

Pore-Scale Imaging of Solid Bitumens: Insights for Unconventional Reservoir Characterization*

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Abstract

Characterizing unconventional reservoirs involves the investigation of a wide range of potential source rock targets at various stages of thermal maturity. These samples may contain a mixture of kerogen, bitumen, oil and pyrobitumen within their fabric. Thus, it is critical that we properly identify and examine each organic phase in order to better understand reservoir properties. In the present study, we have selected samples of gilsonite from a naturally occurring solid hydrocarbon deposit to serve as an analog for characterizing the bitumen phase of generation.

Gilsonite is an aromatic-asphaltic solid bitumen that is found in vertical veins along the eastern portion of the Uinta Basin, Utah. It is thought to be an early generation product from oil-prone Green River Shale source beds and is similar to low maturity crude oil in composition. It has a high nitrogen content, low sulfur content, high melting point (fusibility) and is soluble in organic solvents. We have used a variety of analytic methods to characterize this material, including standard optical organic petrology and scanning electron microscopic imaging to examine the occurrence of organic porosity.

Optical organic petrology analysis using both air and oil immersion objectives show that the polished gilsonite surfaces are typically dark grey and featureless. Macerals and inorganics are absent. Visual estimates suggest that fractures make up approximately 1% of the conchoidal fracture plane, while the pencillated variety contains approximately 2% fractures along with 5% shallow pits. Scanning electron microscopic images also show the occurrence of fractures within gilsonite, but the matrix contains no evident organic porosity.

The results of our analyses suggest that, unlike pyrobitumen, pre-oil solid bitumen represented by gilsonite was found to contain no significant occurrences of organic nanoporosity within its matrix. Gilsonite does have minor pitting and fractures, but these do not represent an effective interconnected pore network and are probably artifacts of weathering/sampling. Thus, this material would not represent a potential candidate for in-situ hydrocarbon storage capacity. Whether this is typical of all naturally occurring solid bitumen is debatable, considering that gilsonite

has undergone some secondary alteration via devolatilization and limited biodegradation. Nevertheless, the pore-scale imaging of this solid bitumen provides potentially important new insights for unconventional reservoir characterization.

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LABORATORIES

PORE-SCALE IMAGING OF SOLID BITUMENS: INSIGHTS FOR UNCONVENTIONAL RESERVOIR CHARACTERIZATION

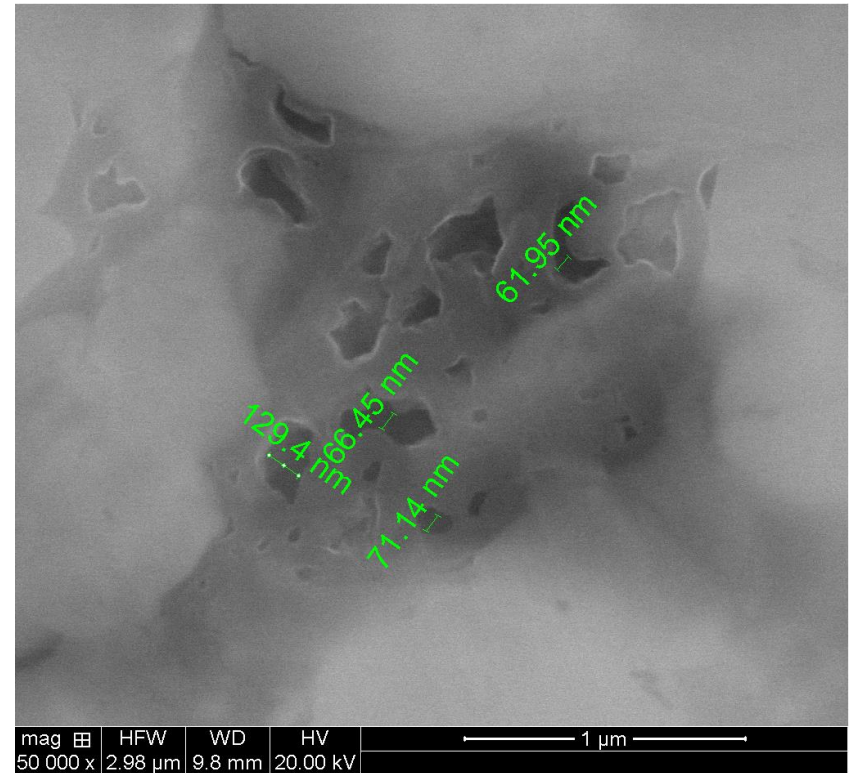
Tim E. Ruble¹, Kultaransingh (Bobby) Hooghan¹, William Dorsey¹, Wayne R. Knowles¹ and Christopher D. Laughrey²

¹ Weatherford International Ltd., Houston, TX

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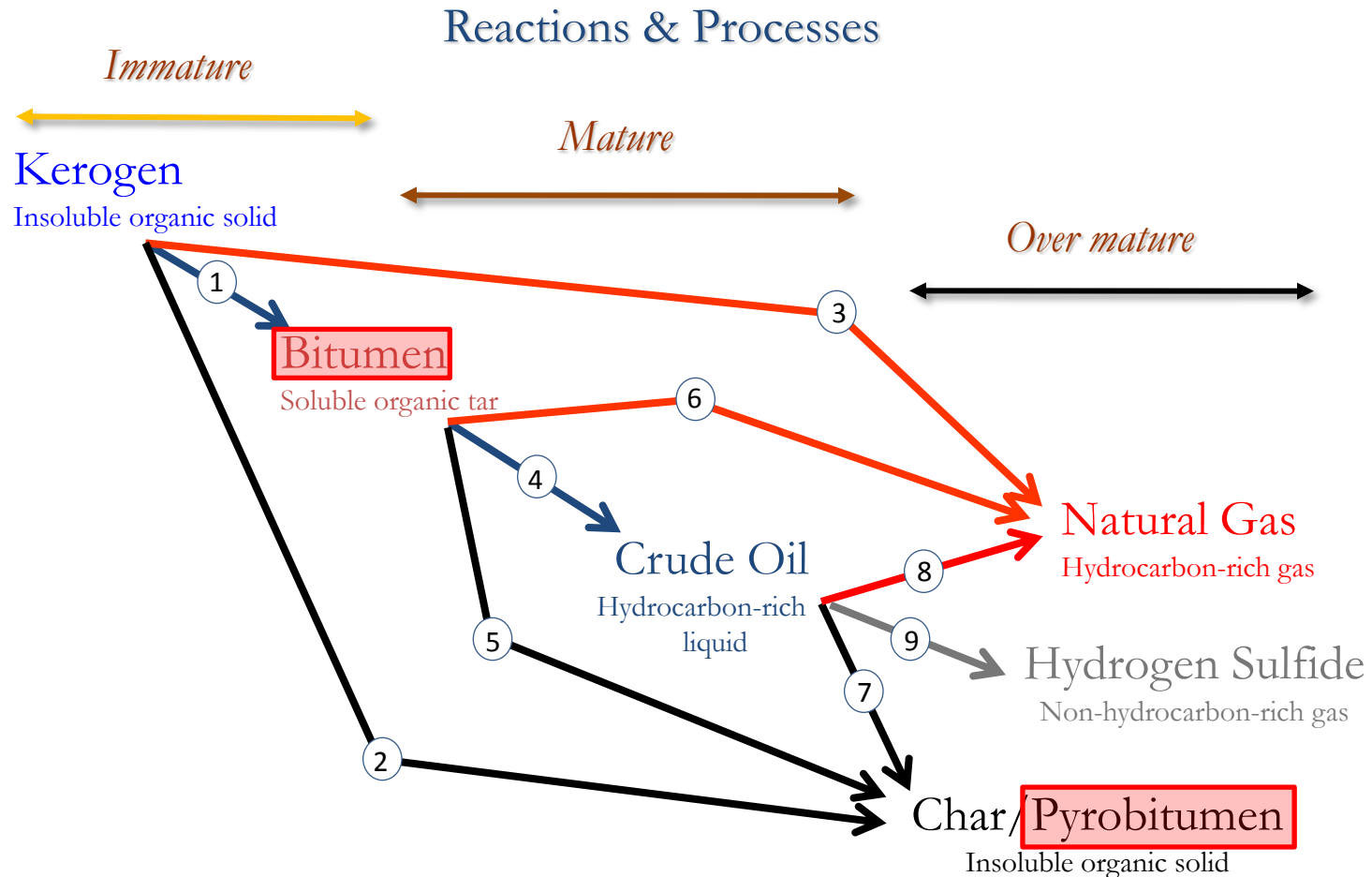
Investigation of Gilsonite may aid our understanding of Unconventional Oil Systems

- Better understand the nature and occurrence of bitumen.
- Develop testing methods to distinguish movable oil versus immobile bitumen.
- Use SEM imaging to examine the occurrence of organic porosity within bitumen.
- Understand role of water emulsions in bitumen occurrence and mobility.



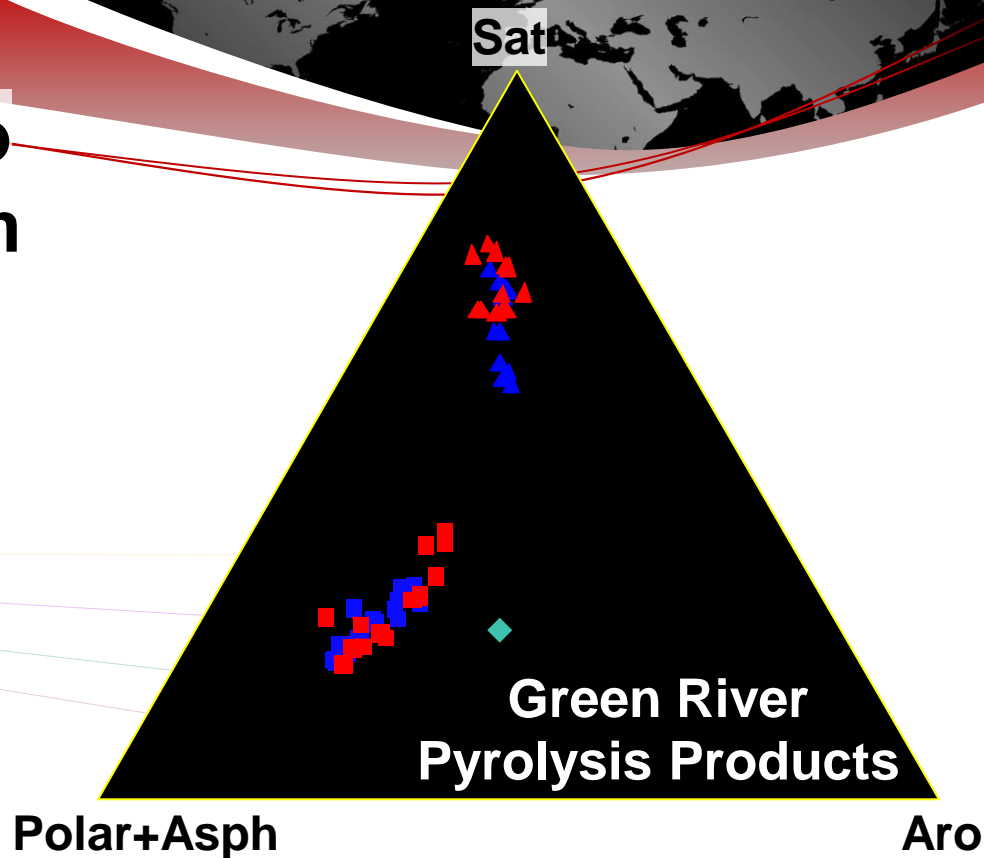
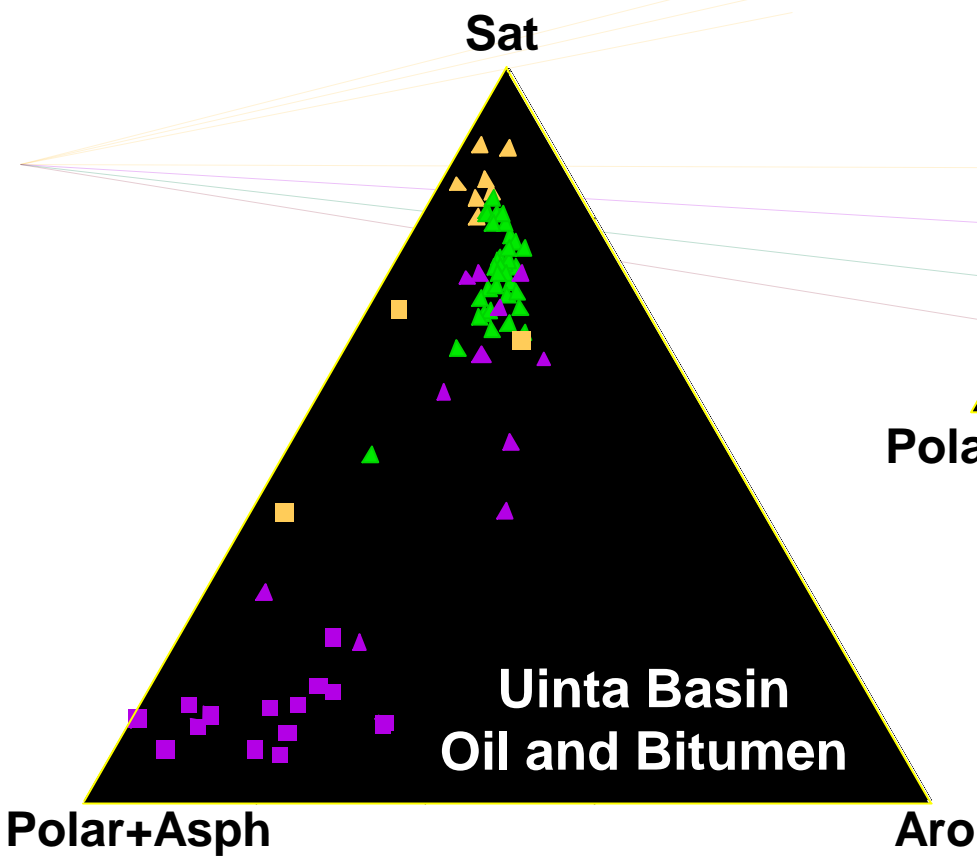
PYROBITUMEN HAS
NANOPORES.....WHAT ABOUT
BITUMEN?

Hydrocarbon Generation



from Lewan (2011)

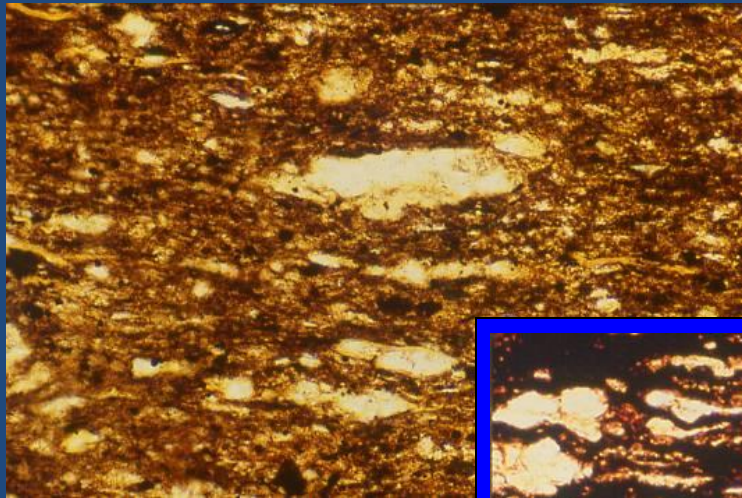
Why is it important to distinguish “oil” from “bitumen”?



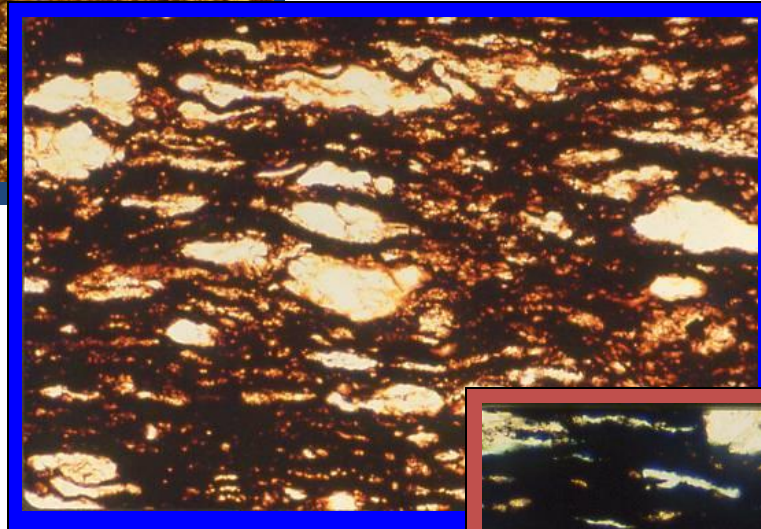
Because nature does!

from Ruble et al. (2001)

Primary petroleum migration

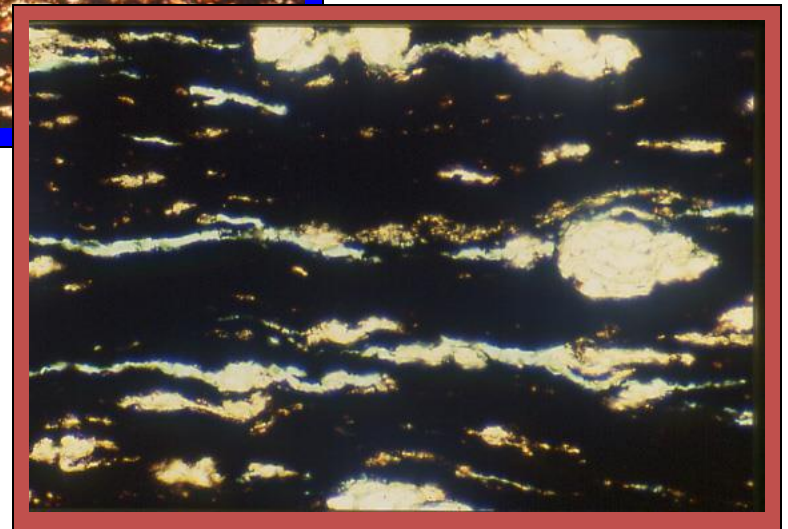


immature unheated



kerogen-bitumen
300°C/72h

125 μm

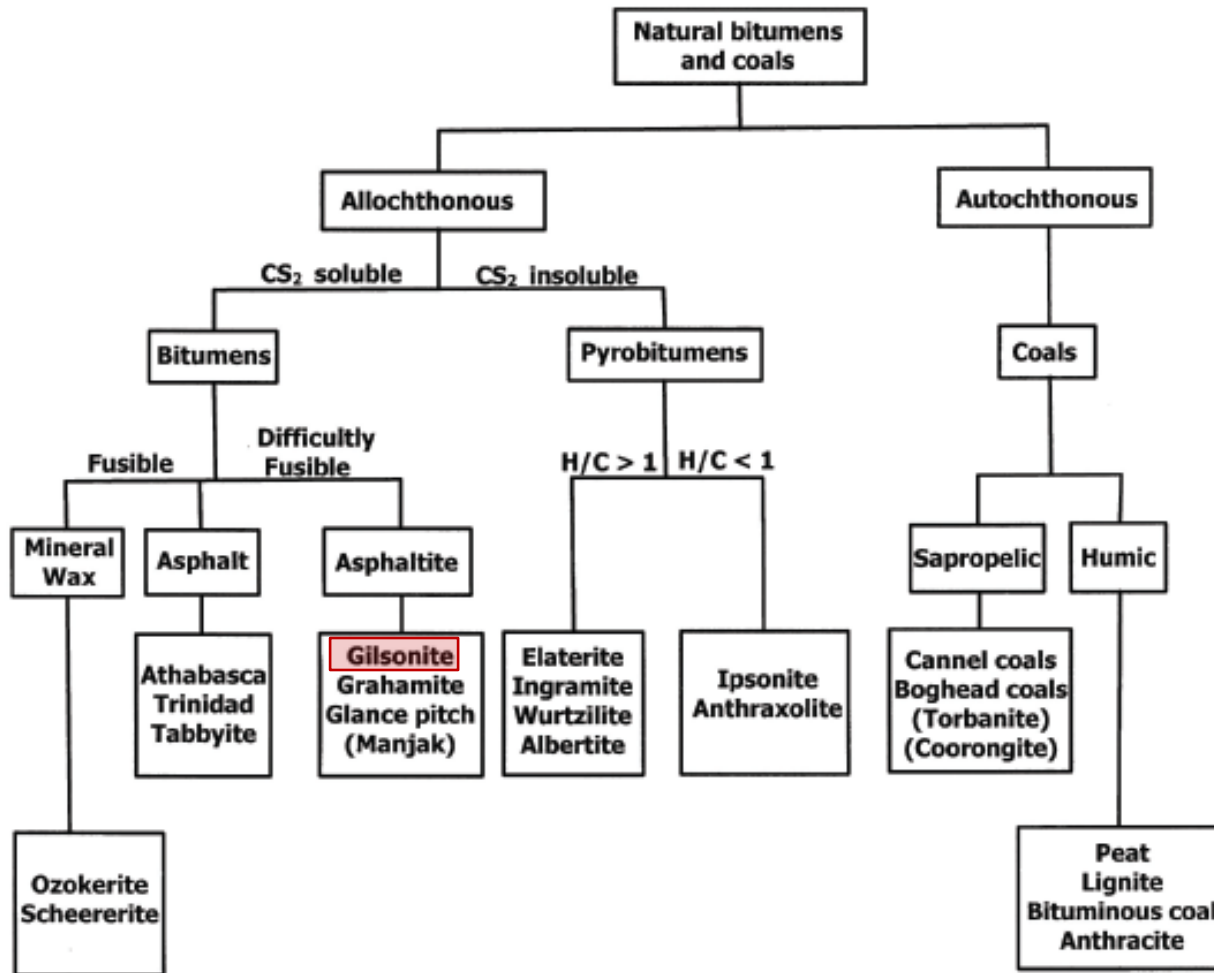


bitumen-oil 352°C/72h

Hydrous Pyrolysis of Woodford Shale Cores

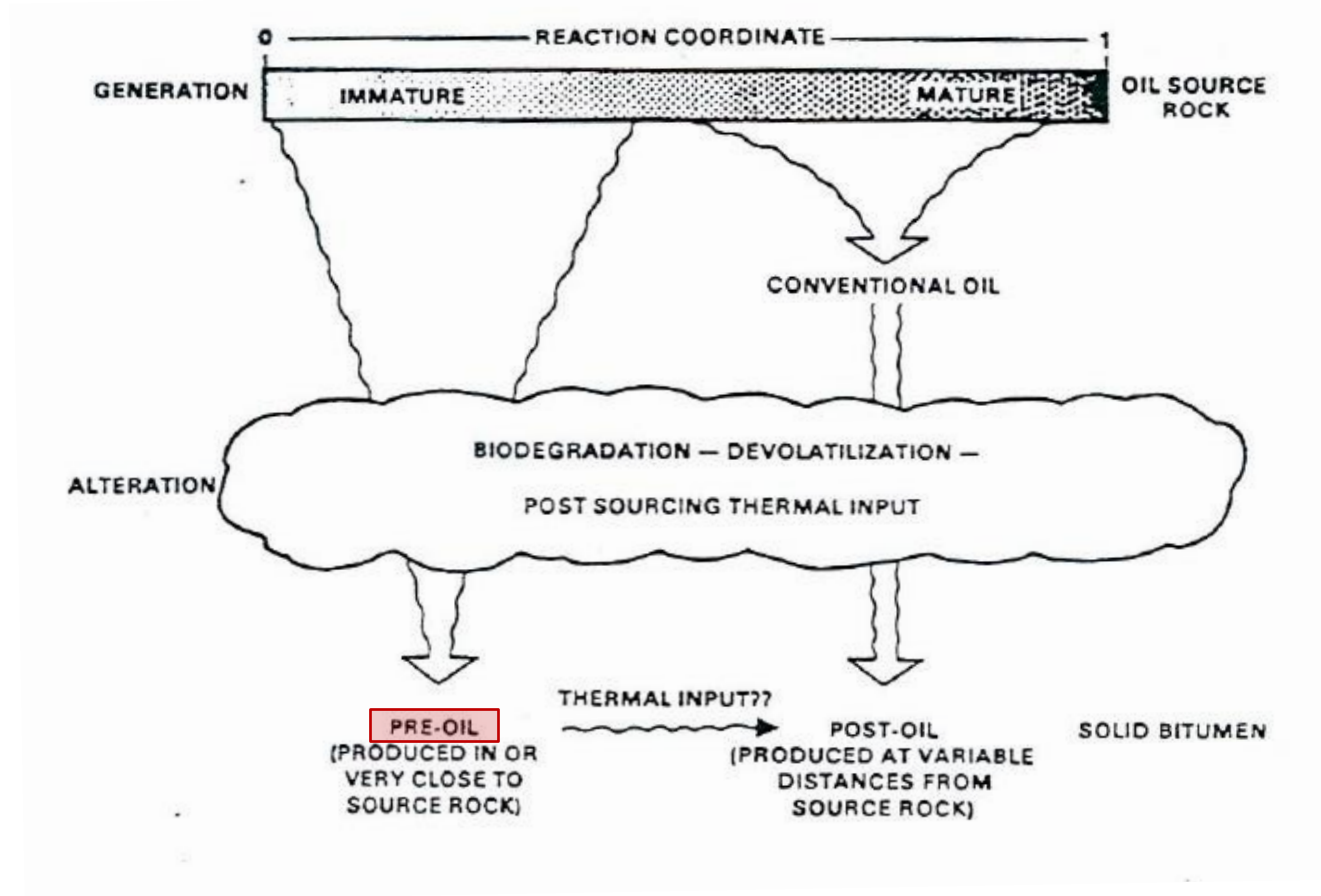
from Lewan (1987)

Generic Classification Scheme for Bitumens



from Hunt (1979)

Genetic Classification Scheme for Bitumens



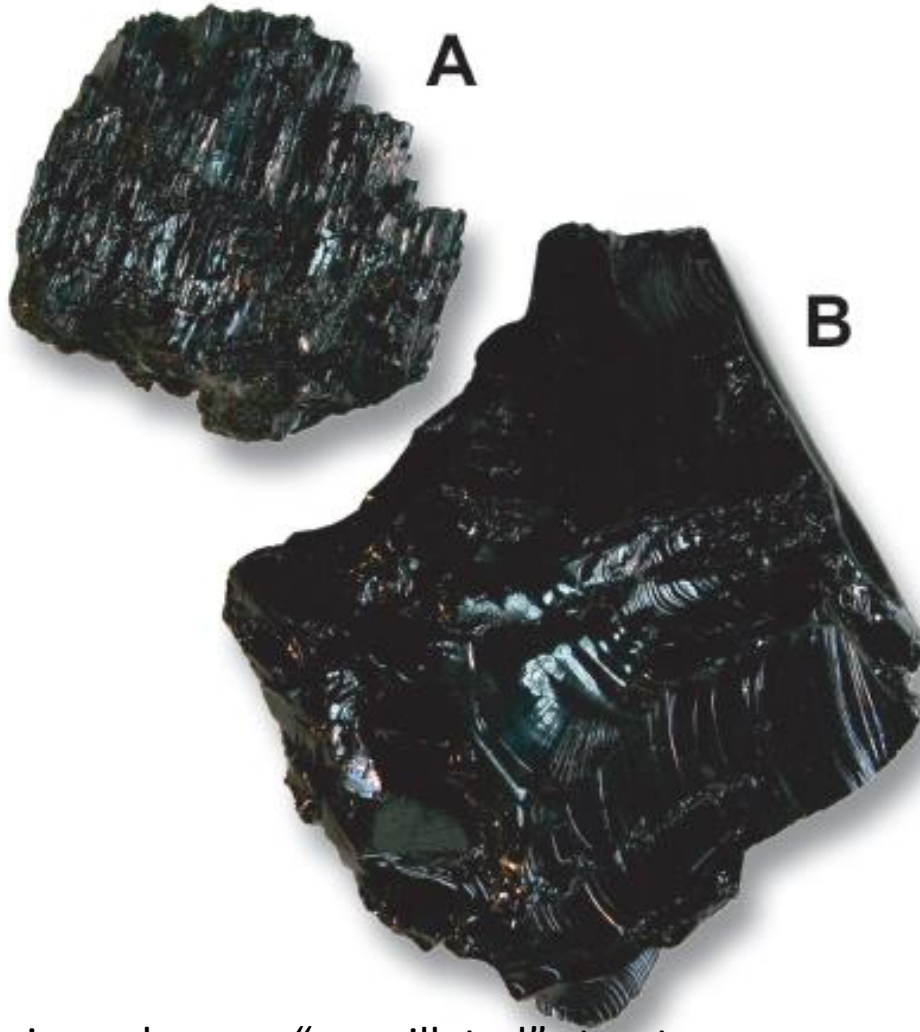
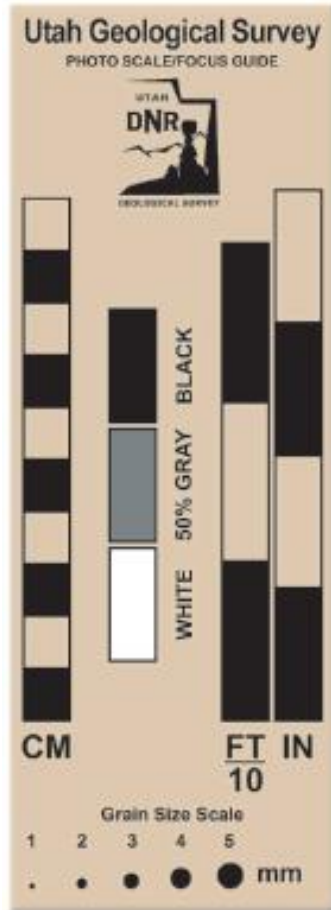
from Curiale (1986)

Gilsonite Characteristics

- Solid to semi-solid
- Occurs in dikes or veins
 - Up to 5m x 40km
- Similar to low maturity crude oil in composition
- Aromatic-asphaltic hydrocarbons
- High nitrogen, low sulfur content
- Soluble in organic solvents (CS₂)
- High melting point (fusibility)



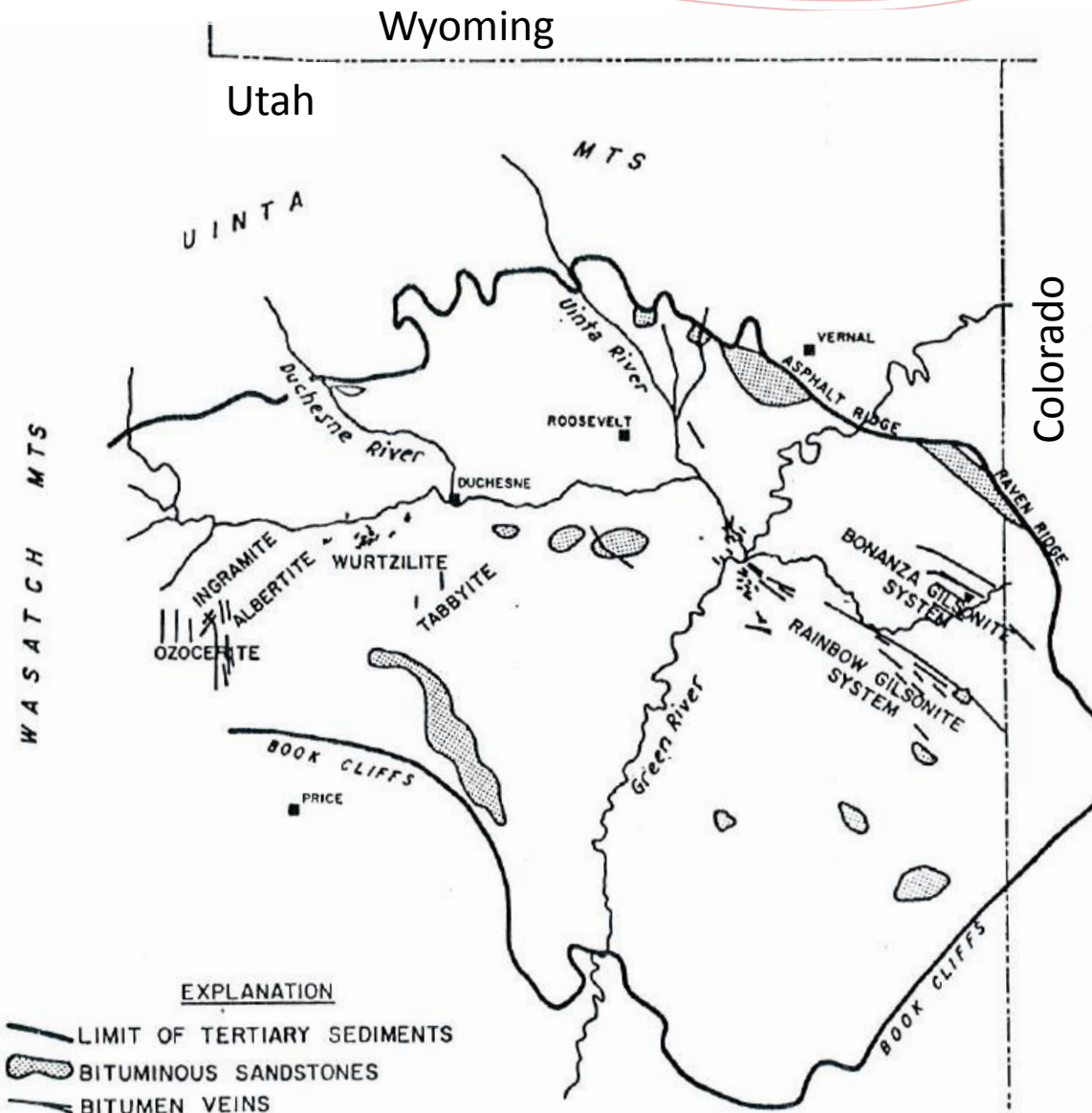
Gilsonite Textures



- A) Gilsonite showing columnar "pencillated" structure
- B) Gilsonite select grade showing "conchoidal" fracturing

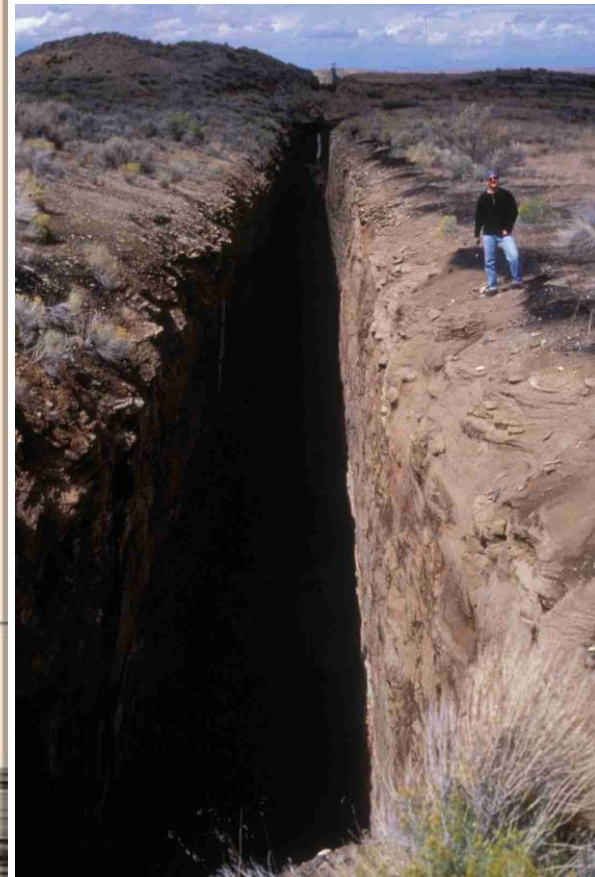
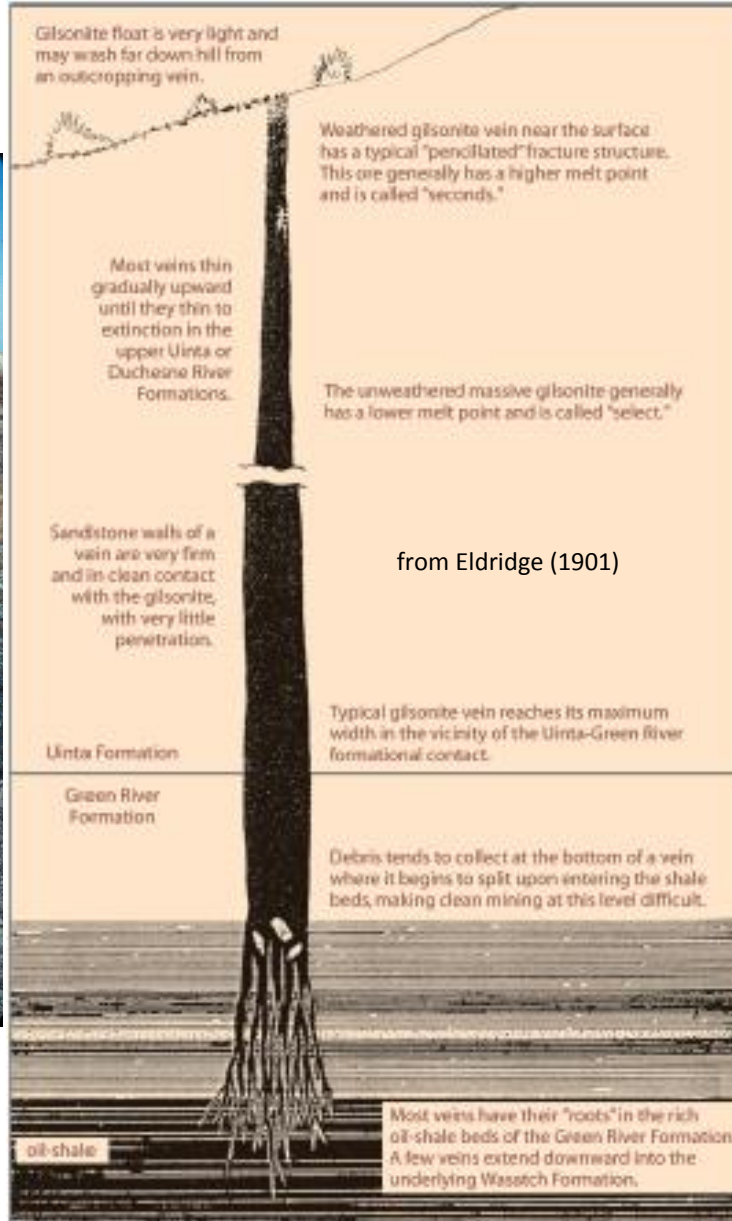
from Boden & Tripp (2012) UGS Special Study 141

Uinta Basin Solid Hydrocarbon Deposits

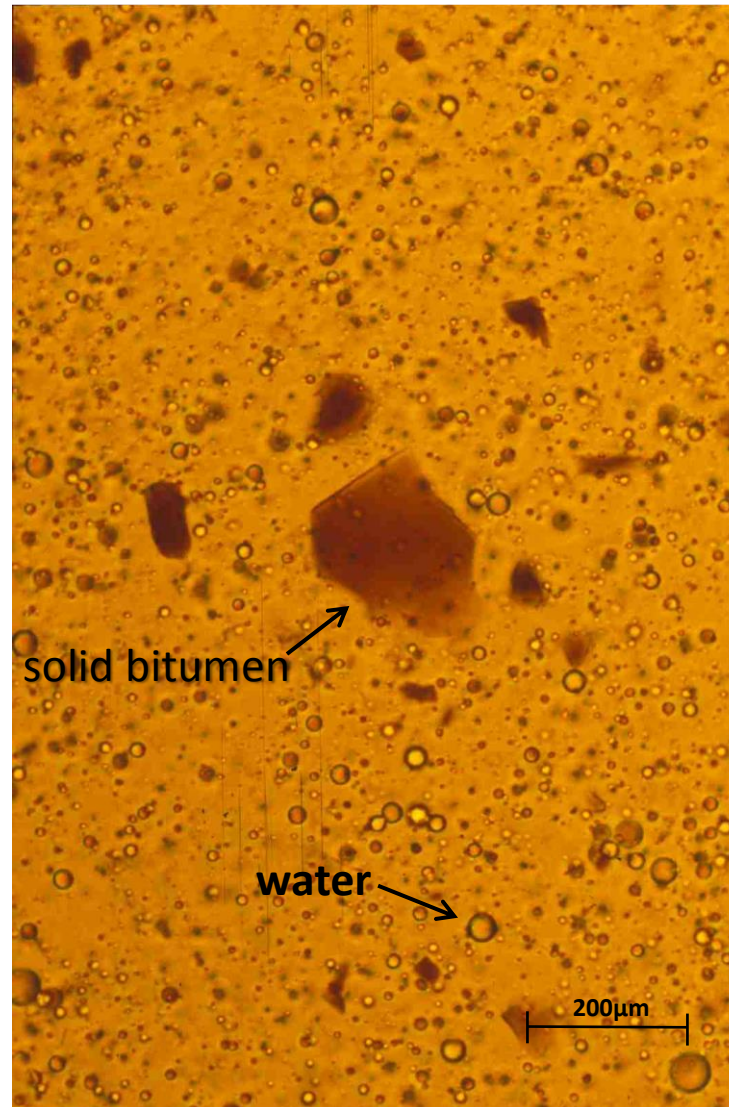


from Hunt (1963)

Gilsonite Veins



Gilsonite Tar - a mobile bitumen phase



Viscous
Water in Bitumen/Oil
Emulsion

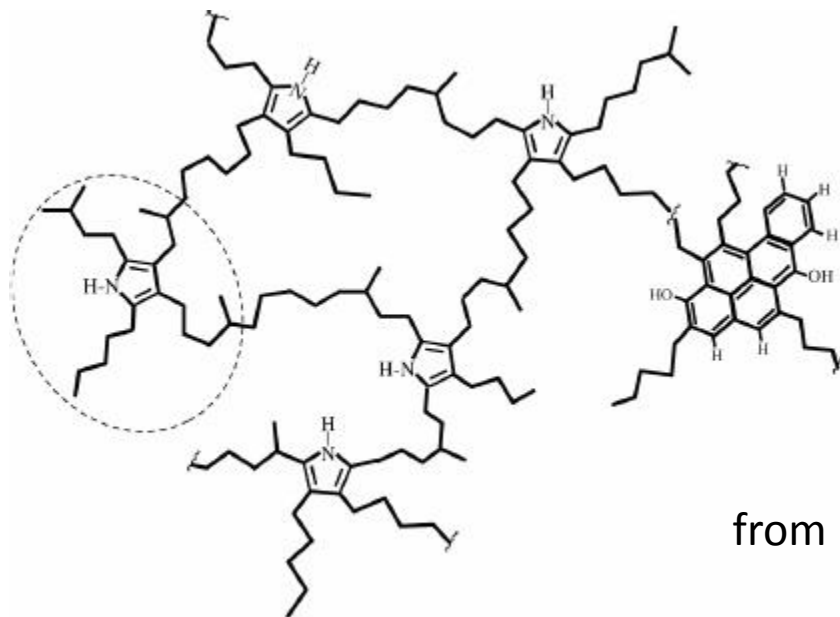
Actively seeping
into excavated
gilsonite veins

Gilsonite precursor
prior to solidification

Origin of Gilsonite

1. Lacustrine oil shale kerogen → bitumen
 - Low thermal stress
 - Pre-oil generation stage
2. Expulsion from shale source rock – exceeds retention capacity – creates hydraulic fractures through extensive stress, viscous bitumen migrated vertically through fractures
3. Secondary alteration after injection into veins
Solidification, devolatilization, possible biodegradation
→ **Solid Bitumen**

Gilsonite Chemical Structure Model



from Helms et al. (2012)

Model of the main components of gilsonite structure. A typical “monomer unit” containing one pyrrolic aromatic ring and 21 carbons is outlined by the dashed ellipse.

Gilsonite exists as a highly diverse mixture of molecules of various size and structure. The model structure shown here represents the high molecular weight fraction, while smaller fragments are also common.



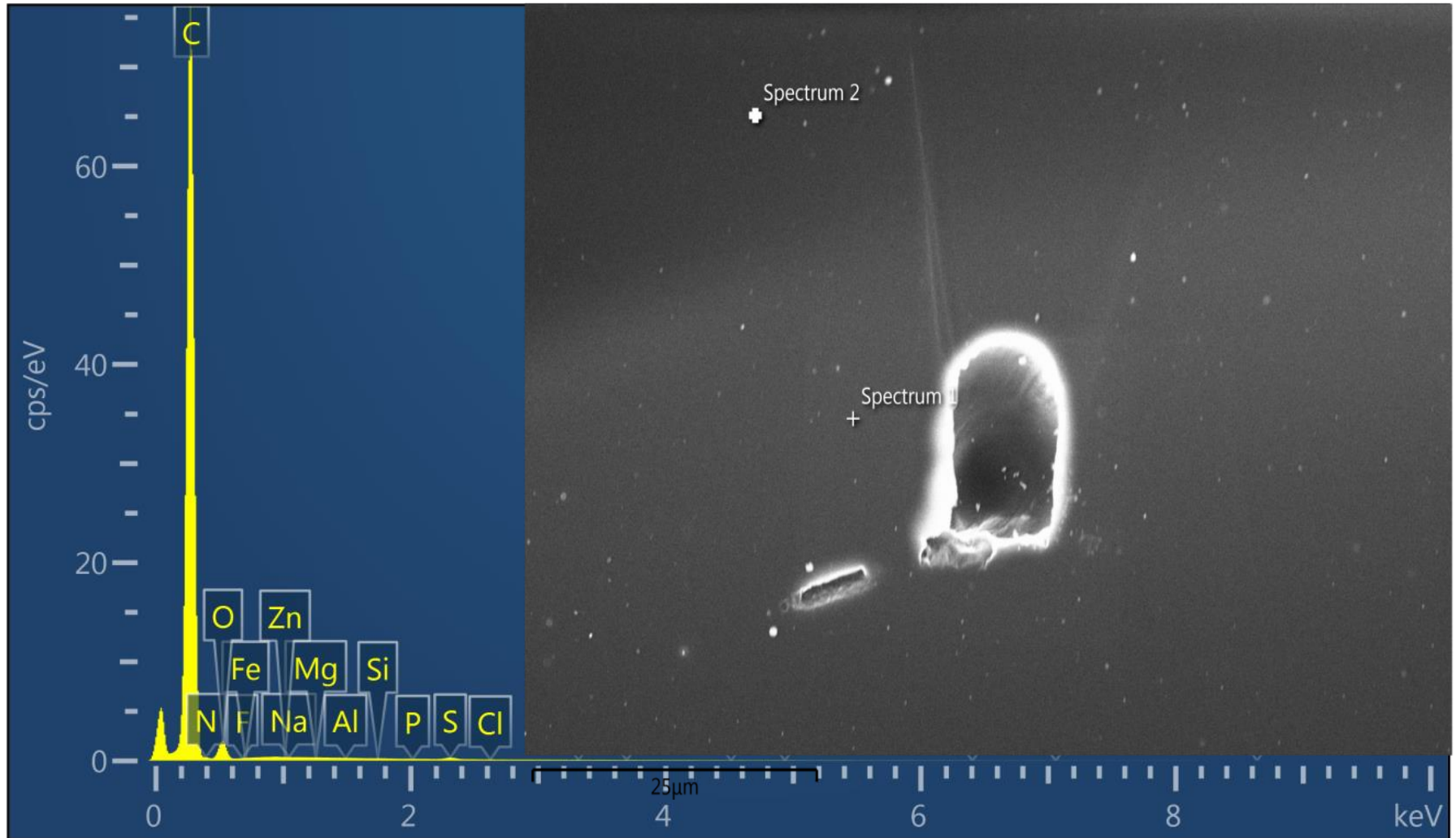
Gilsonite Elemental Analyses

	Elemental analysis ^a	Solid state NMR	ESI-FT-ICR-MS number weighted	ESI-FT-ICR-MS intensity weighted
³ Carbon wt%	80 (84.0)	83%	79.61	80.10
³ Hydrogen wt%	(10.67)	11%	10.57	11.15
³ Nitrogen wt%	3.3 (2.40)	≥3%	3.11	3.25
³ Sulfur wt%	(0.50)	N/A	0.88	1.04
³ Oxygen wt%	(3.74)	3.2%	5.83	4.32
³ H/C atomic ratio	(1.51)	1.49	1.59	1.67

^a Values in parentheses are averages based on literature values (Bell and Hunt, 1963; Clark et al., 1983; Jacob, 1989).

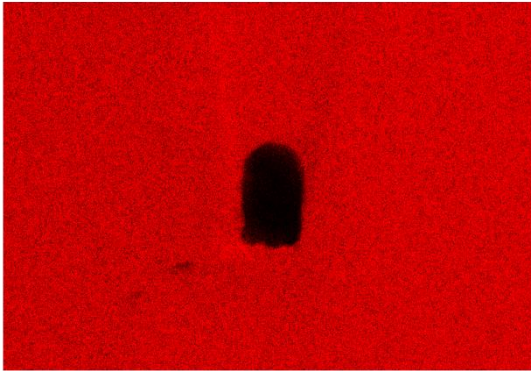
from Helms et al. (2012)

Gilsonite EDS Spectra

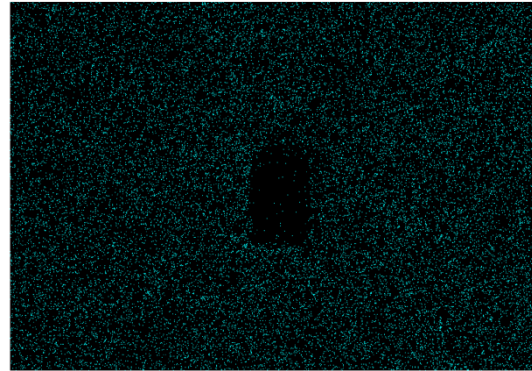


Gilsonite EDS Elemental Maps

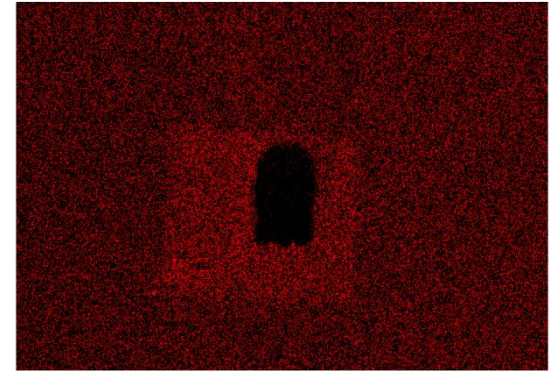
C K α 1_2



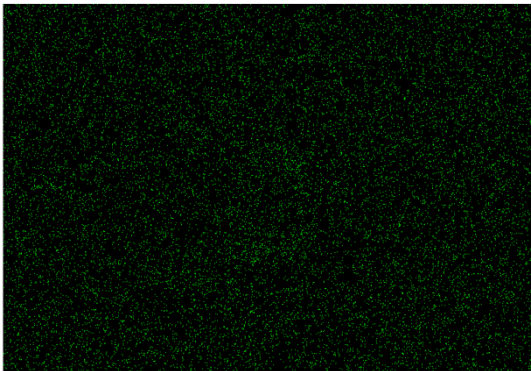
N K α 1_2



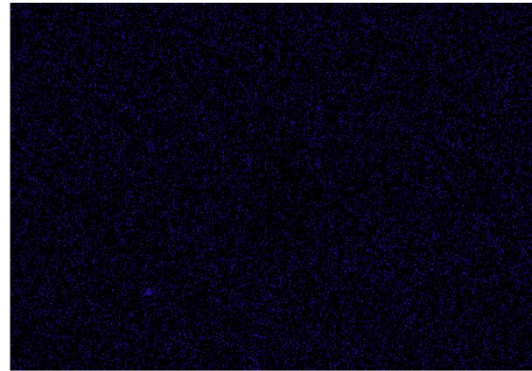
O K α 1



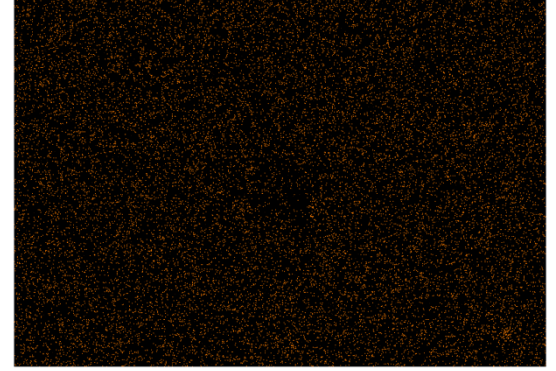
P K α 1



S K α 1



Si K α 1



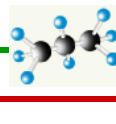
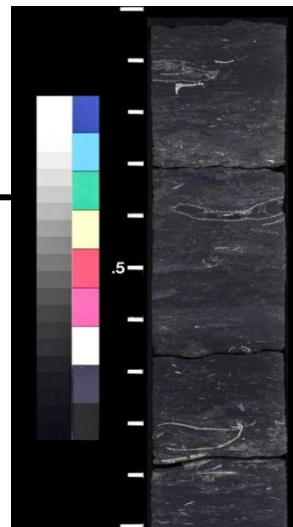
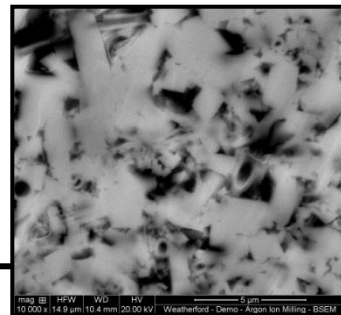
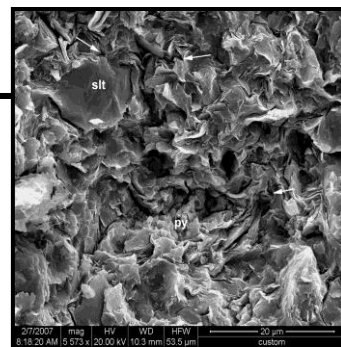
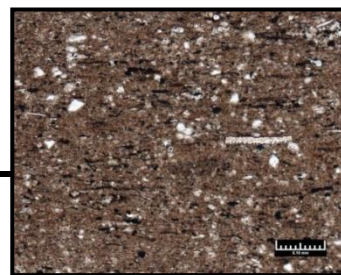
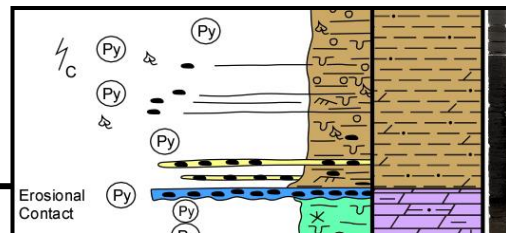
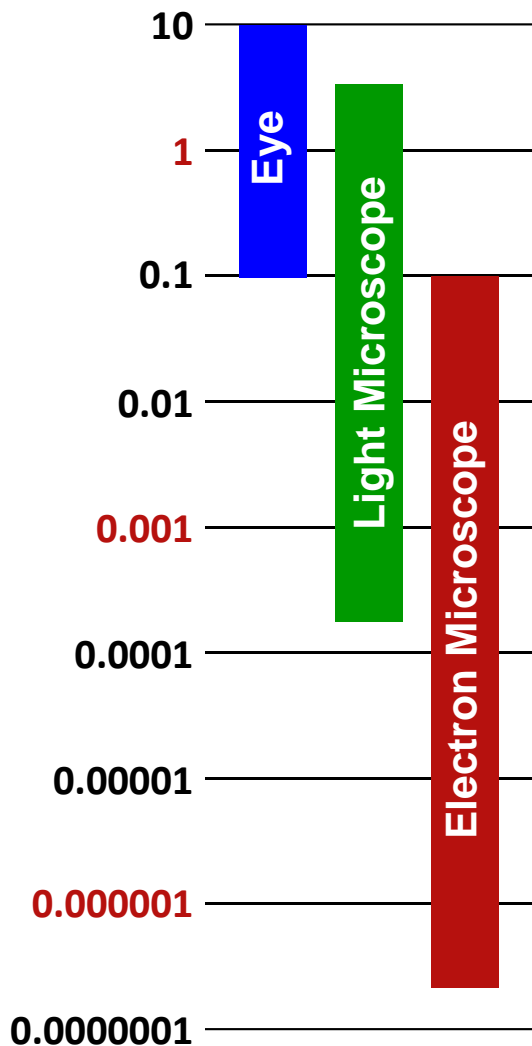
Scale of Characterization

Scale in Millimeters

Millimeter

Micrometer

Nanometer



Oil Molecules – 0.5 to 3 nm



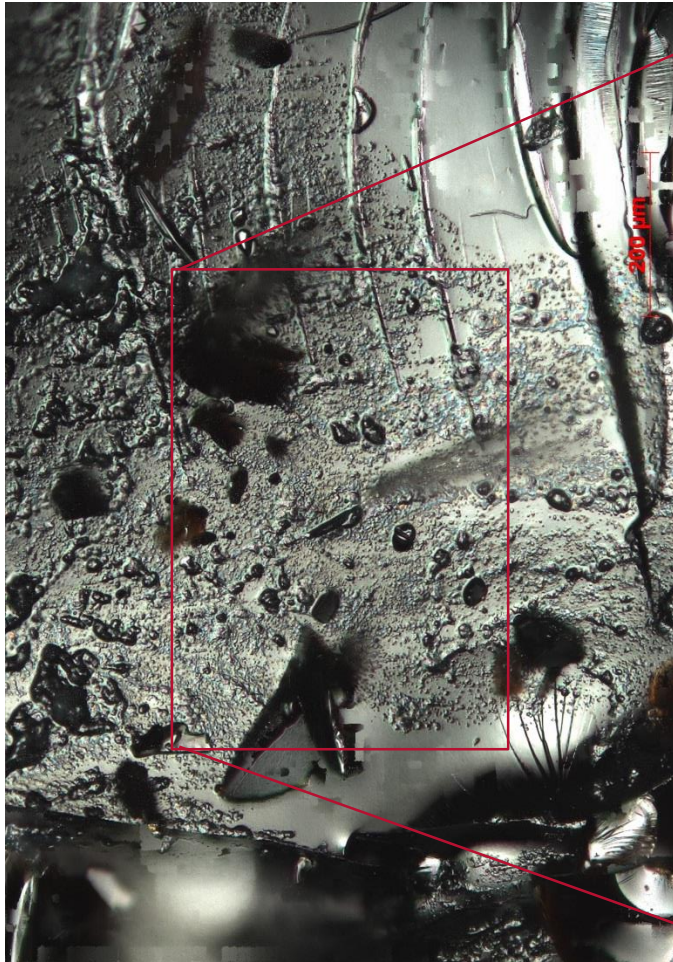
Methane Molecule – 0.4 nm

Pencillated Gilsonite

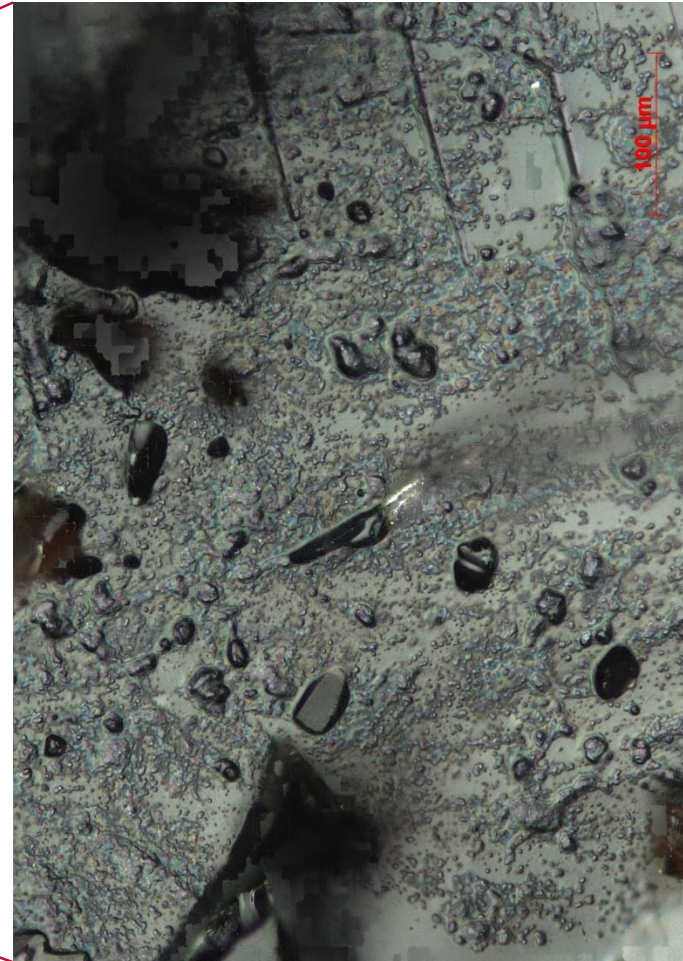


Pencillated Gilsonite

20X Air Objective



40X Air Objective

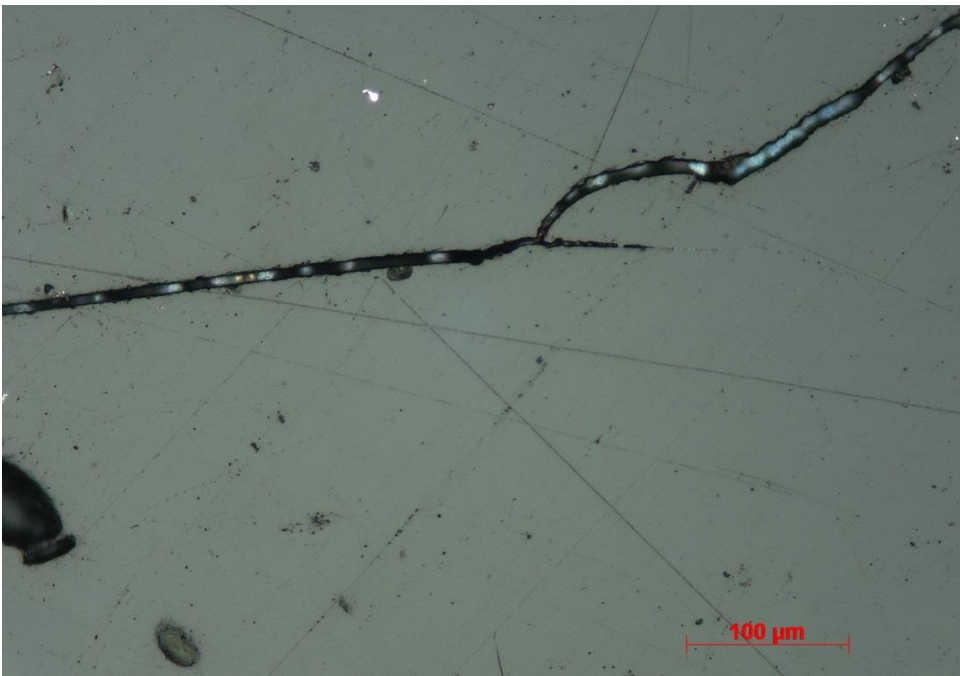


Micropore to mesopore size pitting evident



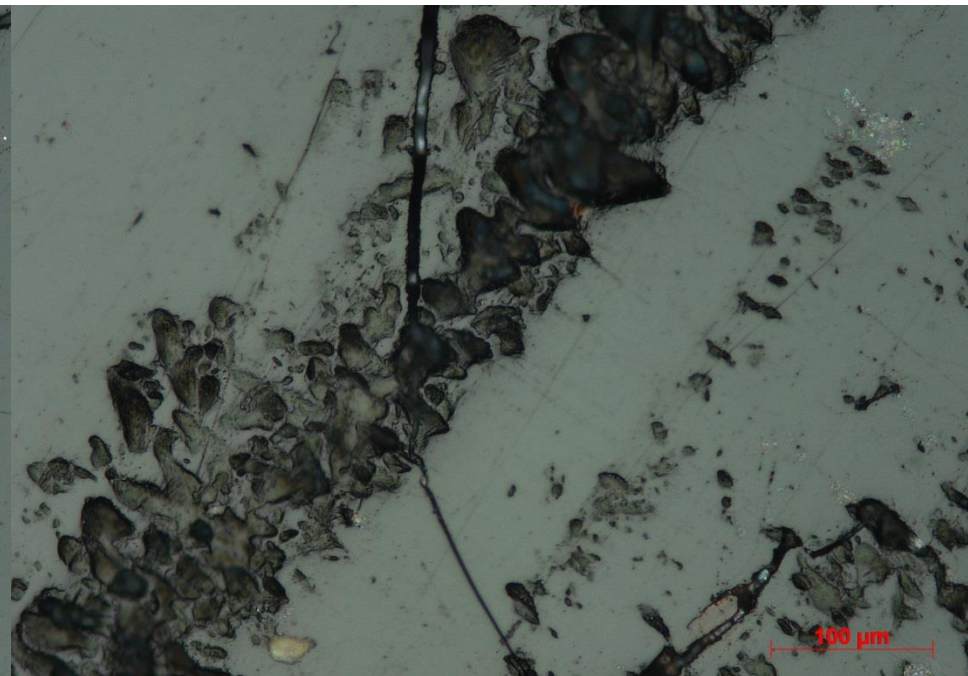
Pencillated Gilsonite

40X Air Objective



Typical field of view of 'pencillated' plane using 40x air objective.

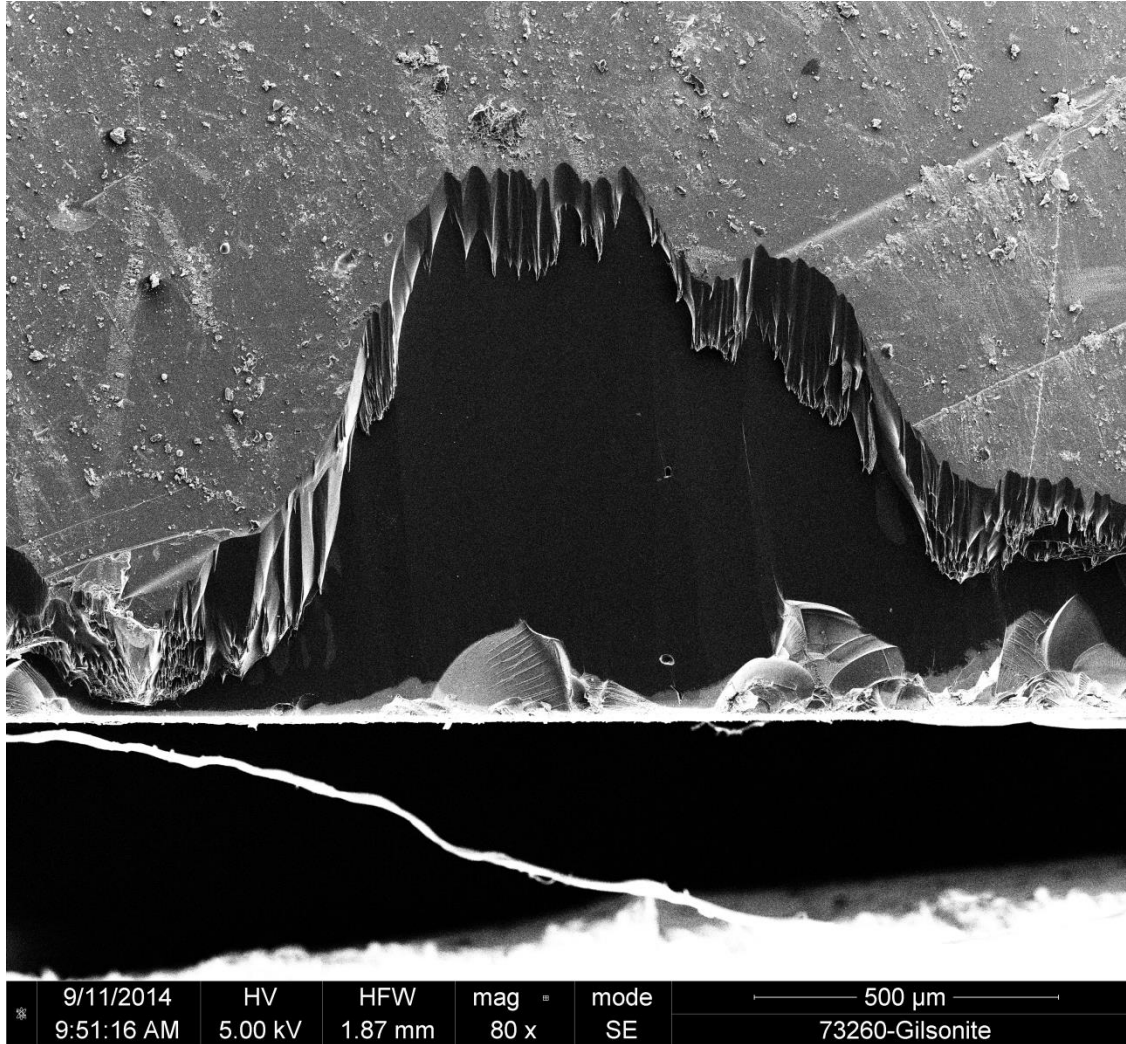
40X Air Objective



Additional photomicrograph showing less common pitted pattern not attributed to polishing process.

Pencillated Gilsonite

80X SEM

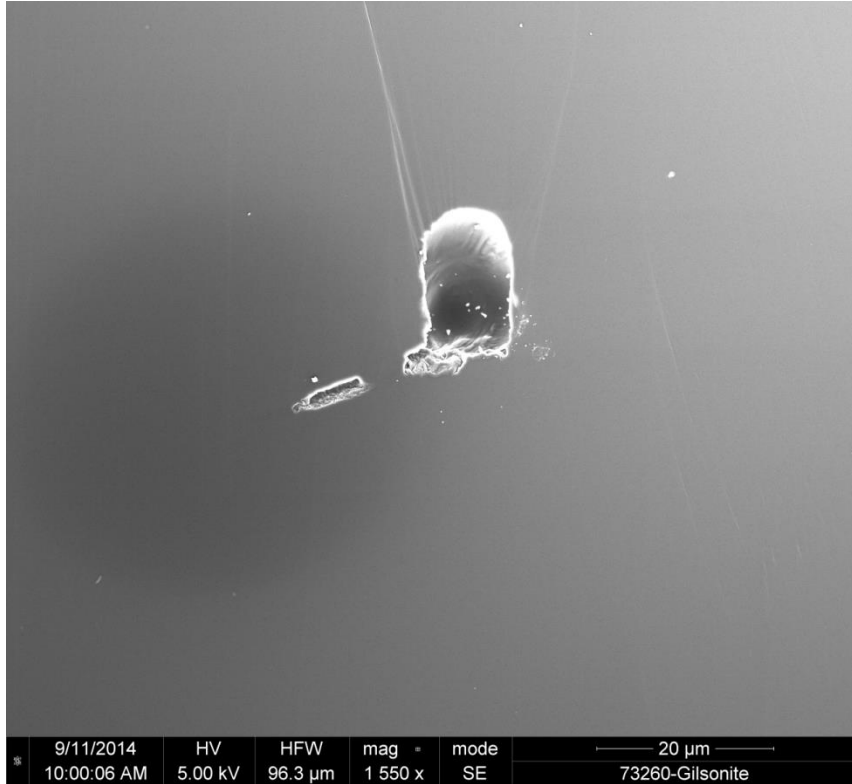


Ion milled surface exhibits no nanoporosity with rare micropore pits

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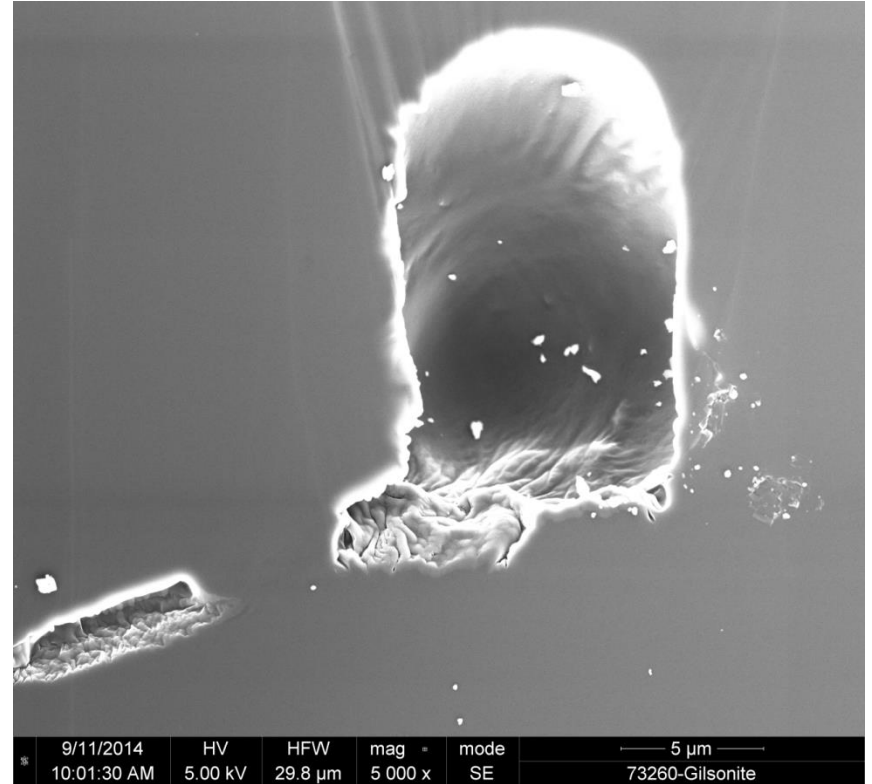
Pencillated Gilsonite

1550X SEM



Matrix is featureless and rare micropore pits are discontinuous

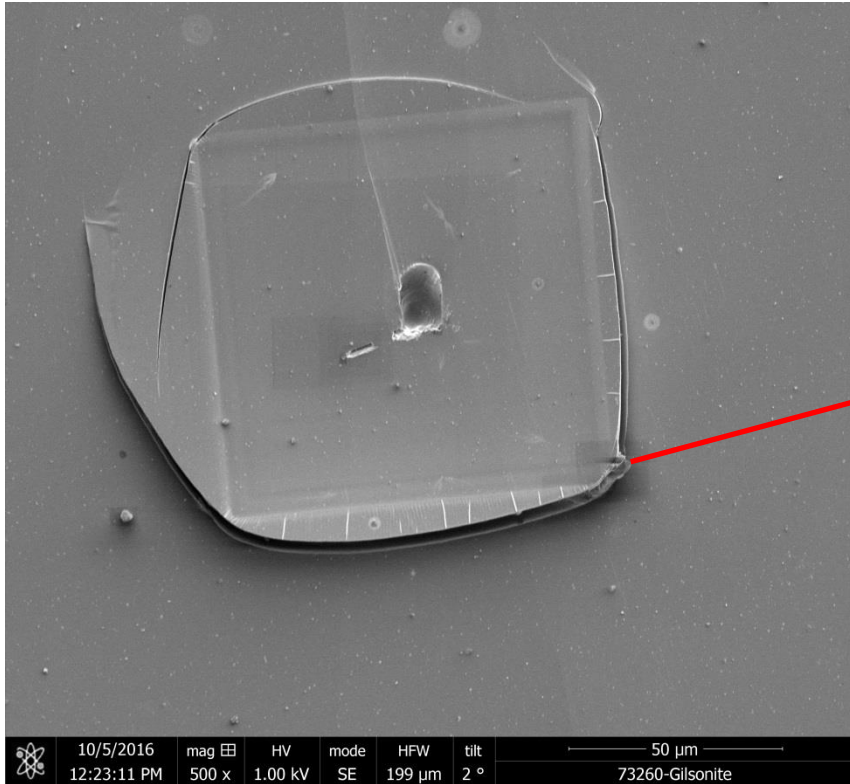
5000X SEM



Enlarged view of discontinuous micropore pits

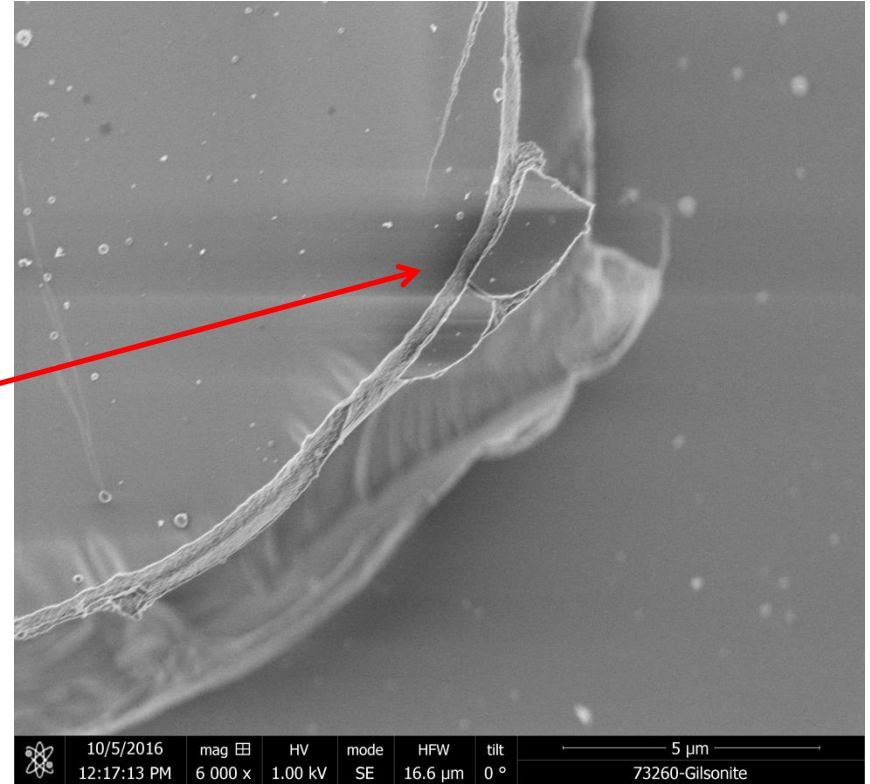
Pencillated Gilsonite

500X SEM



Matrix exhibits alteration associated with SEM imaging upon re-examination

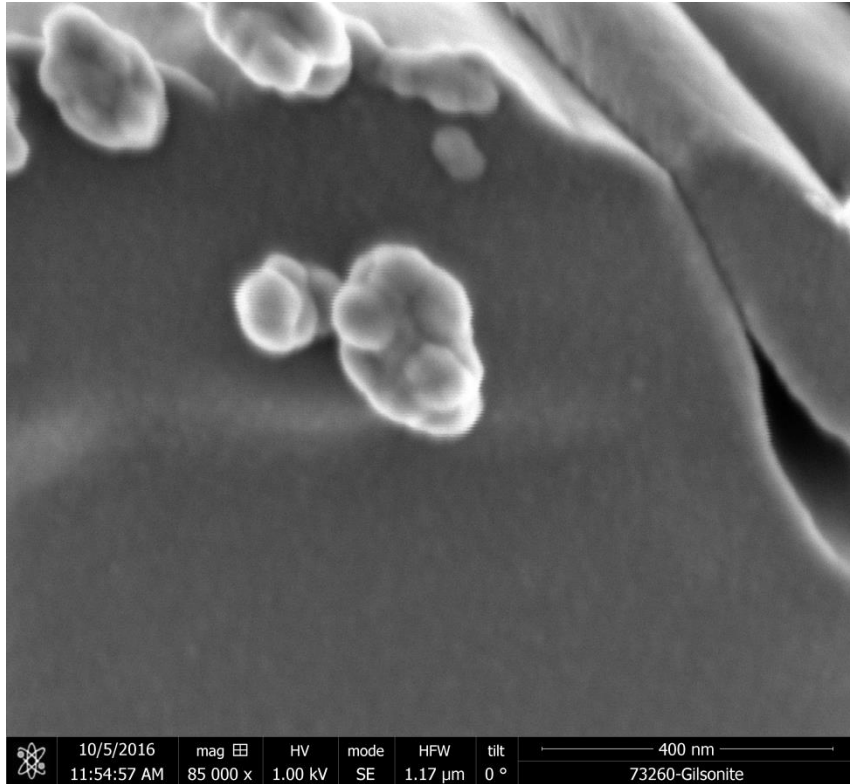
6000X SEM



Observed SEM induced fractures and distortion within matrix

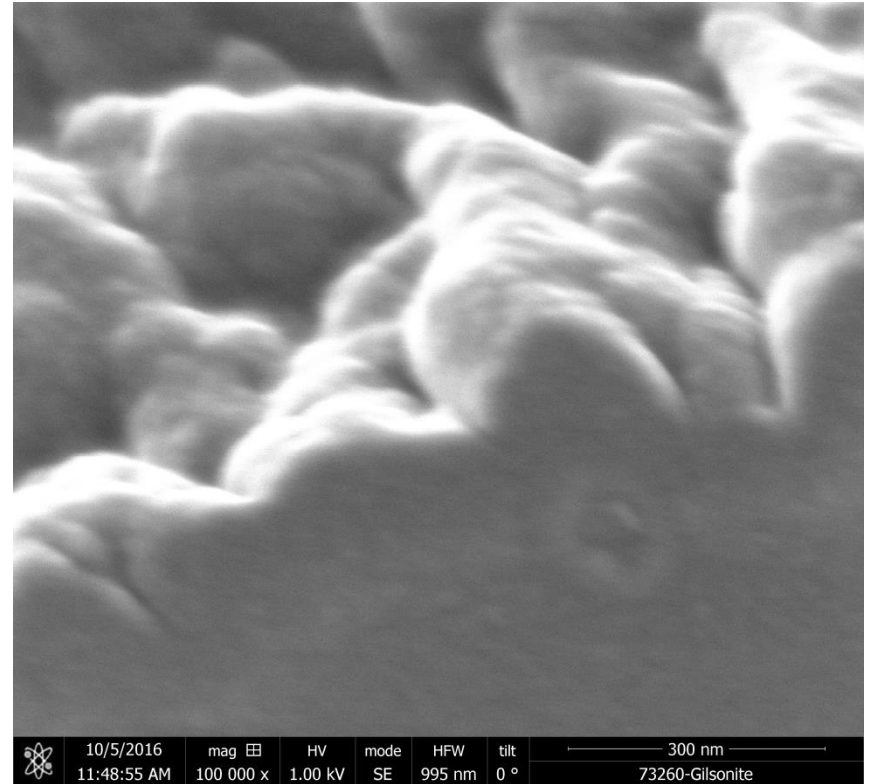
Pencillated Gilsonite

85000X SEM



No occurrence of organic porosity even at high magnification

100000X SEM

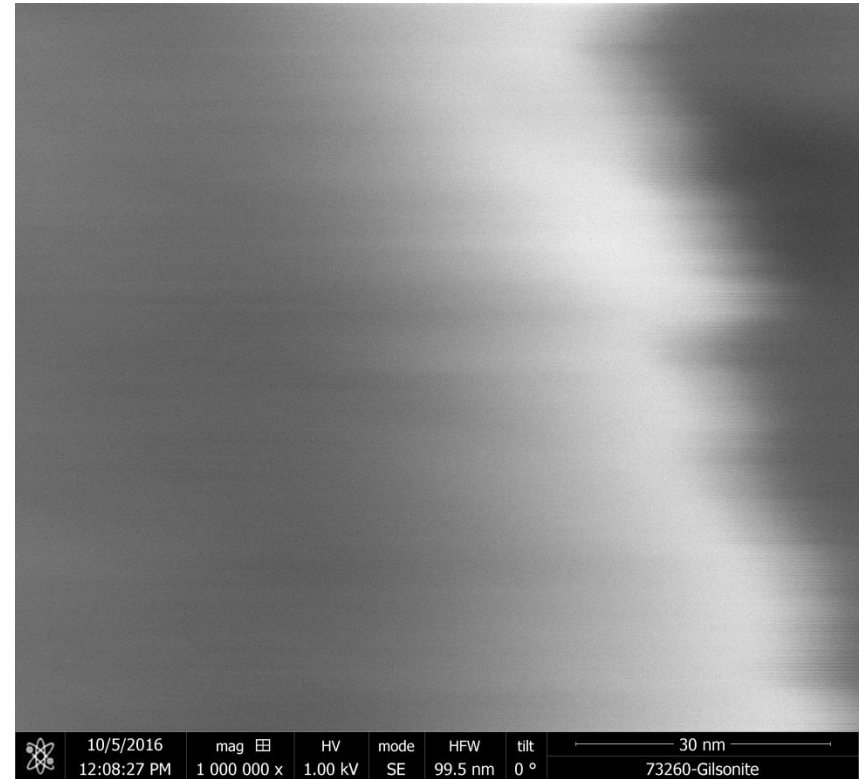


Subtle textural features do not appear to be associated with organic porosity

Pencillated Gilsonite



1000000X SEM



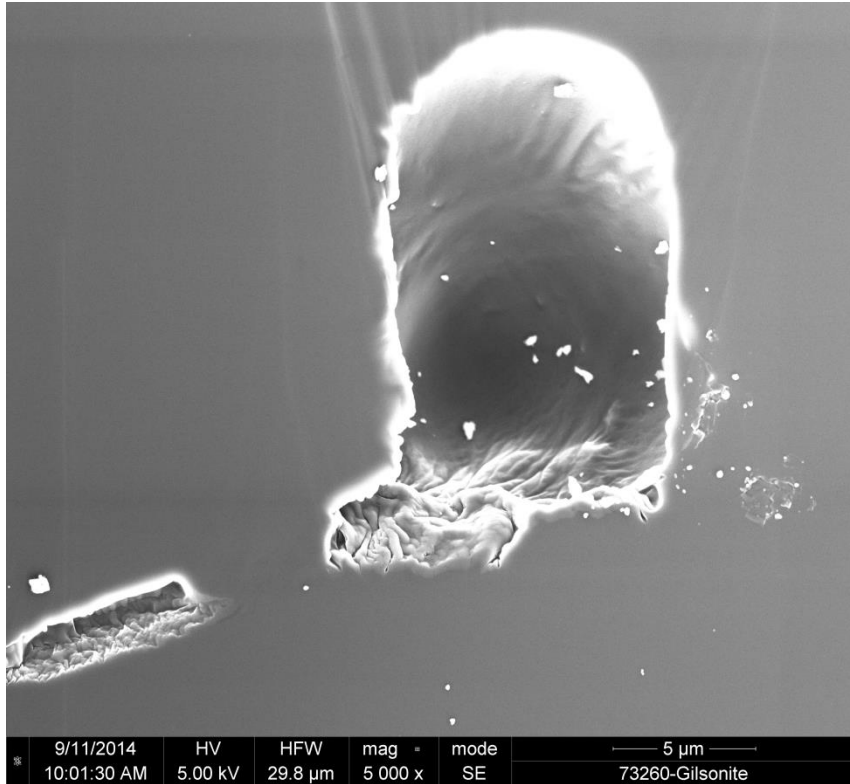
Better...Stronger...Faster

Bobby Hooghan is the Million Magnification Man

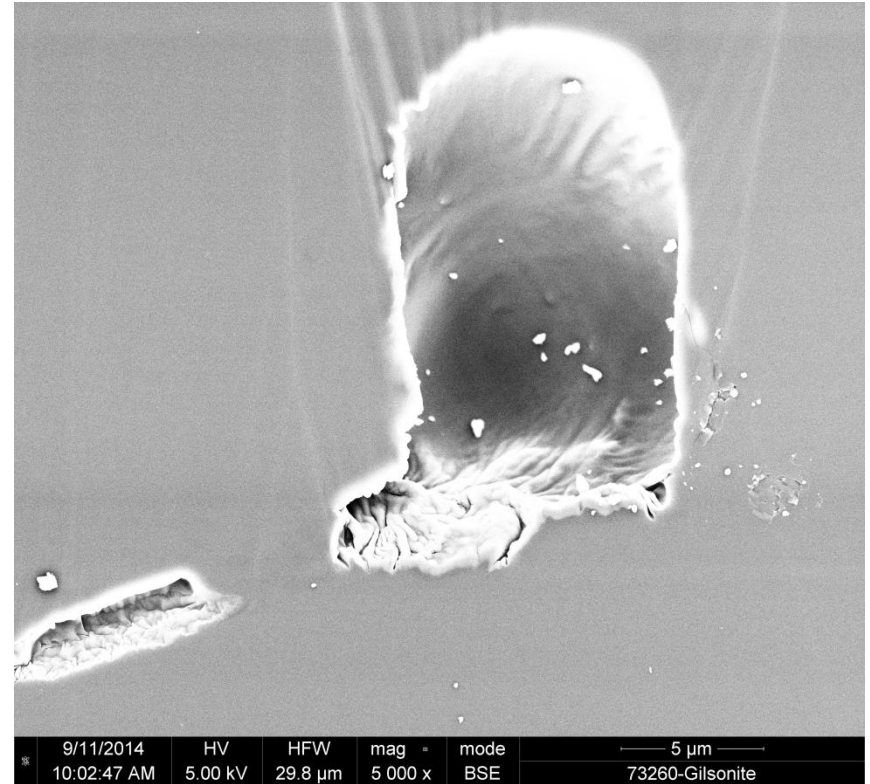
Still no visible organic porosity

Pencillated Gilsonite

5000X SEM SE



5000X SEM BSE

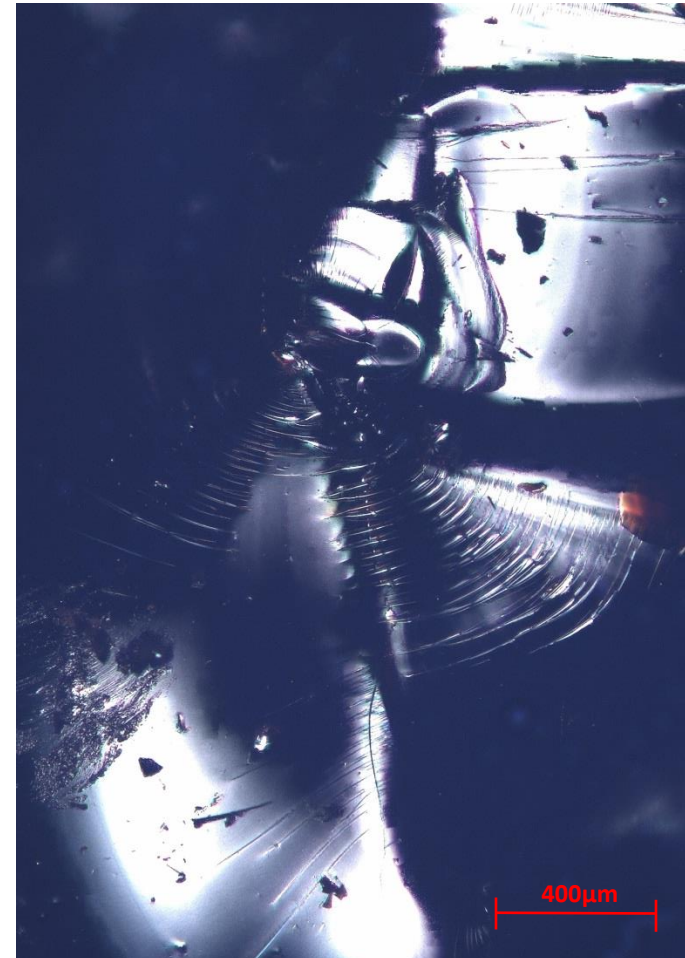


No major difference between secondary electron image and backscatter image.....still no evident porosity

Conchoidal Gilsonite

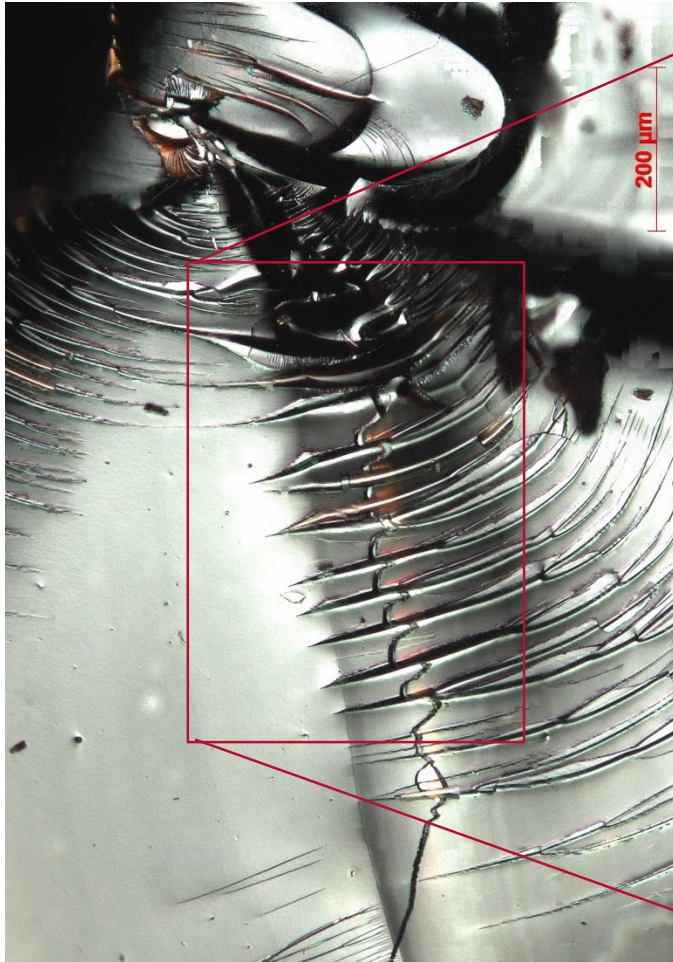


10X Air Objective

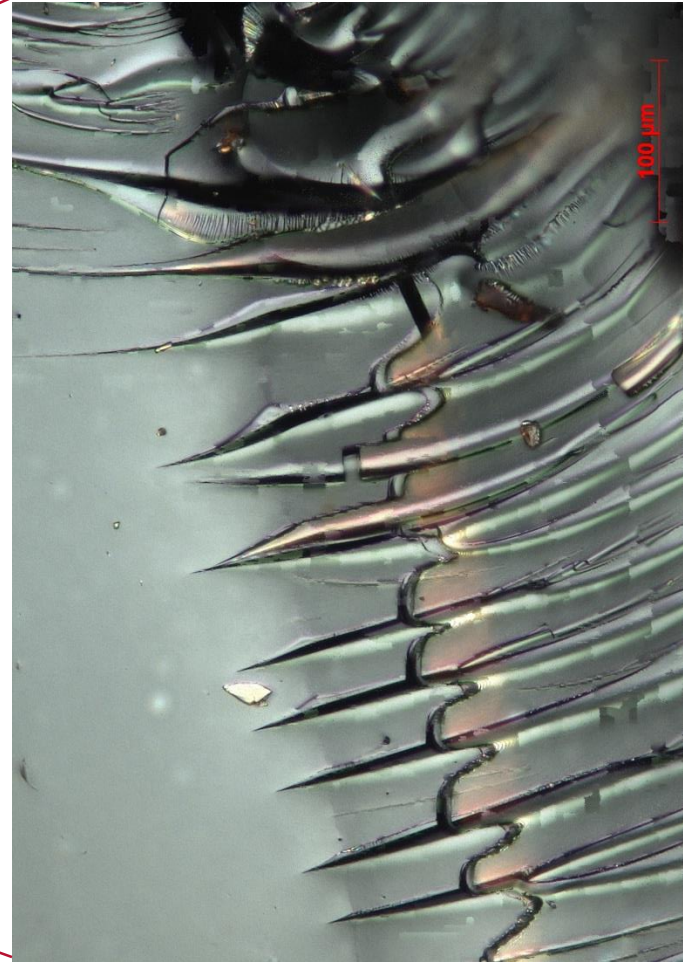


Conchoidal Gilsonite

20X Air Objective

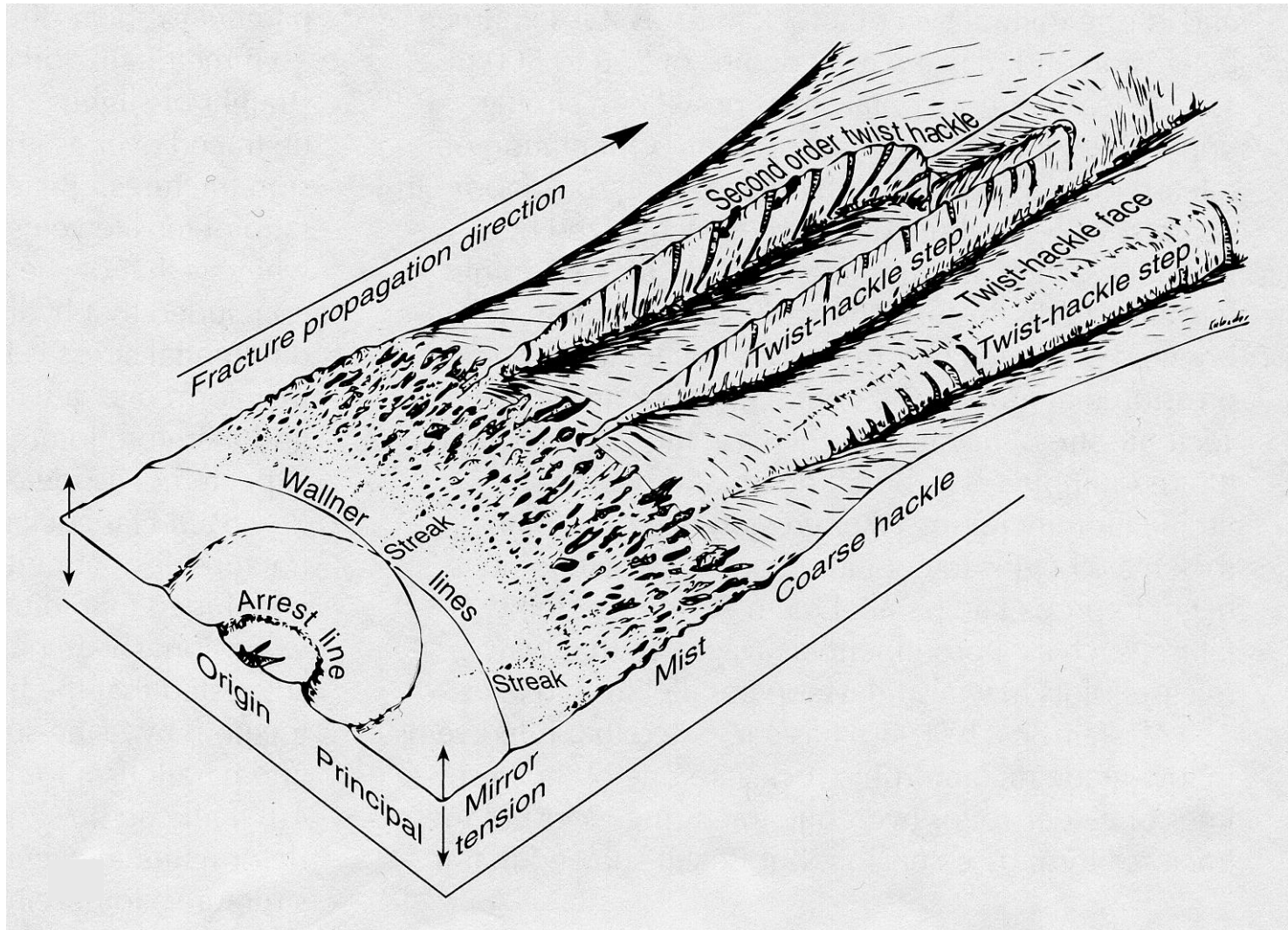


40X Air Objective



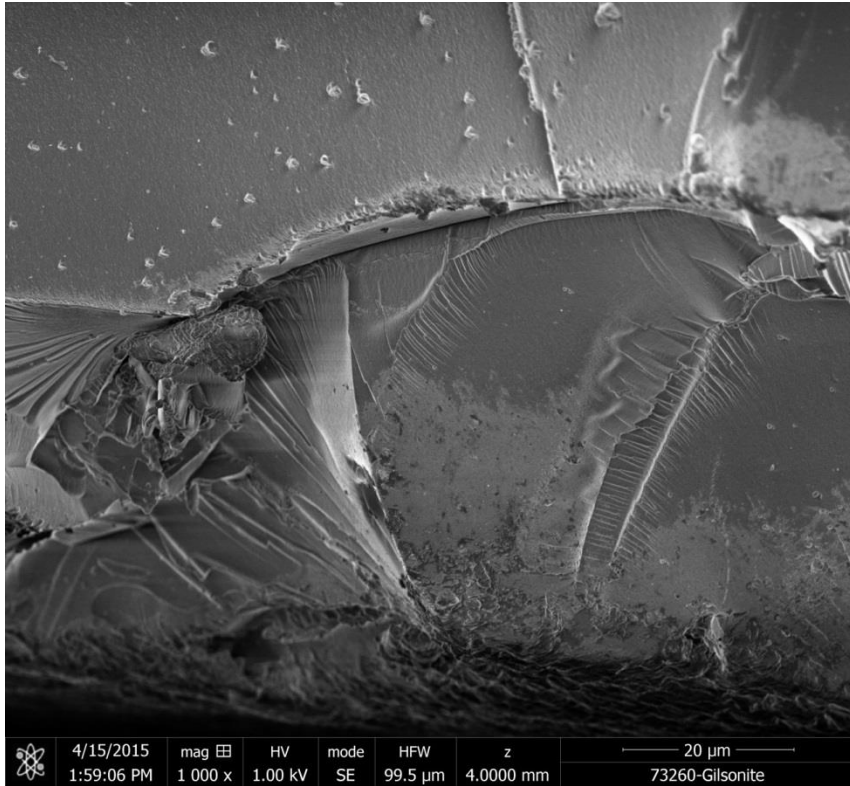
Typical twist hackle mark created by breaking sample

Joints and Shear Fractures



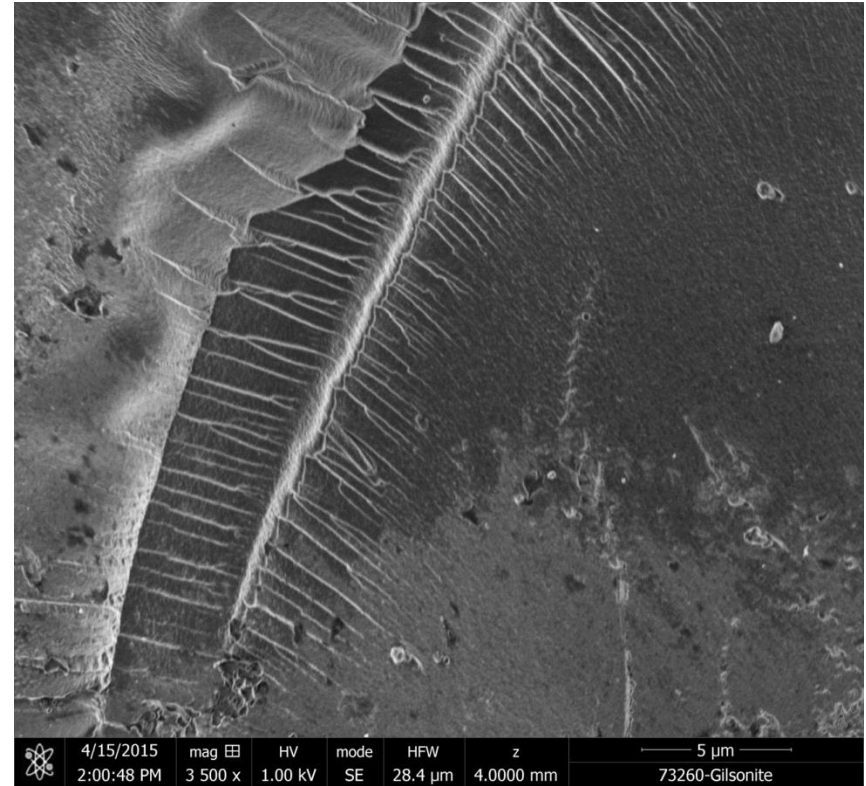
Conchoidal Gilsonite

1000X SEM



Conchoidal fractures evident but matrix shows no organic porosity

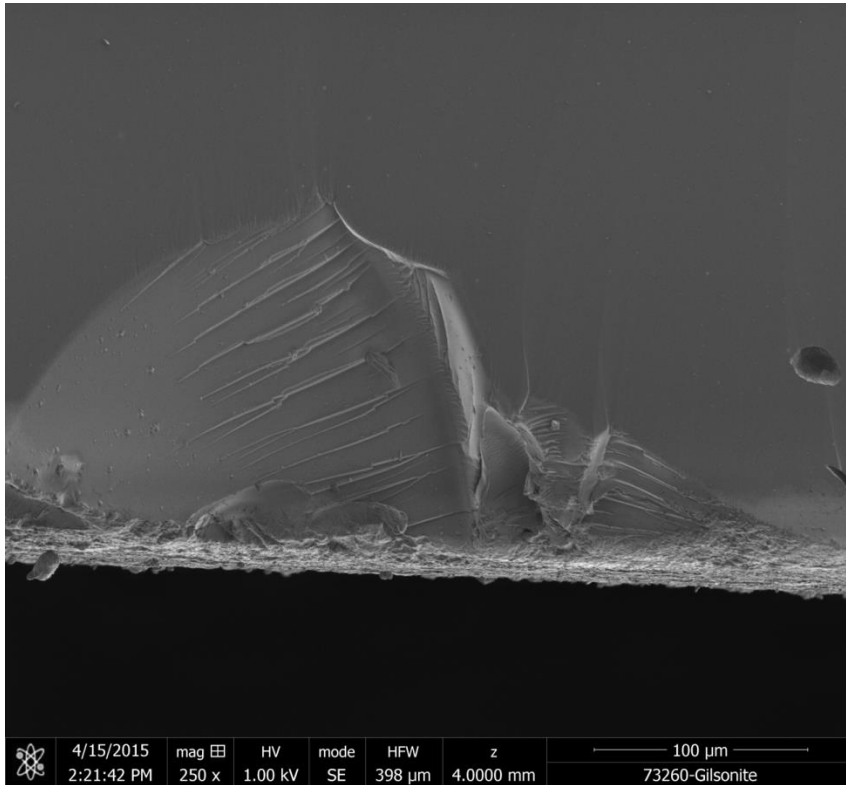
3500X SEM



Fracture plane with twist hackle marks

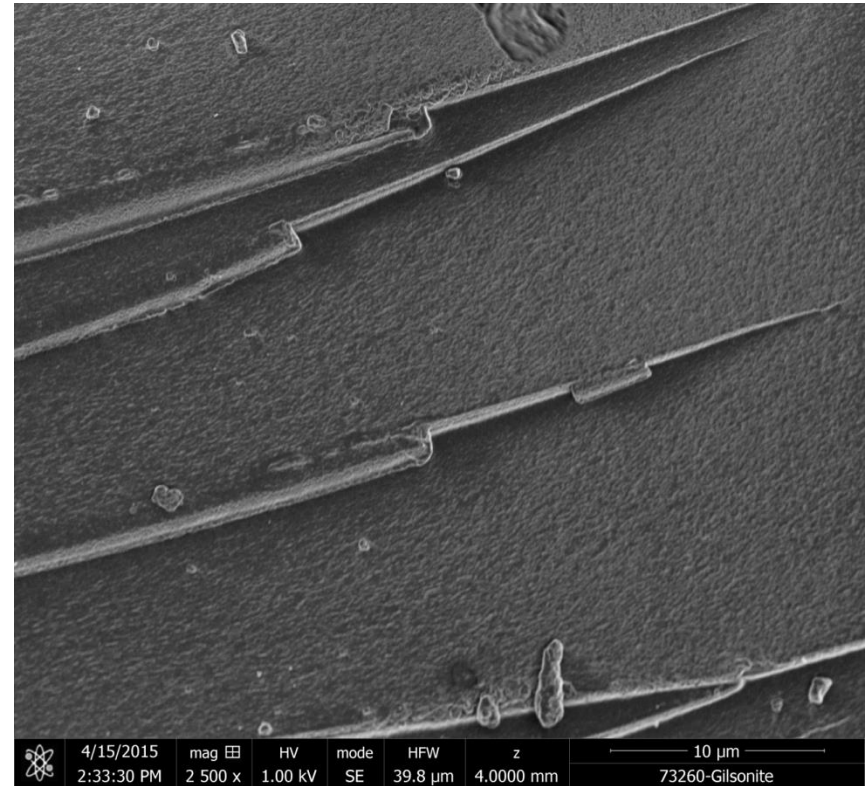
Conchoidal Gilsonite

250X SEM



Edge of conchoidal fracture surface

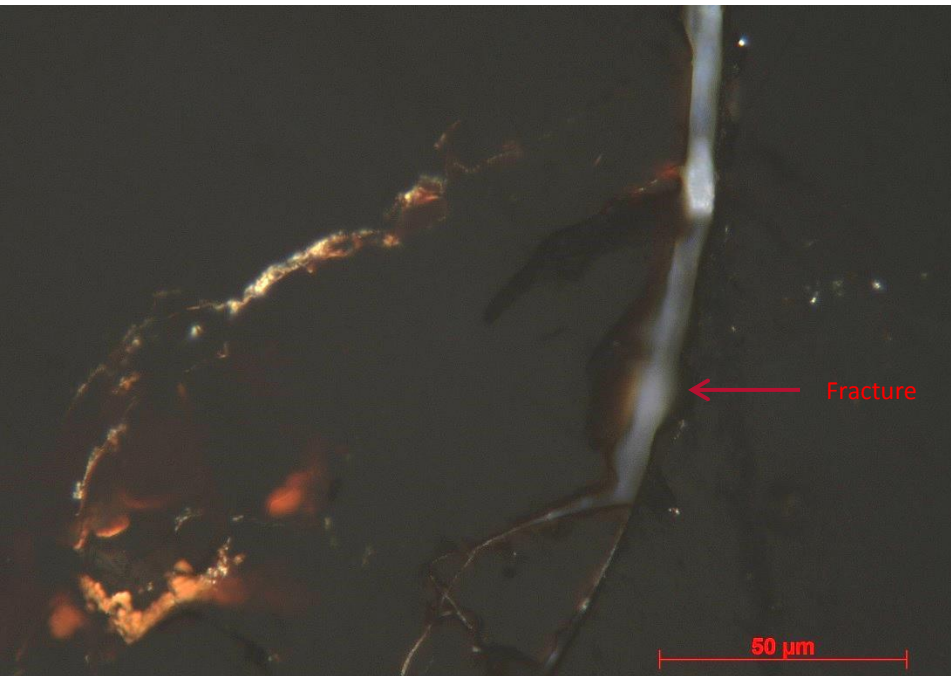
2500X SEM



Enlarged twist hackle marks

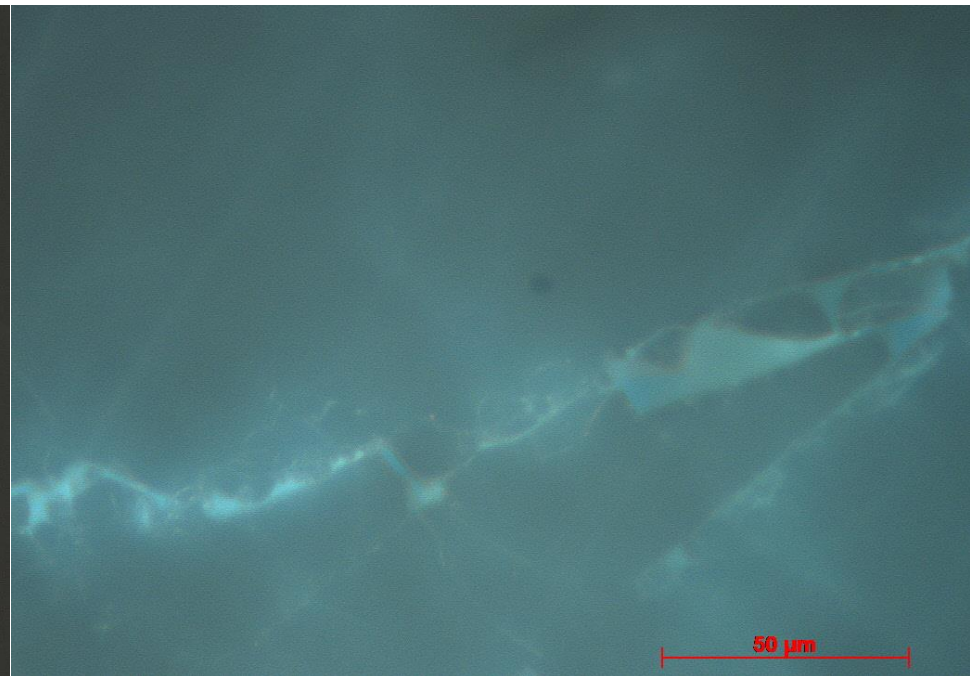
Conchoidal Gilsonite under Oil Immersion

80X Oil Immersion Objective



Reflected white light photomicrograph of conchoidal fracture plane. Note translucent brown area to left typical of bitumens.

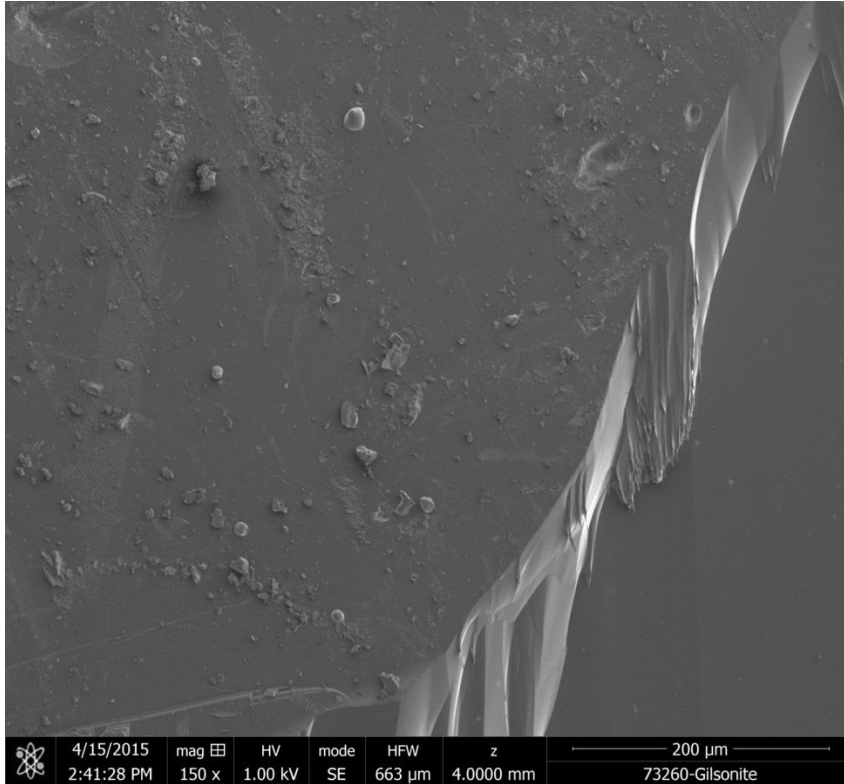
80X Oil Immersion Objective



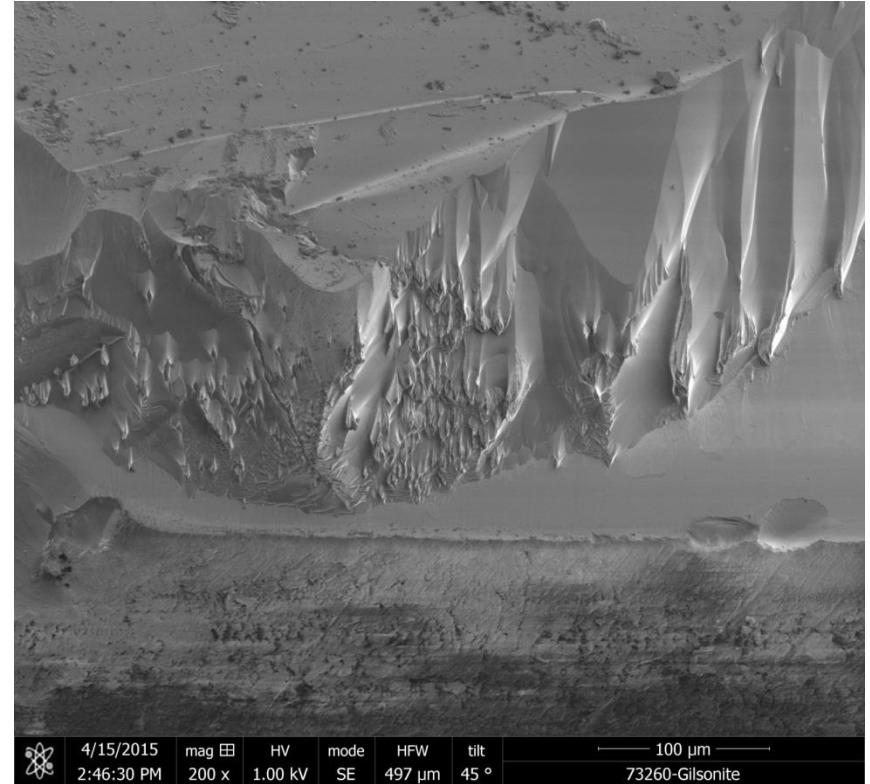
Reflected UV photomicrograph of fracture in conchoidal plane showing a 'fog' of mobile hydrocarbons leeching from fracture.

Gilsonite Tilted View Imaging

90°/150X SEM



45°/200X SEM

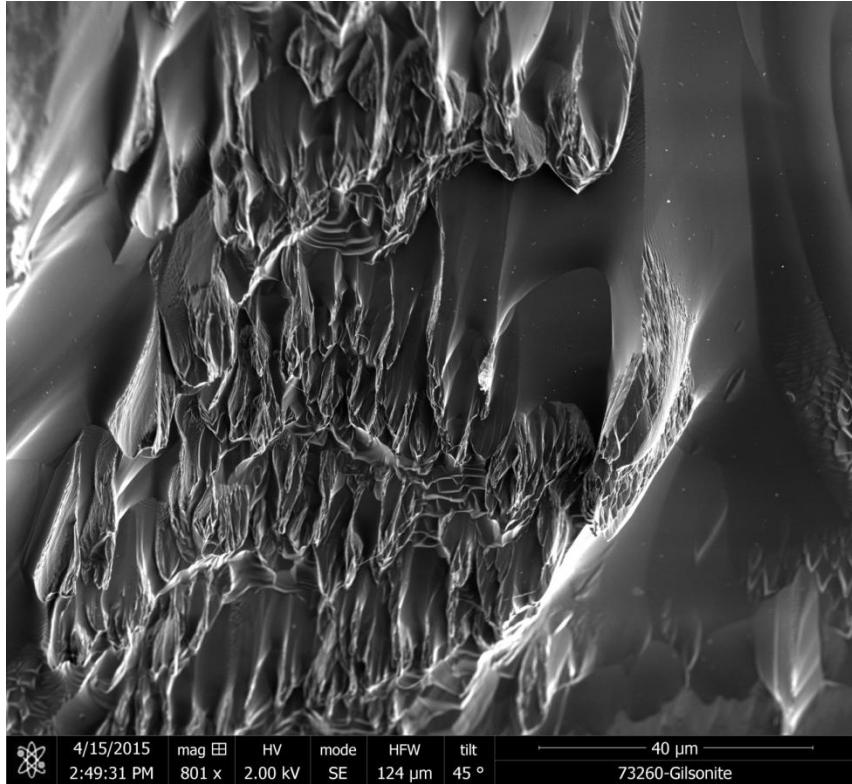


Ion milled contact – perpendicular view

Ion milled contact – tilted view

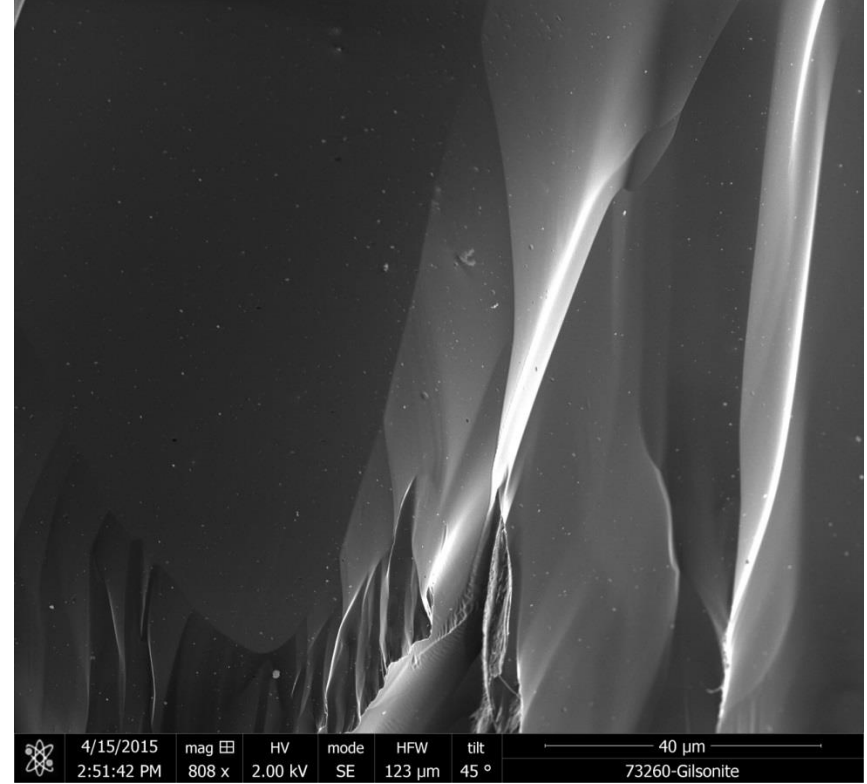
Gilsonite Tilted View Imaging

45°/800X SEM



Conchoidal fracture surfaces in enlarged ion milled contact – tilted view

45°/800X SEM



Absence of nanoporosity in enlarged ion milled contact – tilted view

Organic Petrology & SEM Observations

- Polished surfaces are typically dark grey and featureless. Macerals and inorganics are absent.
- Visual estimates suggest that fractures make up approximately 1% of the conchoidal fracture plane.
- Visual estimates of the pencillated plane suggest approximately 2% of the surface contains fractures but also 5% shallow pits.
- SEM imaging at all scales and angles confirms morphology of fractures and pitting along with the absence of organic porosity in gilsonite.

Summary Thoughts

- Unlike pyrobitumen, pre-oil solid bitumen represented by gilsonite was found to contain no significant organic nanoporosity in its matrix.
- Gilsonite does have minor pitting and fractures, but these do not represent an effective interconnected pore network and are probably artifacts of weathering/sampling.
- Bitumen and oil are thought to represent two different immiscible phases when water is present, especially water dissolved in the liquid bitumen phase.
- Depending upon thermal maturity, unconventional source rock reservoirs may contain bitumen, oil or both, which form a continuous expulsion/migration pathway.

References

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