mosaics

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.



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BIB-SEM

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Method

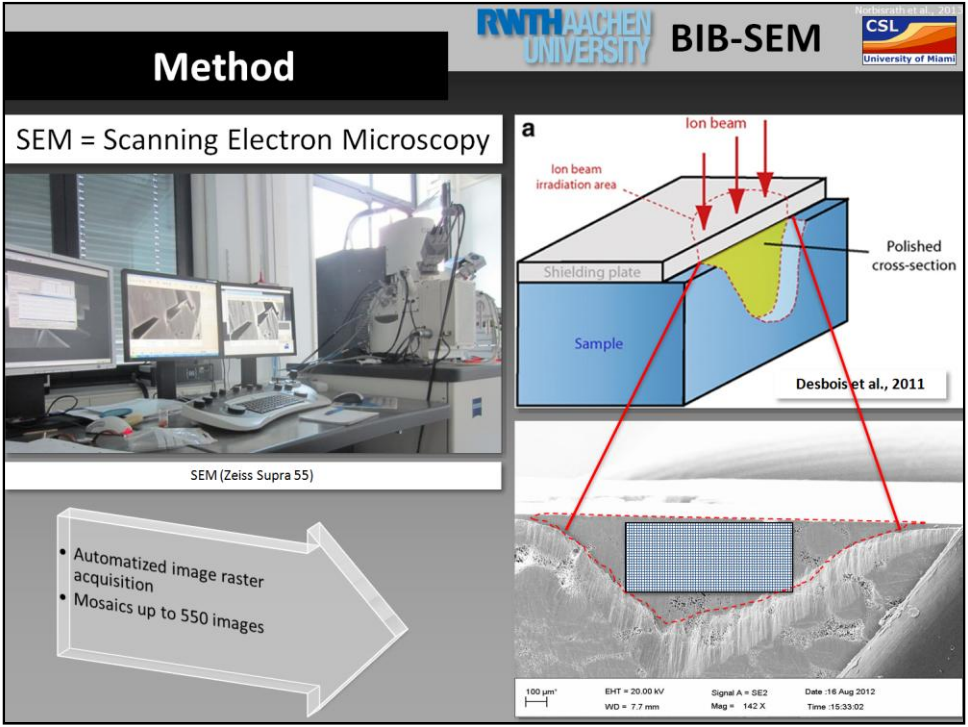
BIB = Broad-Ion-Beam milling

JEOL SM-09010
cross section polisher

- Large 2D surfaces (up to 2mm²)
- No surface damage
- No polishing dust
- Pores quantifiable

Desbois et al., 2011

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.



Norbisrath et al., 2013

Method

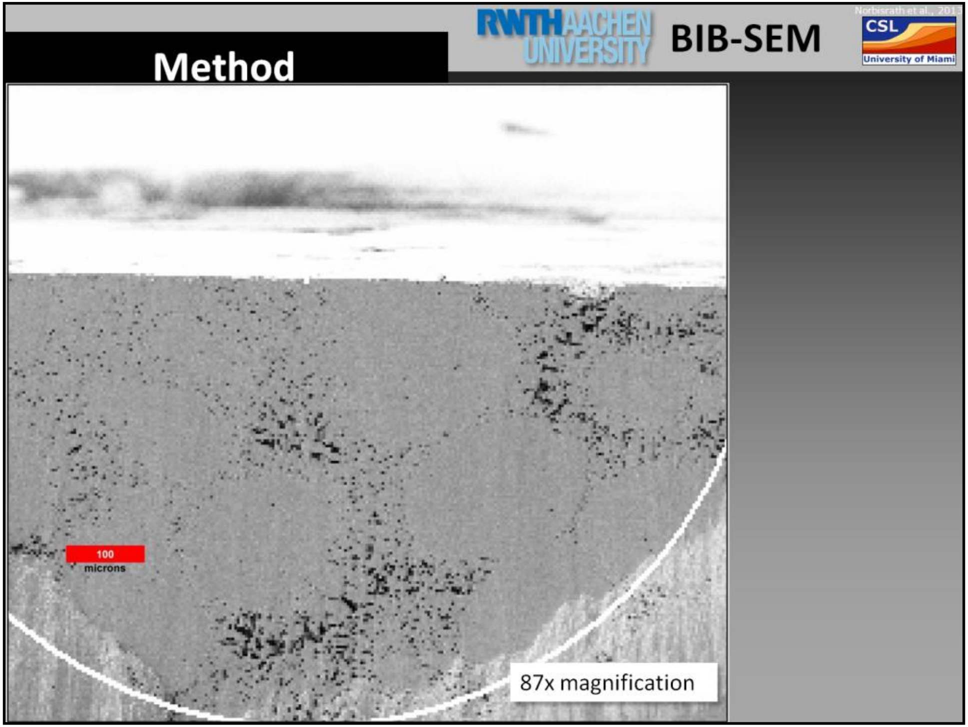
BIB-SEM

SEM = Scanning Electron Microscopy

SEM (Zeiss Supra 55)

- Automatized image raster acquisition
- Mosaics up to 550 images

Presenter's notes: One can acquire image rasters of the surfaces, such as shown here.



Method

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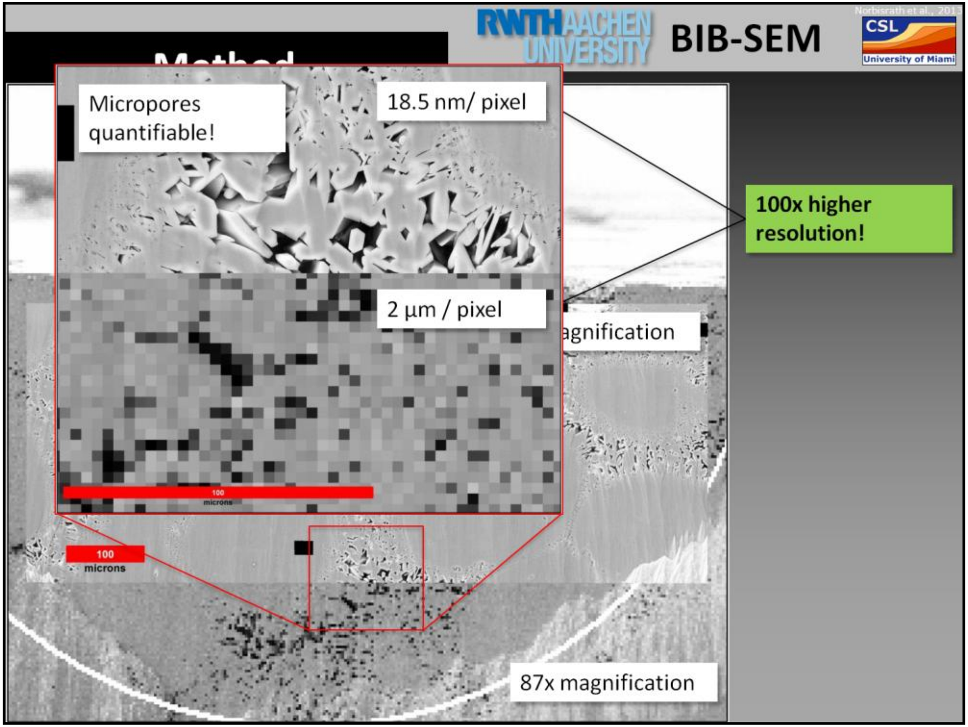
87x magnification

Presenter‘s notes: To show the increase in resolution: This is the polished surfaces shown at magnification similar to optical light microscopy



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Micropores quantifiable!

18.5 nm/ pixel

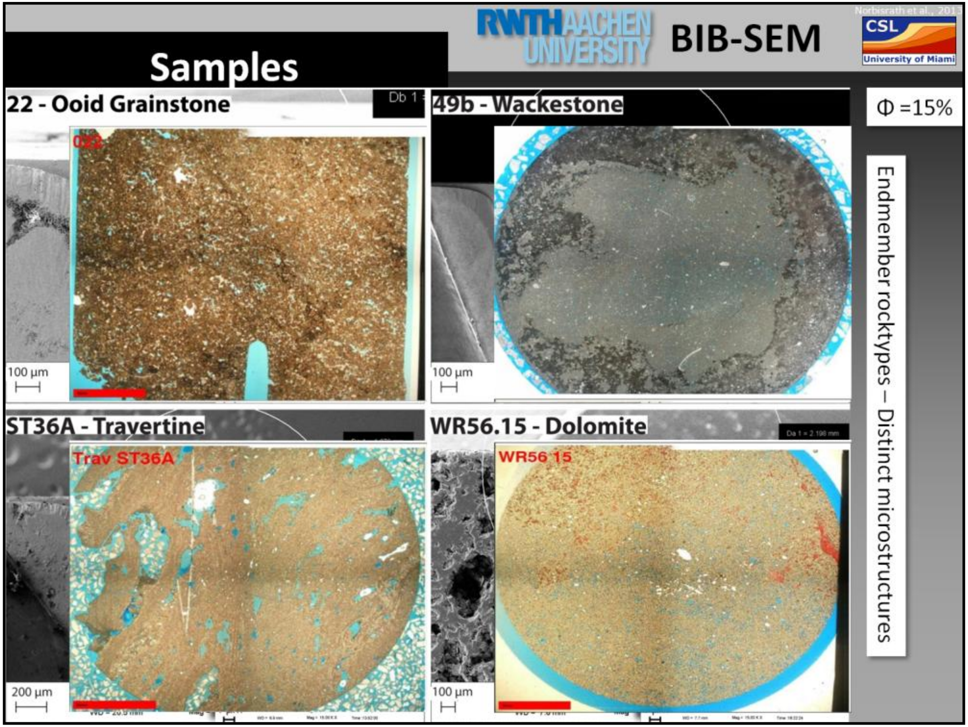
2 µm / pixel

agnification

100x higher resolution!

87x magnification

Presenter's notes: If we take a closer look,we can see the dramatic increase in resolution, about 100 times higher!!



Samples

BIB-SEM

Norbisrath et al., 201

22 - Ooid Grainstone

49b - Wackestone

ST36A - Travertine

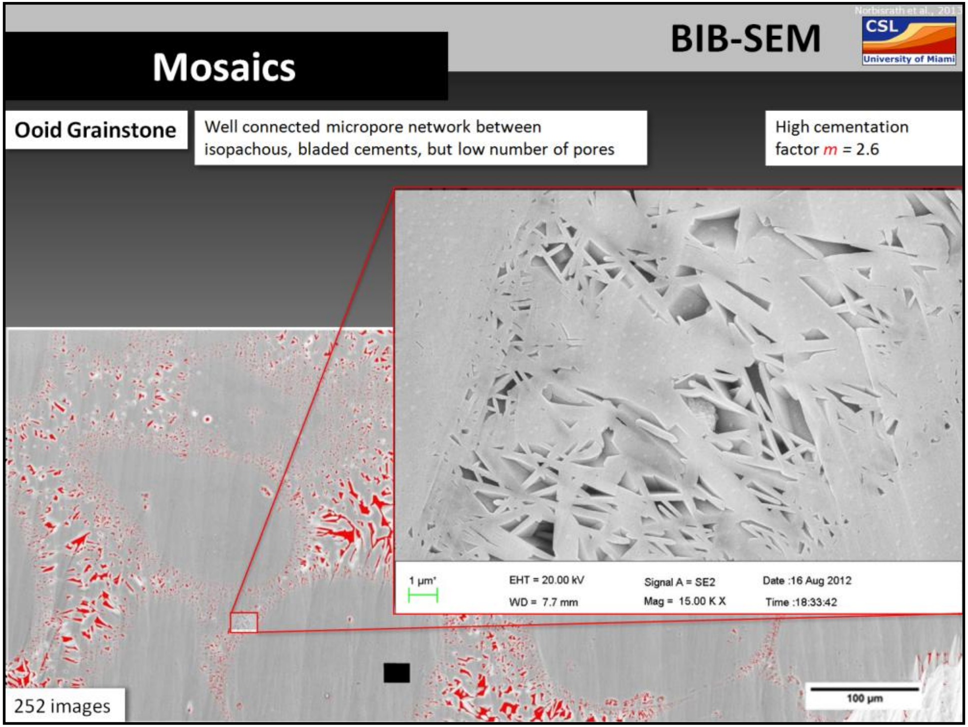
WR56.15 - Dolomite

Φ =15%

Endmember rocktypes – Distinct microstructures

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.



Mosaics

BIB-SEM

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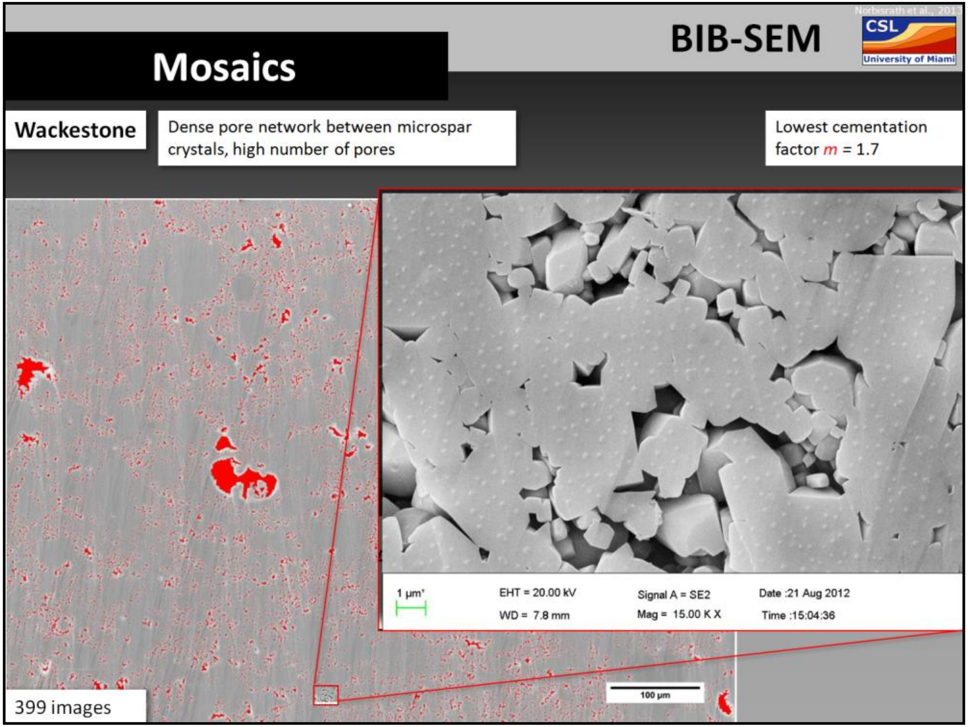
Ooid Grainstone

Well connected micropore network between isopachous, bladed cements, but low number of pores

High cementation factor *m* = 2.6

252 images

Presenter's notes: GIGAPIXEL mosaics; note the scales: Well connected micropore network between isopachous, bladed cements, but low number of pores.



BIB-SEM

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Mosaics

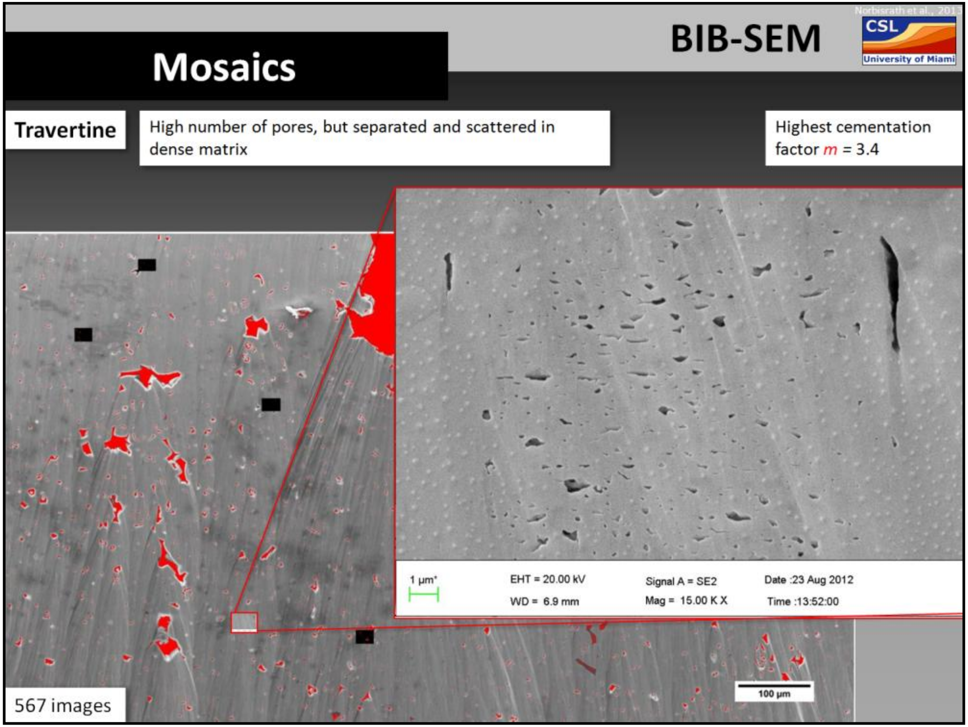
Wackestone

Dense pore network between microspar crystals, high number of pores

Lowest cementation factor $m = 1.7$

399 images

Presenter' notes: Dense pore network between microspar crystals, high number of pores within micritic matrix.



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BIB-SEM

Mosaics

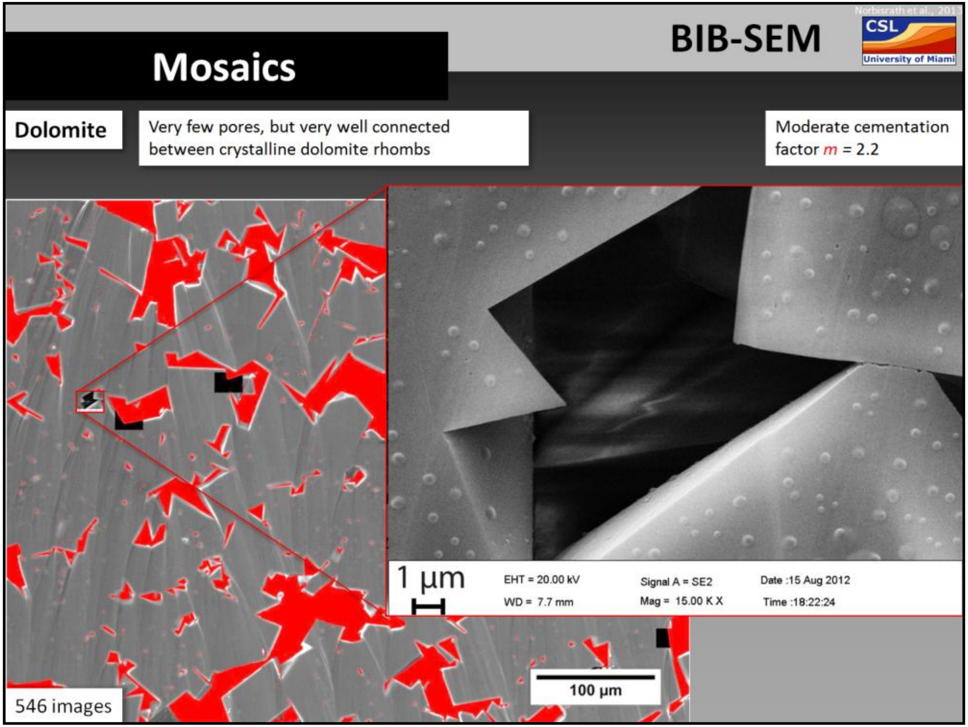
Travertine

High number of pores, but separated and scattered in dense matrix

Highest cementation factor $m = 3.4$

567 images

Presenter’s notes: High number of pores, but separated and scattered in dense matrix



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BIB-SEM

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Mosaics

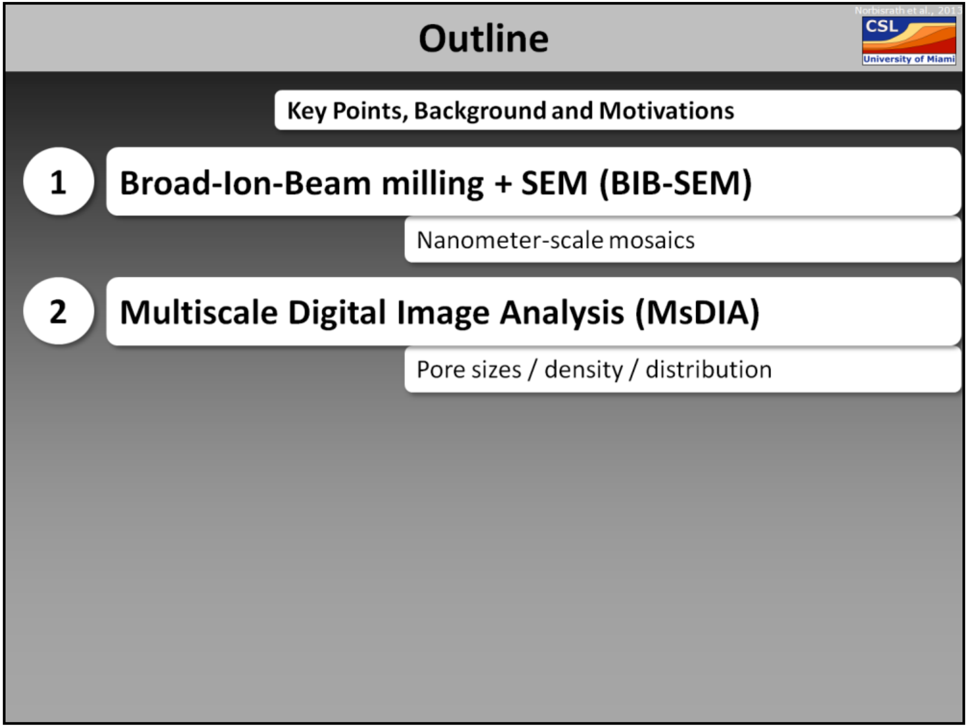
Dolomite

Very few pores, but very well connected between crystalline dolomite rhombs

Moderate cementation factor *m* = 2.2

546 images

Presenter's notes: Very few pores, but very well connected between crystalline dolomite rhombs. Next, what was done with the images.



Outline

Key Points, Background and Motivations

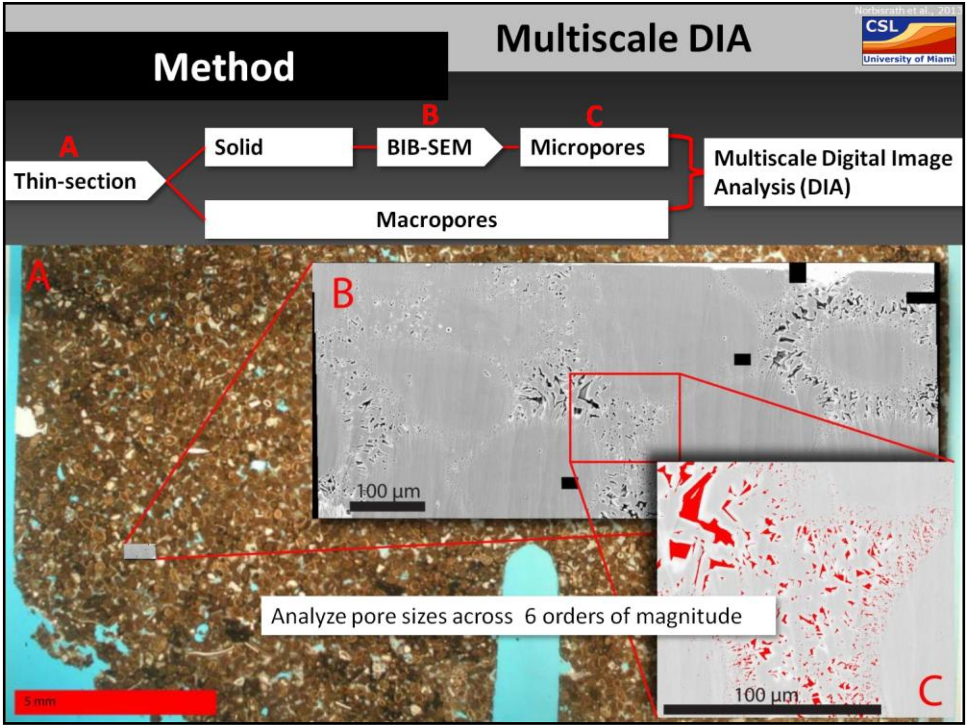
1 **Broad-Ion-Beam milling + SEM (BIB-SEM)**

Nanometer-scale mosaics

2 **Multiscale Digital Image Analysis (MsDIA)**

Pore sizes / density / distribution

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)



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Multiscale DIA

Method

Thin-section

Solid

BIB-SEM

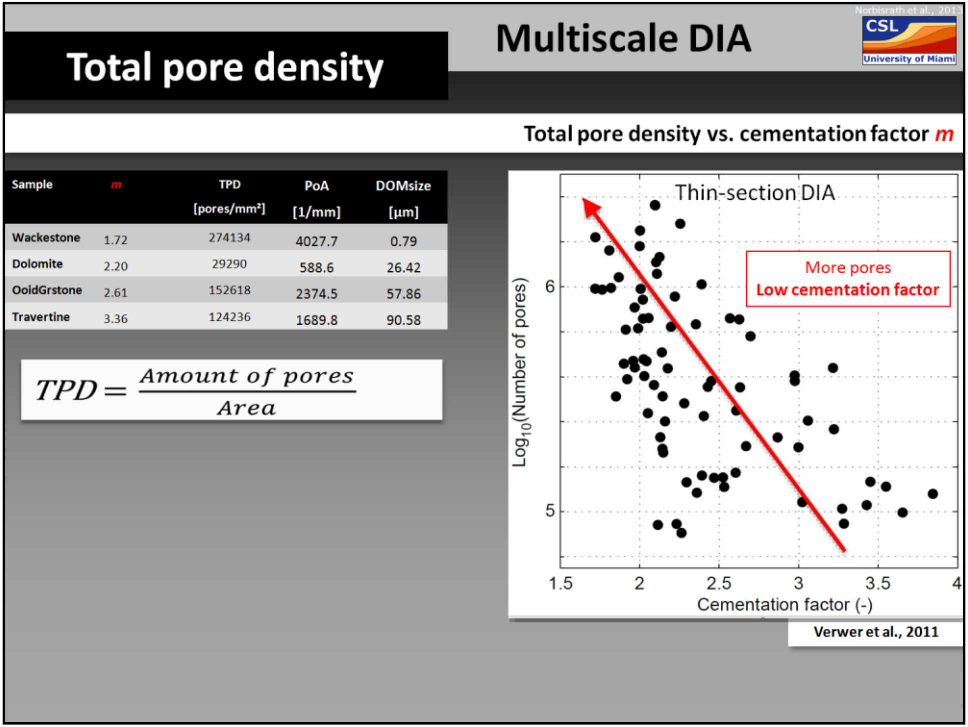
Micropores

Macropores

Multiscale Digital Image Analysis (DIA)

Analyze pore sizes across 6 orders of magnitude

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.



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Multiscale DIA

Total pore density

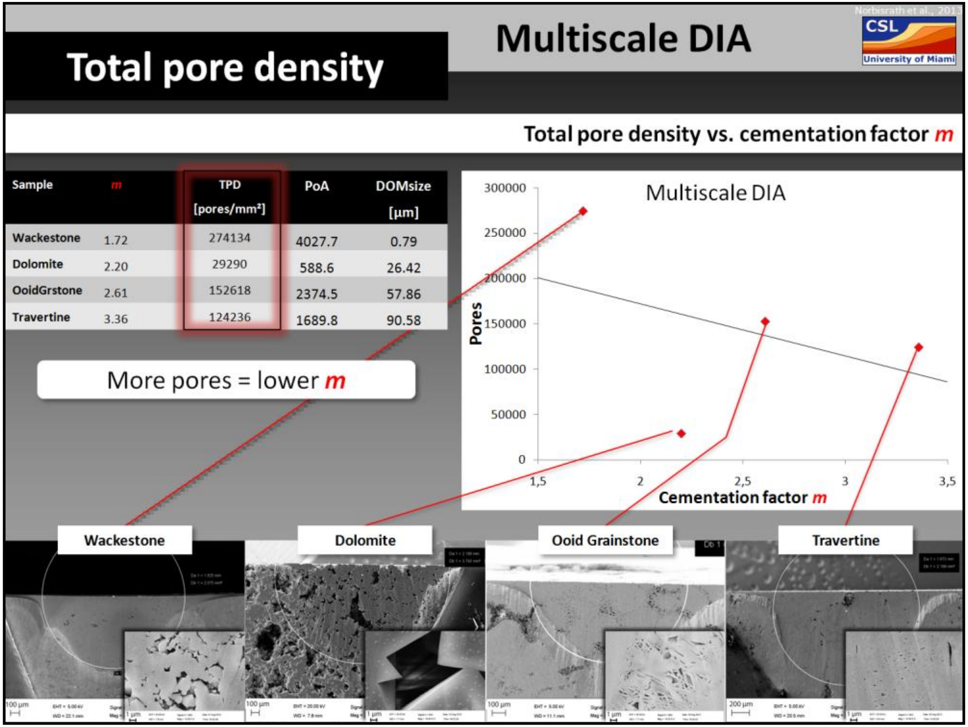
Total pore density vs. cementation factor *m*

Sample	*m*	TPD [pores/mm²]	PoA [1/mm]	DOMsize [µm]
Wackestone	1.72	274134	4027.7	0.79
Dolomite	2.20	29290	588.6	26.42
OoidGrstone	2.61	152618	2374.5	57.86
Travertine	3.36	124236	1689.8	90.58

$$TPD = \frac{Amount\ of\ pores}{Area}$$

Verwer et al., 2011

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.



Total pore density

Multiscale DIA

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Total pore density vs. cementation factor *m*

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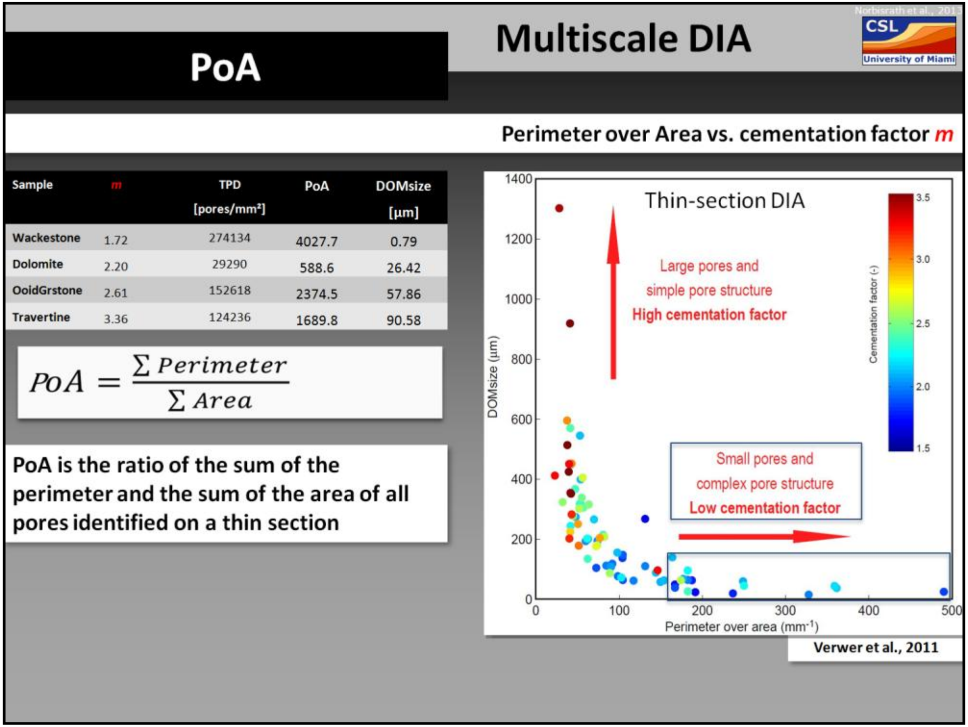
More pores = lower *m*

Presenter’s notes: The samples here are ordered with increasing cementation factor from left to right.



Norbisrath et al., 2013

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Multiscale DIA

PoA

Perimeter over Area vs. cementation factor *m*

Sample	*m*	TPD [pores/mm²]	PoA	DOMsize [µm]
Wackestone	1.72	274134	4027.7	0.79
Dolomite	2.20	29290	588.6	26.42
OoidGrstone	2.61	152618	2374.5	57.86
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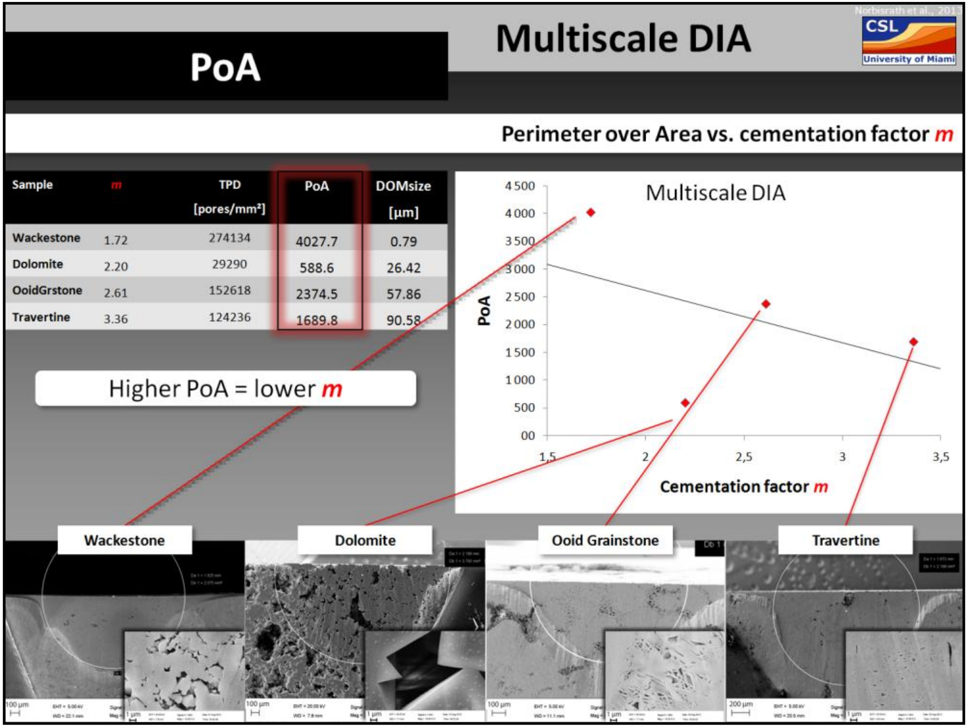
$$PoA = \frac{\sum Perimeter}{\sum Area}$$

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

Verwer et al., 2011

Presenter's notes: PoA describes how complex/complicated the enclosure of the pore system is, regardless of the total amount of porosity.



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Multiscale DIA

PoA

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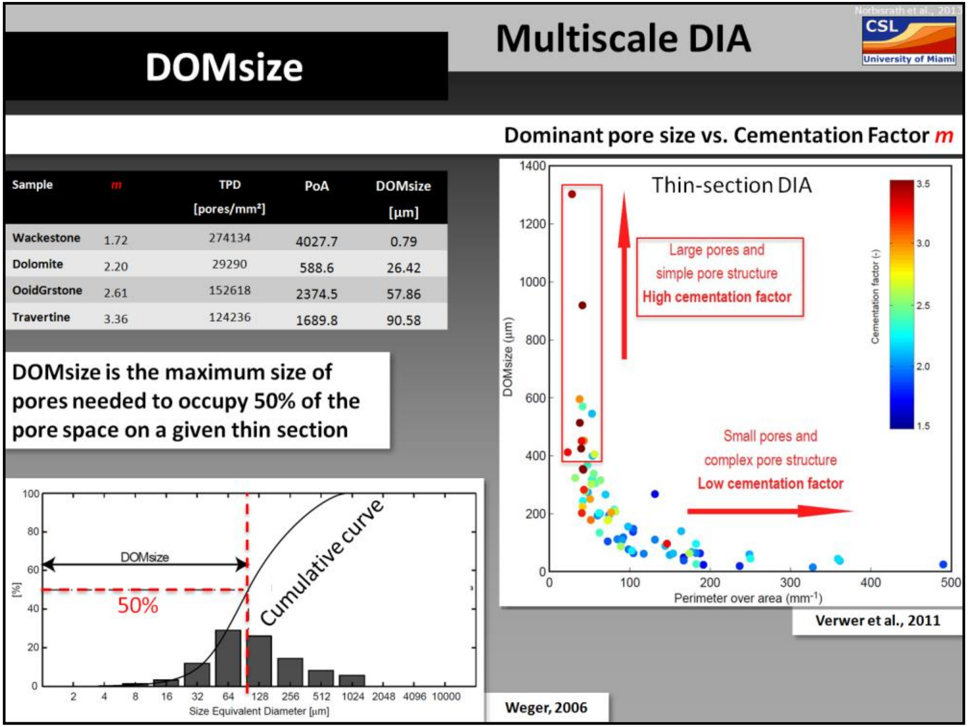
Higher PoA = lower *m*

Presenter's notes: We want to see how pore density and pore size control electrical resistivity and cementation factor m.



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Multiscale DIA

DOMsize

Dominant pore size vs. Cementation Factor *m*

Sample	*m*	TPD [pores/mm²]	PoA	DOMsize [µm]
Wackestone	1.72	274134	4027.7	0.79
Dolomite	2.20	29290	588.6	26.42
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DOMsize is the maximum size of pores needed to occupy 50% of the pore space on a given thin section

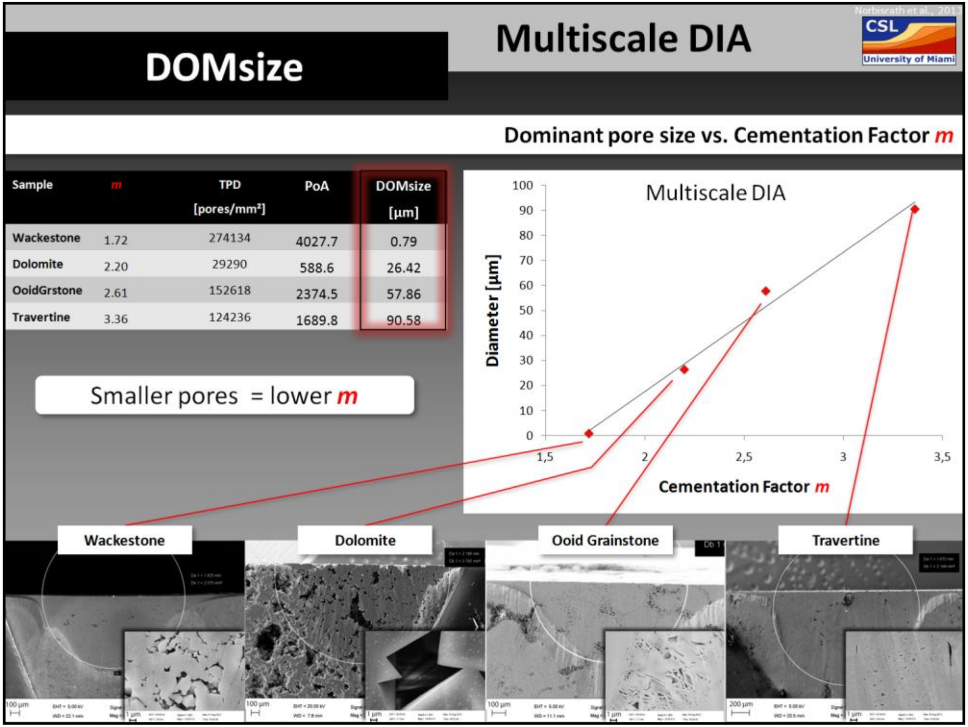
Weger, 2006

Large pores and simple pore structure
High cementation factor

Small pores and complex pore structure
Low cementation factor

Verwer et al., 2011

Presenter's notes: DOMsize is the maximum size of the pores needed to comprise 50% of the porosity on a given cross-section.



Norbisrath et al., 201

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Multiscale DIA

DOMsize

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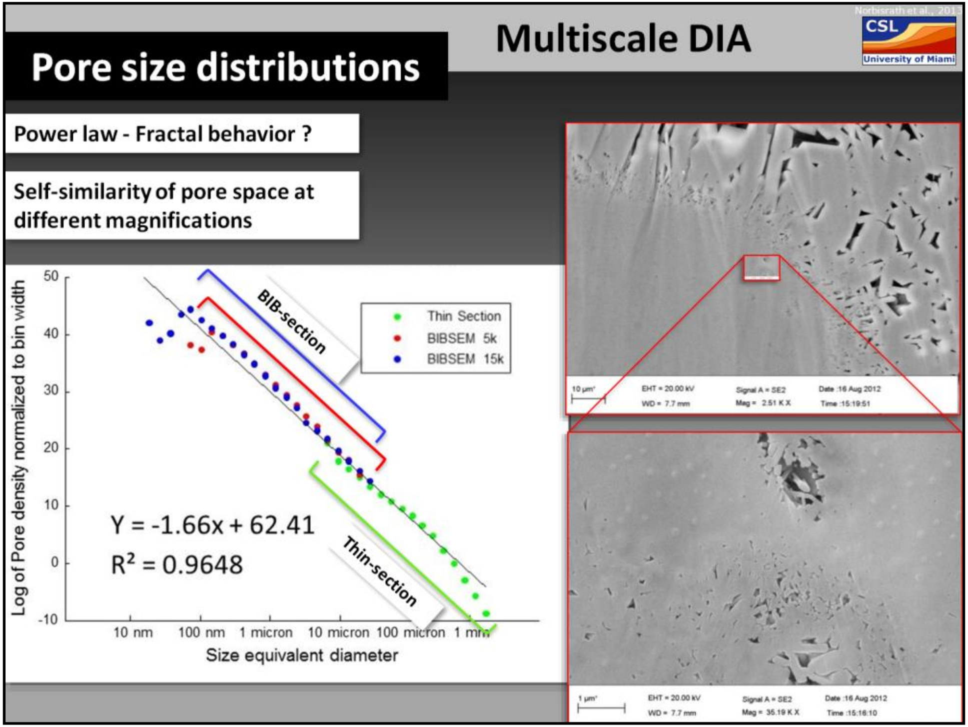
Smaller pores = lower *m*

Presenter's notes: DOMsize is the maximum size of the pores needed to comprise 50% of the porosity on a given cross-section.



Pore size distributions

Multiscale DIA

Norbisrath et al., 2013

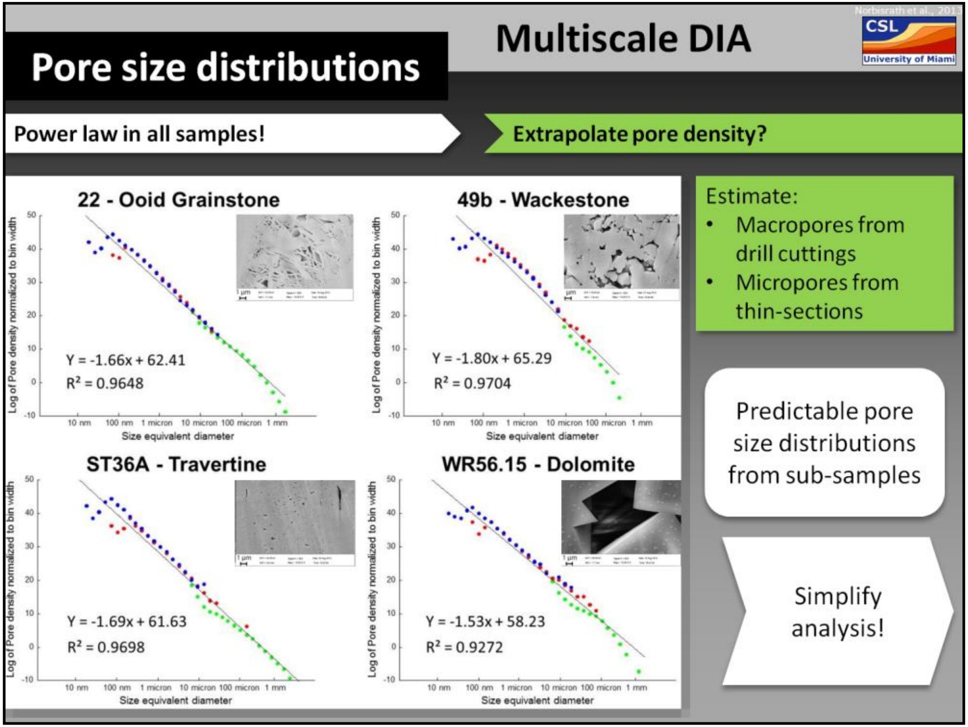
Power law - Fractal behavior ?

Self-similarity of pore space at different magnifications

$Y = -1.66x + 62.41$

$R^2 = 0.9648$

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.



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Multiscale DIA

Pore size distributions

Power law in all samples!

Extrapolate pore density?

22 - Ooid Grainstone

$Y = -1.66x + 62.41$

$R^2 = 0.9648$

49b - Wackestone

$Y = -1.80x + 65.29$

$R^2 = 0.9704$

ST36A - Travertine

$Y = -1.69x + 61.63$

$R^2 = 0.9698$

WR56.15 - Dolomite

$Y = -1.53x + 58.23$

$R^2 = 0.9272$

Estimate:

- Macropores from drill cuttings
- Micropores from thin-sections

Predictable pore size distributions from sub-samples

Simplify analysis!

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:

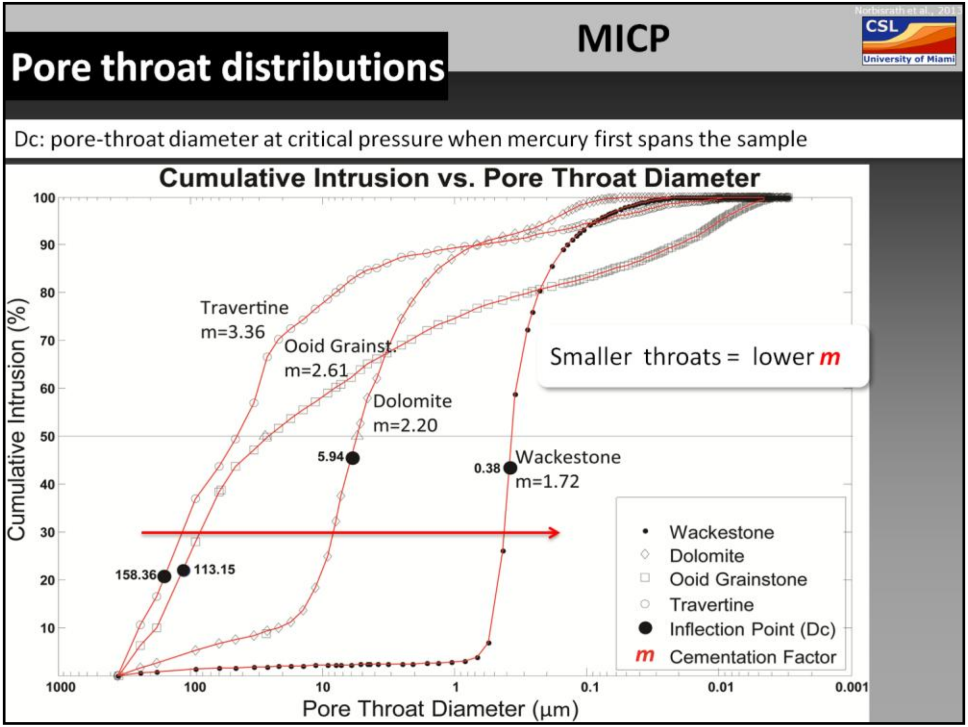
For example: estimate micropores from thin-sections, no time-consuming BIB-SEM needed!!

Or, estimate macropores from drill-cuttings; no expensive coring needed!

Outline

Key Points, Background and Motivations

1. **Broad-Ion-Beam milling + SEM (BIB-SEM)**
   - Nanometer-scale mosaics
2. **Multiscale Digital Image Analysis (MsDIA)**
   - Pore sizes / density / distribution
3. **Mercury Injection Capillary Pressure (MICP)**
   - Throat sizes



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Pore throat distributions

MICP

Dc: pore-throat diameter at critical pressure when mercury first spans the sample

Smaller throats = lower *m*

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 Dc, 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.

Outline

Key Points, Background and Motivations

1. **Broad-Ion-Beam milling + SEM (BIB-SEM)**
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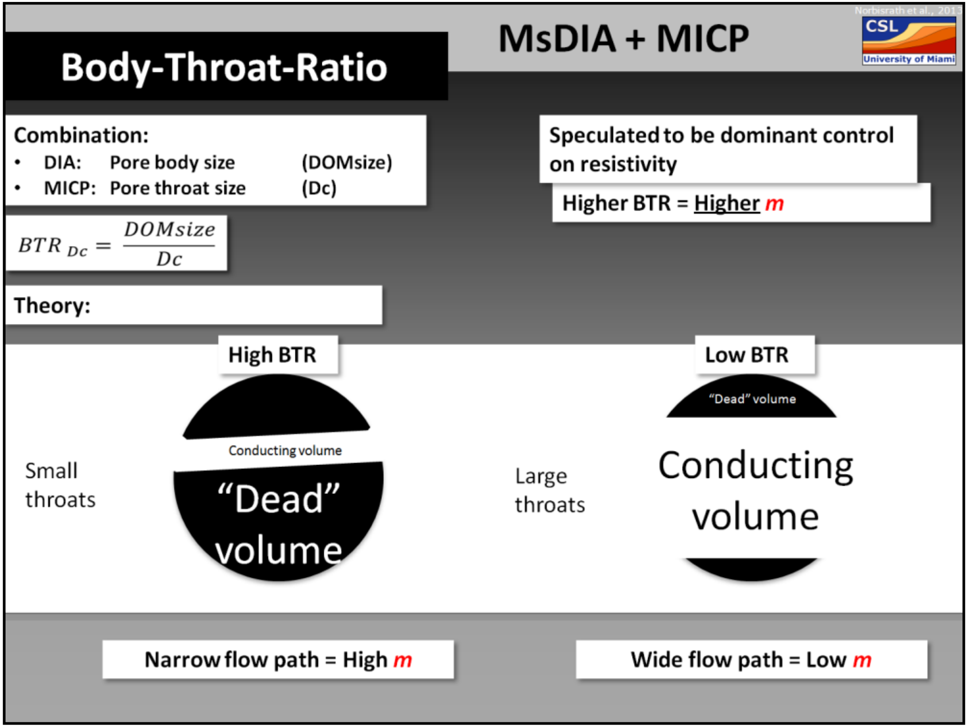
2. **Multiscale Digital Image Analysis (MsDIA)**
   - Pore sizes / density / distribution
3. **Mercury Injection Capillary Pressure (MICP)**
   - Throat sizes
4. **MsDIA + MICP**
   - Pore body-to-throat ratio (BTR)



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Body-Throat-Ratio

MsDIA + MICP

Combination:

- DIA: Pore body size (DOMsize)
- MICP: Pore throat size (Dc)

$$BTR_{Dc} = \frac{DOMsize}{Dc}$$

Speculated to be dominant control on resistivity

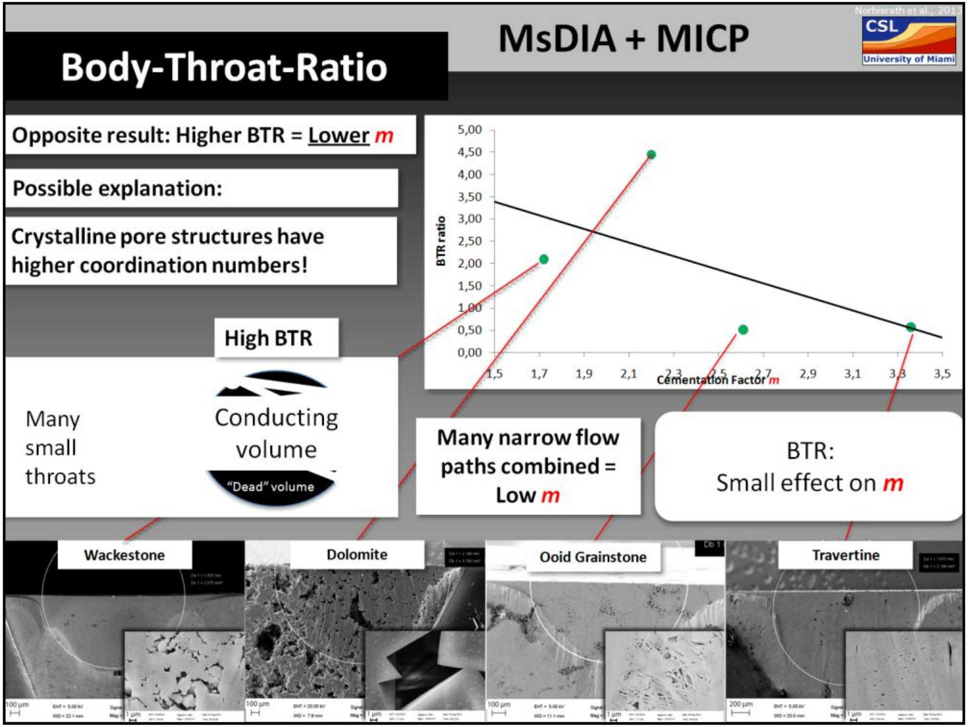
Higher BTR = Higher *m*

Theory:

Narrow flow path = High *m*

Wide flow path = Low *m*

Presenter's notes: Results are averaged pore network parameters.



Body-Throat-Ratio

MsDIA + MICP

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Opposite result: Higher BTR = Lower *m*

Possible explanation:

Crystalline pore structures have higher coordination numbers!

High BTR

Many small throats

Conducting volume

"Dead" volume

Many narrow flow paths combined = Low *m*

BTR: Small effect on *m*

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

- DOMsize: best correlation with *m*

Smaller pores/throats = Better electrical flow

- =/= Permeability

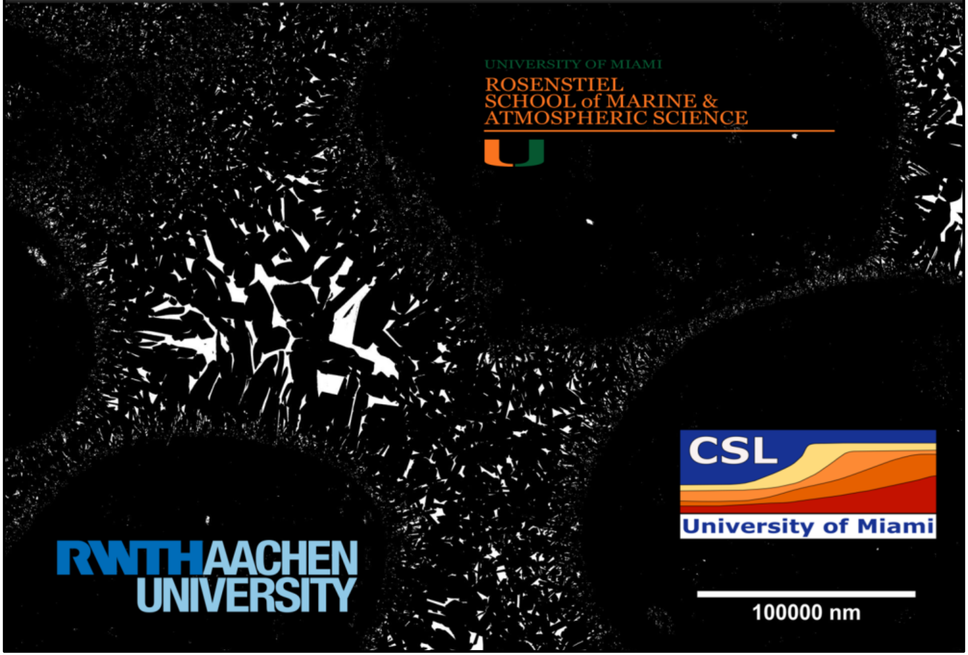
Body-Throat-Ratio = Weak impact on *m*

- Higher BTR =/= Higher *m*

Fractal pore size distributions in carbonates

- Estimate pore density from subsamples
  - Simplify analysis!

Acknowledgments



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