Webinars

Shale Reservoir Characterization: A basic Overview

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Lori Hathon
Assistant Professor of Petroleum Engineering, University of Houston

Dr. Hathon received a Bachelor of Science degree, with Honors, from Michigan State University, and a PhD in Sedimentary Petrology from the University of Missouri. After finishing her education, she spent six years in Exploration and Production with Amoco Production Company, and 20 years in fundamental rock properties research at Shell International E&P, Inc. Her areas of research include forward modeling of clastic reservoir properties (diagenesis, porosity, permeability), imaging and image analysis linked to rock properties measurements (digital rocks), and modeling of geomechanical properties (compressibility and strength). The last several years of her tenure at Shell were devoted to understanding organic matter catagenesis and porosity evolution in shale reservoirs.

Webinars

Bobby has a Master's degree in Physics from Mumbai University and one in Engineering Technology from University of North Texas. He has been doing SEM imaging work since college focusing on FIB/SEM since 1995. He taught courses in FIB at Microelectronic conferences from 1990 to 2012 and has extensive experience in imaging, including Cryo and Environmental Imaging. He has contributed to FIB related handbooks and been awarded three (3) US patents for Microelectronic. For seven years, Bobby worked exclusively on geologic application for Weatherford Labs, where he primarily carried out electron imaging of shales and organic matter characterization using a multitude of techniques and modalities. He has authored several publications in this field and earned one US and one Australian patent for Oil & Gas applications.

Bobby Kultaransingh

What makes a successful Shale Gas/Shale Oil play?

- Success factors include: TOC, porosity, brittleness
- Does quartz content control fracability?
- Does Total Organic Carbon (TOC) control porosity?
- Is organic porosity controlled by thermal maturity?
- What processes lead to the formation of organic matter porosity: kerogen transformation, bitumen cracking, or both?
- Does organic material becomes plastic at high maturity and does organic porosity collapse at a certain maturity called "the line of death"?
- Is primary migration out of shales, and deliverability of shale reservoirs, controlled by the presence of microcracks?
- Imaging studies can help us answer these questions

Agenda

- Empirical Methodology
  - Sampling locations
- Analysis
  - Non destructive
  - Destructive
- Crushed rock analysis
- Intact sample analysis
- Correlation

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Empirical Methodology

- Selecting Sampling locations
  - Helical CT scans of whole core
  - Core Gamma and XRF (for building geologic model; locations for drilling core plugs)
- Statistical sampling
  - Every foot-numerous redundant measurements
- Geologic sampling
  - Depending on facies-multiply sample each facies type to ensure that variability within facies is captured (~5 measurements per sample minimum)

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Empirical Methodology

- **Multimodal Imaging**
- SEM
  - SE/BSE/EDS/CL
    - SE – best for porosity imaging
    - BSE – compositional (density) information
    - EDS – bulk chemistry/mineralogy
    - CL – authigenic vs detrital phases
      - Very important for mechanical properties
  - Low voltage imaging
  - Segmentation implications

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Empirical Methodology

- **Multimodal Imaging**
- Transmitted and Reflected Light
  - Transmitted light
    - Relative volumes of carbonate (+phosphate and sulfate)/quartz (+feldspar)/Clay
    - Terrestrial vs intra-basinal constituents
  - Reflected light imaging for identification of OM types, volume percent pyrite
  - Fluorescent light imaging
    - OM identification at low maturity
    - Mineral phase fluorescence

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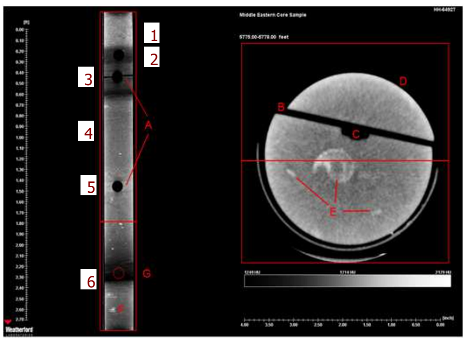
Empirical Methodology

- **Multimodal Imaging**
- Acoustic Microscopy
  - Velocity by lamina type in shale reservoirs (due to small particle and pore size, ray tracing not required)
- Laser Raman Microscopy
  - Identification of OM type
  - Estimation of OM Maturity
  - **All image types are registered to one another and to micro-CT of core plug and whole core Helical CT images**

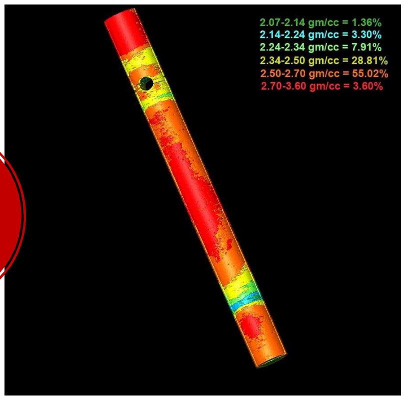
Helical CT scans

- Characterize
  - Density variations
  - Induced artifacts etc
- Lithotyping
  - Different densities
    - Plug locations for downstream tests

Helical CT scans



6 rock types are immediately recognizable from whole core CT images:
A function of relative gray levels and bedding/bioturbation



Helical CT scans (Segmented bulk densities)

Core Plug Analysis

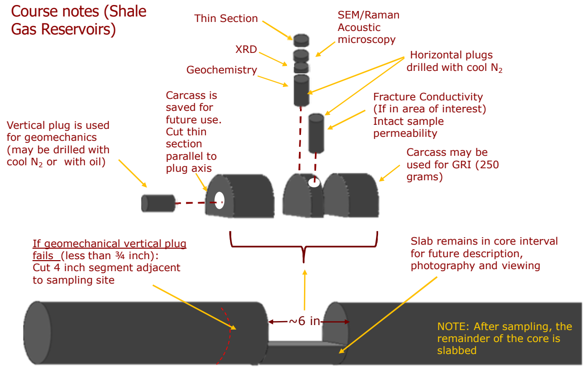
- Non destructive (core plug scale)
  - Micro-CT scans (~15 microns per pixel)
- If on horizontal core plug, use end trim sections
- If on vertical core plugs, cut thin section parallel to plug axis from carcass material
  - Characterize bedding plane perpendicular variability
- Destructive (pore scale)
  - 0.4" trim for dielectric property measurement
  - End trim thin section and mirror section for ion milling
    - Argon Ion Milling (notch vs entire end trim)
    - FIB/SEM (3D reconstruction)

Core plug analysis

- Destructive
- Thin Section
  - Transmitted and reflected light imaging of entire section
  - Organic Petrology
  - Raman spectroscopy for Organic matter typing and thermal maturity
- Mirror SEM sample (Argon Ion Milled)
  - Optical mosaic imaging: correlate with SEM mosaics
  - Use as navigational map in SEM
    - White and UV light for optical imaging
  - Raman spectroscopy for Organic matter typing and thermal maturity
  - Acoustic microscopy

Shale Sampling Methodology

Modified after Nautilus Course notes (Shale Gas Reservoirs)



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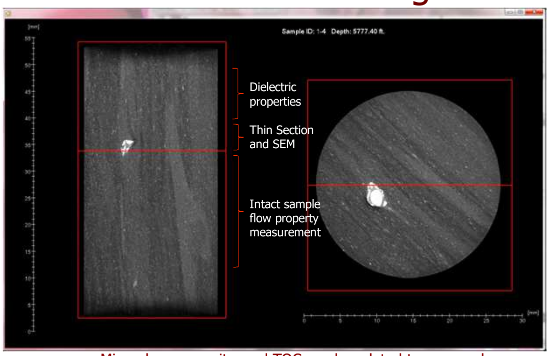
Micro-CT Scanning



Mineralogy, porosity, and TOC can be related to gray scale values for each lamina type present in the sample

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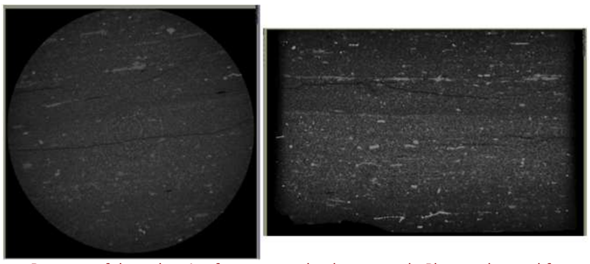
Micro-CT Scanning



Presence of through-going fractures can be documented. Plug can be used for permeability measurement if fractures can be healed with epoxy prior to measurement.

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Core to Pore

- Protocol for core to pore
- Argon Ion Milling (AIM) Entire plug end trim
- Registration of Multi modal Images
  - Micro-CT to Optical Mosaics
  - Thin Section to Optical Mosaics
  - Optical Mosaic to SEM (Low magnification)

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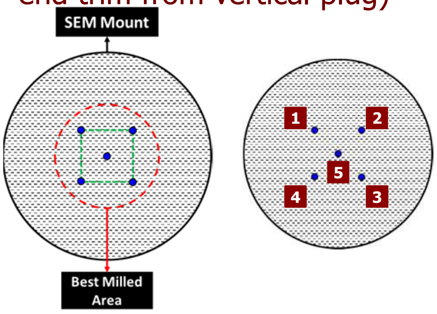
Mosaic set up (best when sample is homogeneous, e.g. end trim from vertical plug)



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Micro-CT and Optical mosaic images

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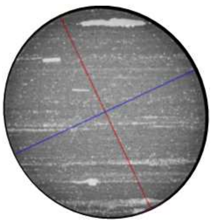
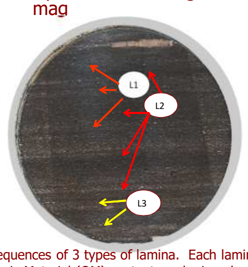
- Micro-CT scan



- Optical Mosaic image 16X mag



This one inch core plug contains repeated sequences of 3 types of lamina. Each lamina type is characterized in terms of porosity, total Organic Material (OM) content, and mineralogy via segmentation of multiple image mosaics from each lamina type. Porosity and OM content are of prime economic importance. Mineralogy (detrital and authigenic) is related to brittleness, a fundamental control on the effectiveness of wellbore stimulation. Porosity, organic content, and mineralogy are mapped to the 1" core plug CT scan for upscaling to the core plug scale.

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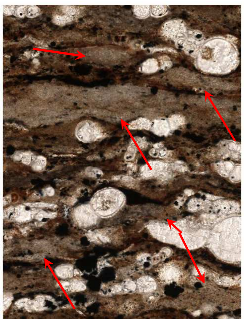
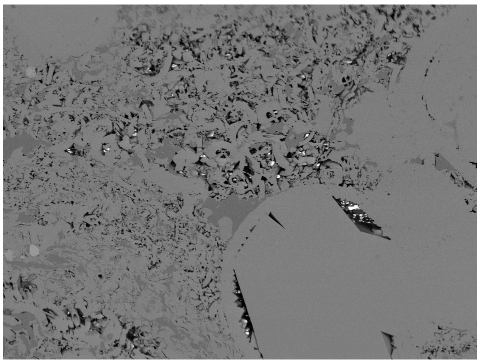
Transmitted Light Imaging of Lamina Types

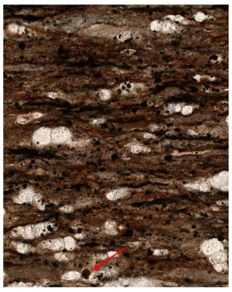


Transmitted light image of porous peloidal lamination (Type L3). This lamina type is highest in porosity (peloids, red arrows, are highly porous), has the highest carbonate content and the lowest clay and OM content of the lamina types identified.

SEM Imaging of Lamina Types

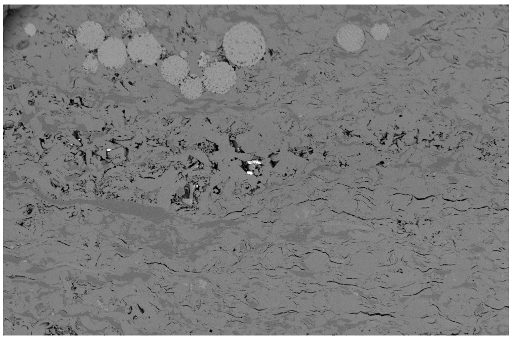


Transmitted Light Imaging of Lamina Types

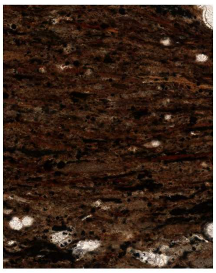


Transmitted light image of moderately peloidal, clay and organic matter rich lamination (type L1). This lamina type makes up the majority of the sample, and is intermediate in porosity, OM, and clay content between type 3 and type 2.

SEM Imaging of Lamina Types

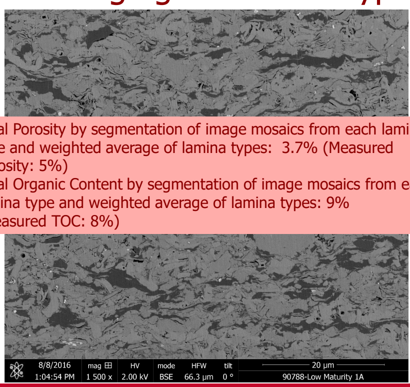


Transmitted Light Imaging of Lamina Types



Transmitted light image of clay and organic matter rich lamination (type L2). This lamina type is highest in TOC and clay mineral content (lowest in carbonate content), and lowest in porosity of the three lamina types identified.

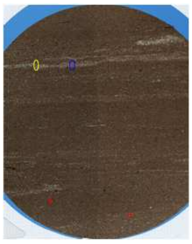
SEM Imaging of Lamina Types



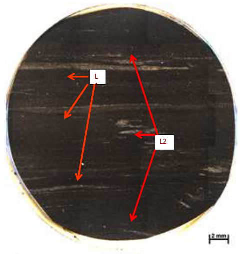
Total Porosity by segmentation of image mosaics from each lamina type and weighted average of lamina types: 3.7% (Measured Porosity: 5%)
Total Organic Content by segmentation of image mosaics from each lamina type and weighted average of lamina types: 9% (Measured TOC: 8%)

Thin section and Optical mosaic images

- Thin section mosaic (transmitted light)



- Optical Mosaic image 16X mag (reflected light)



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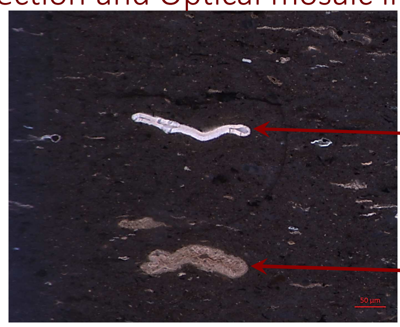
Thin section and Optical mosaic images

Images of region highlighted in yellow show the characteristics of the clay-rich (left) and silt-rich (right) lamina. These differences are further highlighted in mosaic images of the lamina taken at various magnifications and with different signals in the SEM.

Thin section and Optical mosaic images

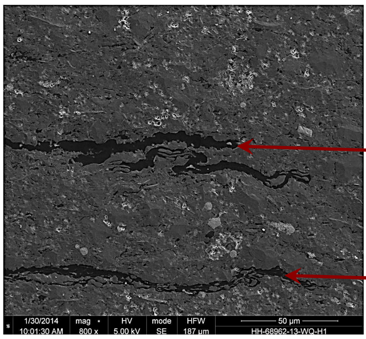


Tasmanites (algal) cyst

Agglutinated Foram – deep water below CCD

Large Tasmanites cycsts and dispersed low maturity SOM are visible at low magnification in fluorescent light in the Woodford Formation (Vitrinite Reflectance Equivalent ~ 0.48%).

Thin section and SEM images



Large Tasmanites cycsts and dispersed low maturity OM are visible at low magnification in the scanning electron microscope.

Comparison of lamina in SEM

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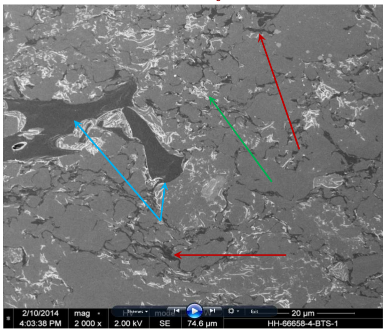

Typical view of fine grained, clay rich lamina. Segmented porosity is 1%, all associated with clay minerals (green arrows). Organic content is 6.5% including both terrestrial (vitrinite; blue arrows) and algal (red arrows) material.

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Comparison of Lamina in SEM

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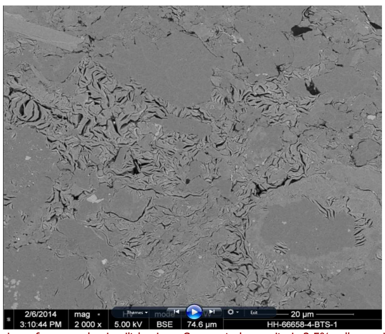

Typical view of porous clay in silt lamina. Segmented porosity is 2.5%, all associated with clay minerals. Organic content is less than 1% in this laminar type.

Pore↔Core

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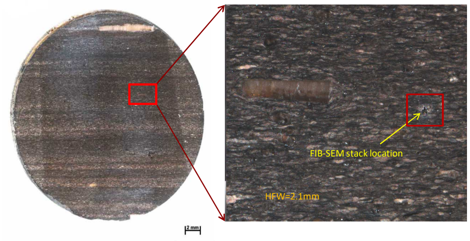
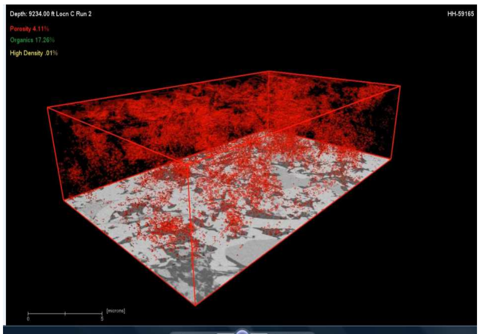


FIB volumes are acquired in locations where extensive 2D characterization has been performed. 2D image analysis results are used together with analysis of the FIB volume to put FIB analysis results into a larger context.

Multiscale Imaging

20 µm x 7 µm FIB/SEM Volume of Shale Reservoir



Low Voltage Imaging Discussion Agenda

- Contemporary Electron imaging
  - Voltages/Energies used
  - Advantages/disadvantages
- Why low voltage?
  - 2D porosity measurements
  - More accurate/representative surface images
- Experimental details
- Comparison of images
  - Comparison of porosity numbers
- How low can you go?
- Summary

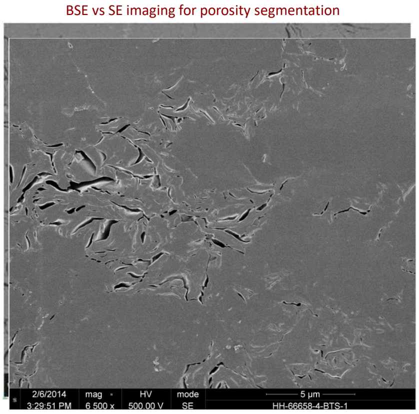
Contemporary Electron Imaging

- Advantages (10-20Kev)
  - Fewer lens aberrations
  - Energy dispersive spectroscopy analysis
- Disadvantages
  - Deeper sample penetration
  - Surface features fuzzy
    - Subsequently, image segmentation difficult

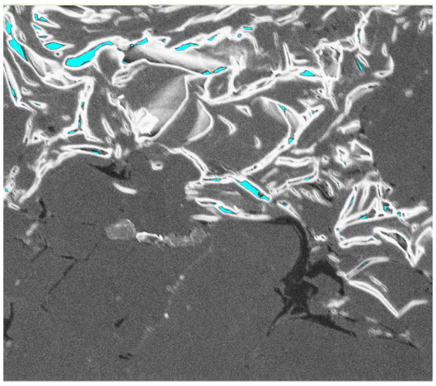
Why Low Voltage?

- Image and study/analyze surface features of given rock (after Argon Ion Milling)
- Study rock fabric
  - Conduct 2 D imaging (surface features accentuated, shallow sample penetration).
  - Image/scout top surface of shale sample (s)..
  - Scout locations for 3D FIB-SEM datasets..
  - Obtain high resolution images of representative areas..
  - Conduct 2D surface porosity measurements

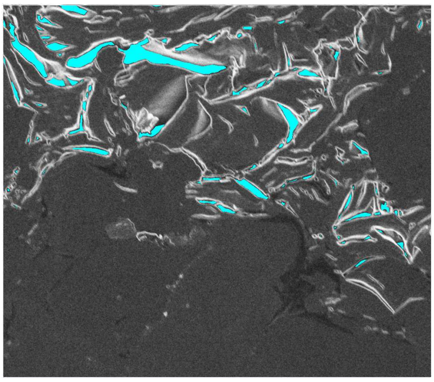
BSE vs SE imaging for porosity segmentation



Porosity 0.63% 6500x, 2kv SE image



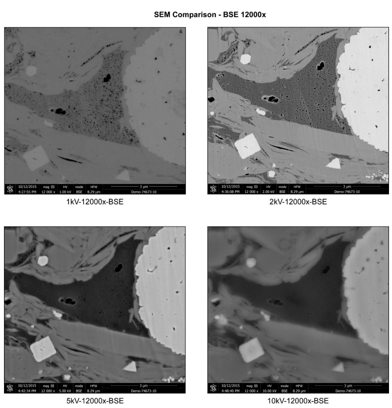
Porosity 1.72-2.32% 6500x, 1kv SE image



Multiple interpretations of pore backs are shown.

Imaging at Different voltages

Low Voltage Imaging Summary Slide



Conclusions & Summary

- Low voltage imaging feasible for studying 2D porosity accurately ..
- Not without challenges..
- Can be part of comprehensive package
  - EDS scan
  - 2D porosity numbers
  - AS &V at same location
    - Should result in more accurate picture

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Other Imaging Modalities

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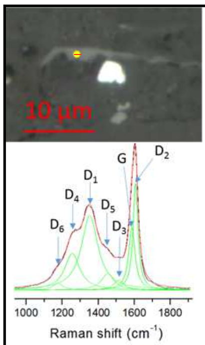
Raman spectrum of marine organic material



Deconvolution of Raman spectrum of a marine organic material at 1.2% Ro.

Table 1 [6]. Explanation of G, D1, D2, D3, D4 band.

First-order Raman bands and vibration modes reported for soot and graphite (vs = very strong, s = strong, m = medium, w = weak)

Band[a]	Raman shift (cm^{-1})			Vibration mode[b]
	Soot	Disordered graphite[c]	Highly ordered graphite[d]	
G	~1580 cm^{-1}, s	~1580 cm^{-1}, s	~1580 cm^{-1}, s	Ideal graphitic lattice (E_{2g}-symmetry) [6,17]
D1 (D)	~1350 cm^{-1}, vs	~1350 cm^{-1}, m	–	Disordered graphitic lattice (graphene layer edges, A_{1g} symmetry) [6,17]
D2 (D′)	~1620 cm^{-1}, s	~1620 cm^{-1}, w	–	Disordered graphitic lattice (surface graphene layers, E_{2g}-symmetry) [17]
D3 (D″, A)	~1500 cm^{-1}, m	–	–	Amorphous carbon (Gaussian [26] or Lorentzian [3,18,27] line shape)
D4 (I)	~1200 cm^{-1}, w	–	–	Disordered graphitic lattice (A_{1g} symmetry) [10], polyenes [3,27], ionic impurities [18]

[a] Alternative band designations of earlier studies are given in brackets.
[b] Lorentzian line shape unless mentioned otherwise.
[c] Polycrystalline graphite (<100 nm) and boron-doted HOPG [17].
[d] Single graphitic crystals (>100 nm) and HOPG [17].

Table 2 [7]. A more detailed deconvolution of Raman spectra of carbonaceous materials.

Summary of peak/band assignment

Band name	Band position, cm^{-1}	Description	Bond type	References
G_L	1700	Carbonyl group C=O	sp^2	[28]
G	1590	Graphite E_{2g}^2; aromatic ring quadrant breathing; alkene C=C	sp^2	[4,28]
G_R	1540	Aromatics with 3–5 rings; amorphous carbon structures	sp^2	This work
V_L	1465	Methylene or methyl; semi-circle breathing of aromatic rings; amorphous carbon structures	sp^2, sp^3	[5,12,28]
V_R	1380	Methyl group; semi-circle breathing of aromatic rings; amorphous carbon structures	sp^2, sp^3	[28]
D	1300	D band on highly ordered carbonaceous materials; C–C between aromatic rings and aromatics with not less than 6 rings	sp^2	[22,28]
S_L	1230	Aryl–alkyl ether; para-aromatics	sp^2, sp^3	[28]
S	1185	$C_{aromatic}$–C_{alkyl}; aromatic (aliphatic) ethers; C–C on hydroaromatic rings; hexagonal diamond carbon sp^3; C–H on aromatic rings	sp^2, sp^3	[12,28,31]
S_R	1060	C–H on aromatic rings; benzene (ortho-di-substituted) ring	sp^2	[28]
R	960–800	C–C on alkanes and cyclic alkanes; C–H on aromatic rings	sp^2, sp^3	[28]

[6] A. Sadezky, H. Muckenhuber, H. Grothe, R. Niessner, U. Pöschl, Raman microspectroscopy of soot and related carbonaceous materials: Spectral analysis and structural information, Carbon, Volume 43, Issue 8, July 2005
[7] Xiaojiang Li, Jun-ichiro Hayashi, Chun-Zhu Li, FT-Raman spectroscopic study of the evolution of char structure during the pyrolysis of a Victorian brown coal, Fuel, Volume 85, Issues 12–13, September 2006

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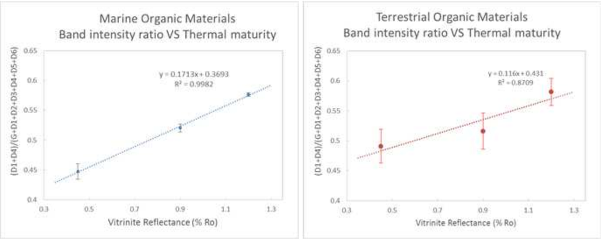
The Intensity ratio (D1+D4)/(G+D1+D2+D3+D4+D5+D6) trends with thermal maturity



The trend corresponds with the result from [7].

[7] Lahfid, A., Beyssac, O., Deville, E., Negro, F., Chopin, C. and Goffé, B. (2010), Evolution of the Raman spectrum of carbonaceous material in low-grade metasediments of the Glarus Alps (Switzerland). Terra Nova, 22: 354–360

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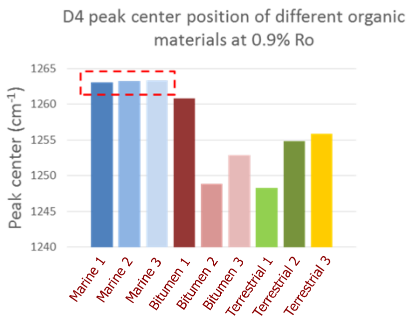
D4 peak center position as an indicator



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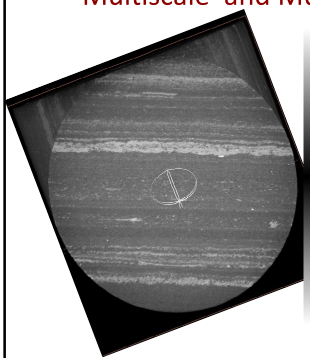
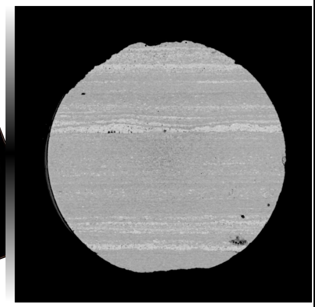
Multiscale and Multi Modal Imaging



Micro CT Image of Core Plug



Acoustic Microscopy image of ion milled end trim surface of sample at left illustrating impedance at the top surface of the sample

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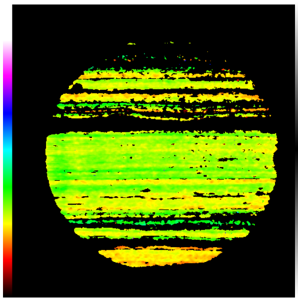
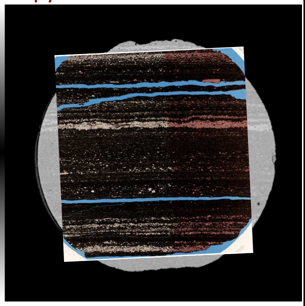
Multiscale and Multi Modal Imaging: Acoustic Microscopy



Acoustic velocity of lamina types: clay and organic matter content from SEM image analysis will be used to develop velocity models.



Impedance image of the top surface of the sample. High reflectivity of carbonate rich (bright lamina) results in limited energy reaching the base of the sample.

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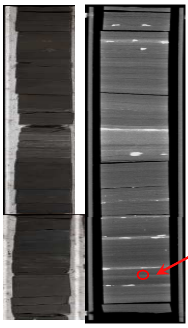
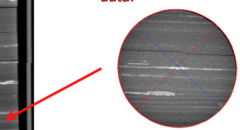
Multiscale Imaging

Registered Whole Core and Core Plug Scan
Images Shown at SPE Forum, 2014

~1m



Bulk properties calculated from high resolution image analysis are mapped to the whole core CT scan. Porosity, TOC, mineralogy, can be used to model bulk density, and acoustic properties at the whole core scale for ties to logging data.



2.5cm ~35 μm/pixel

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Summary of Techniques

Technique	Strengths	Weaknesses	Cost
Helical CT	Rapid, Required for upscaling to wellbore scale	Low Resolution	$$
Core Gamma	Core-log calibration		$
Handheld XRF	TOC proxies	Cannot distinguish oxidation state or detrital vs authigenic phase	$
MicroCT	High resolution, necessary intermediate scale of observation for upscaling, nondestructive interior ROI scanning	Not at pore scale	$ - $$

Summary of Techniques

Technique	Strengths	Weaknesses	Cost
SEM 2D mosaics	Porosity,TOC,mineralogy, detrital versus authigenic phases	No 3D connectivity	$$
FIB/SEM	3D imaging of pore space and connectivity	Very small area difficult to upscale	$$$
Thin Section	Mineralogy, vitrinite reflectance, OM fluorescence, bedding/sedimentary features	Limited resolution, biased estimates of size	$
Raman Microscopy	Organic matter type, thermal maturity estimation	Deconvolution protocols not well established	$
Acoustic Microscopy	Laminar scale acoustic properties	Can't be used in "hybrid" reservoirs	$

Linking Multiscale Multimodal Imaging And Rock Property Measurements

83 joint years of industry experience suggest that we can best understand and model economically important rock properties through joint measurements and registered multimodal/multiscale imaging of rock materials and quantitative image analysis.

Thank you!

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Webinars

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