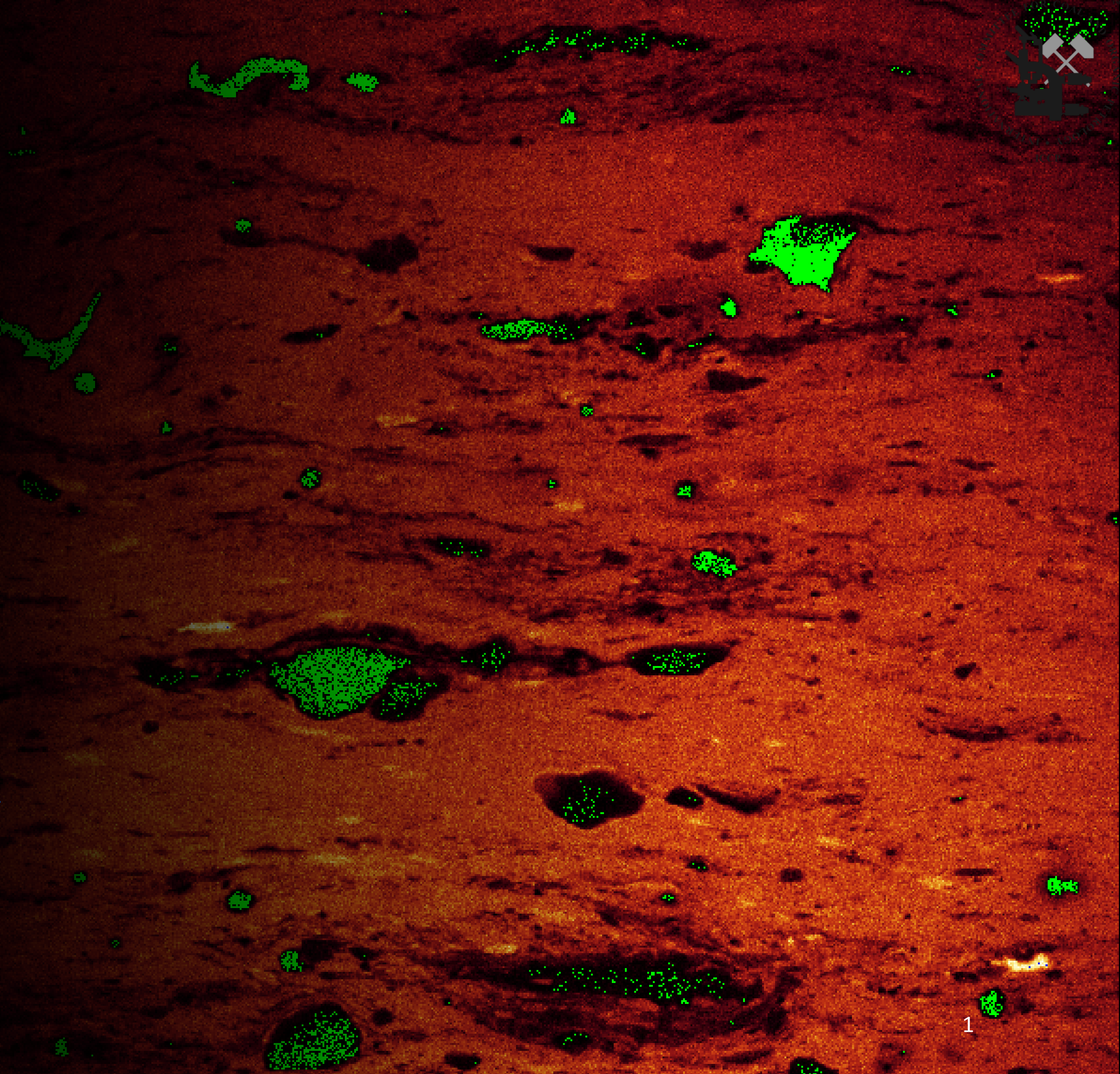
Confocal Laser Scanning Microscopy (CLSM) Working Group: Final Report 2021

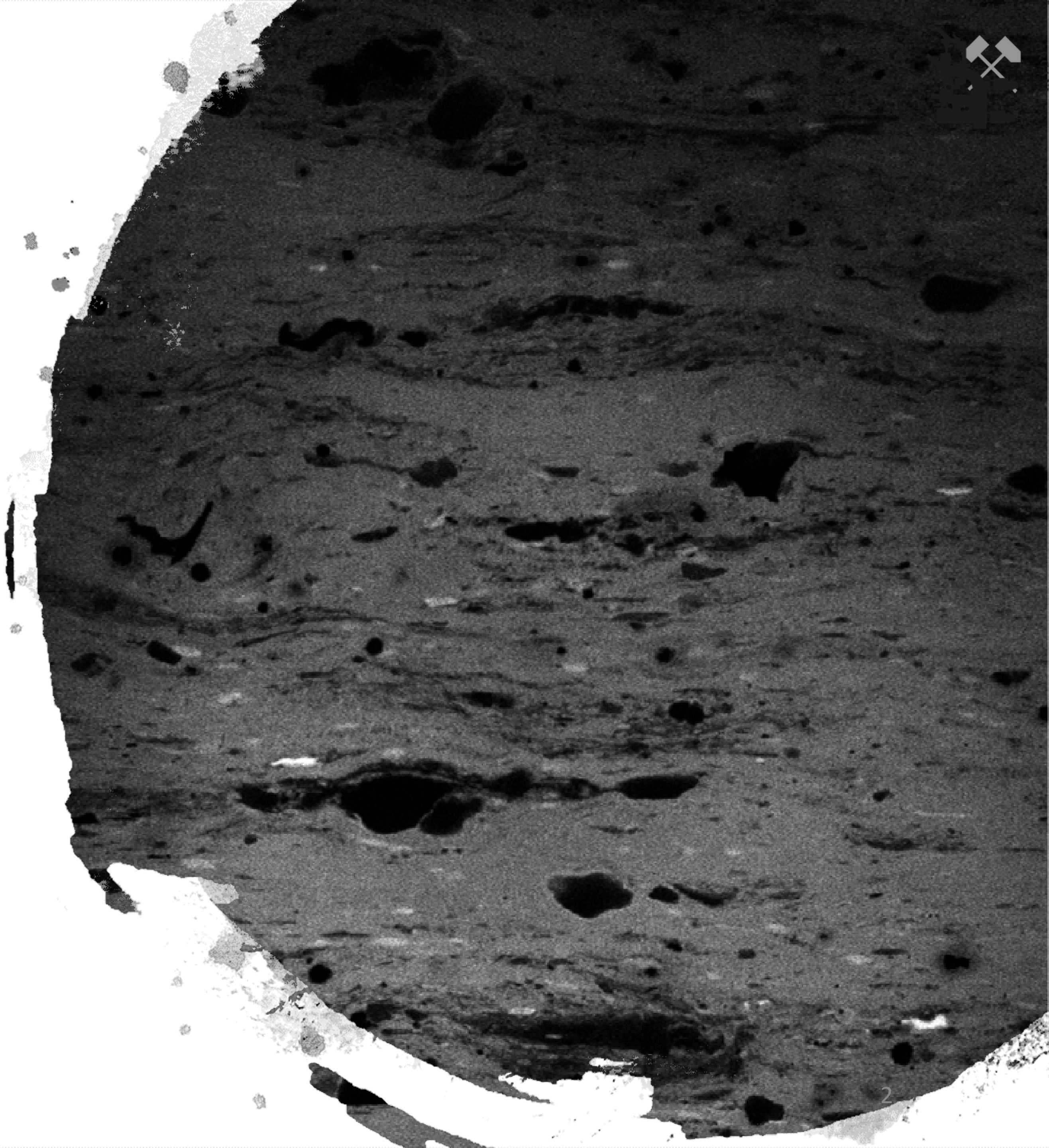
Paul C. Hackley, USGS, phackley@usgs.gov

Jolanta Kus, BGR, jolanta.kus@bgr.de



Outline

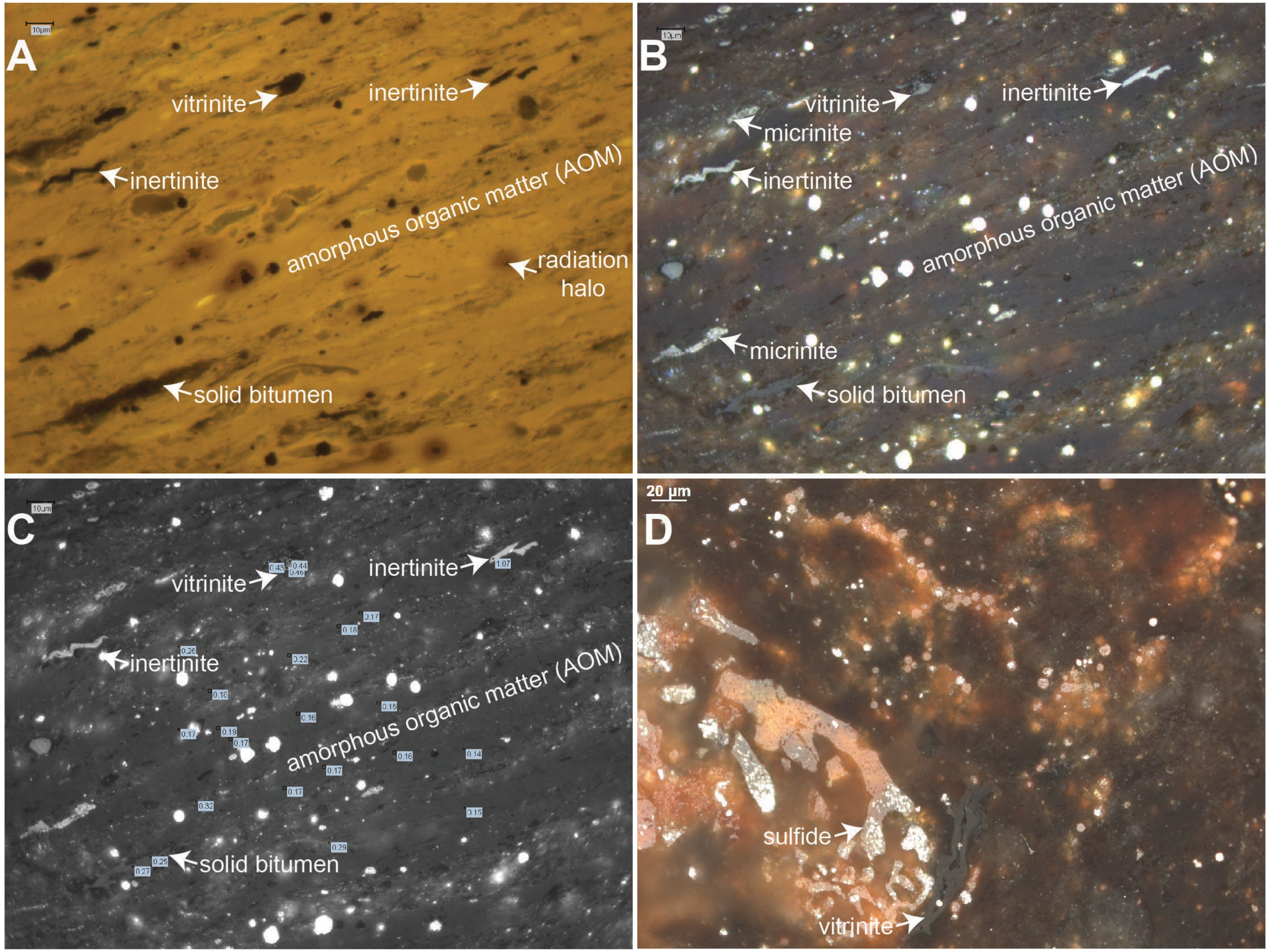
- ❑History of the WG
- ❑Sample used in WG
- ❑AFM/ion milling characterization
- ❑CLSM results
- ❑Outcomes and Summary



History of the CLSM WG

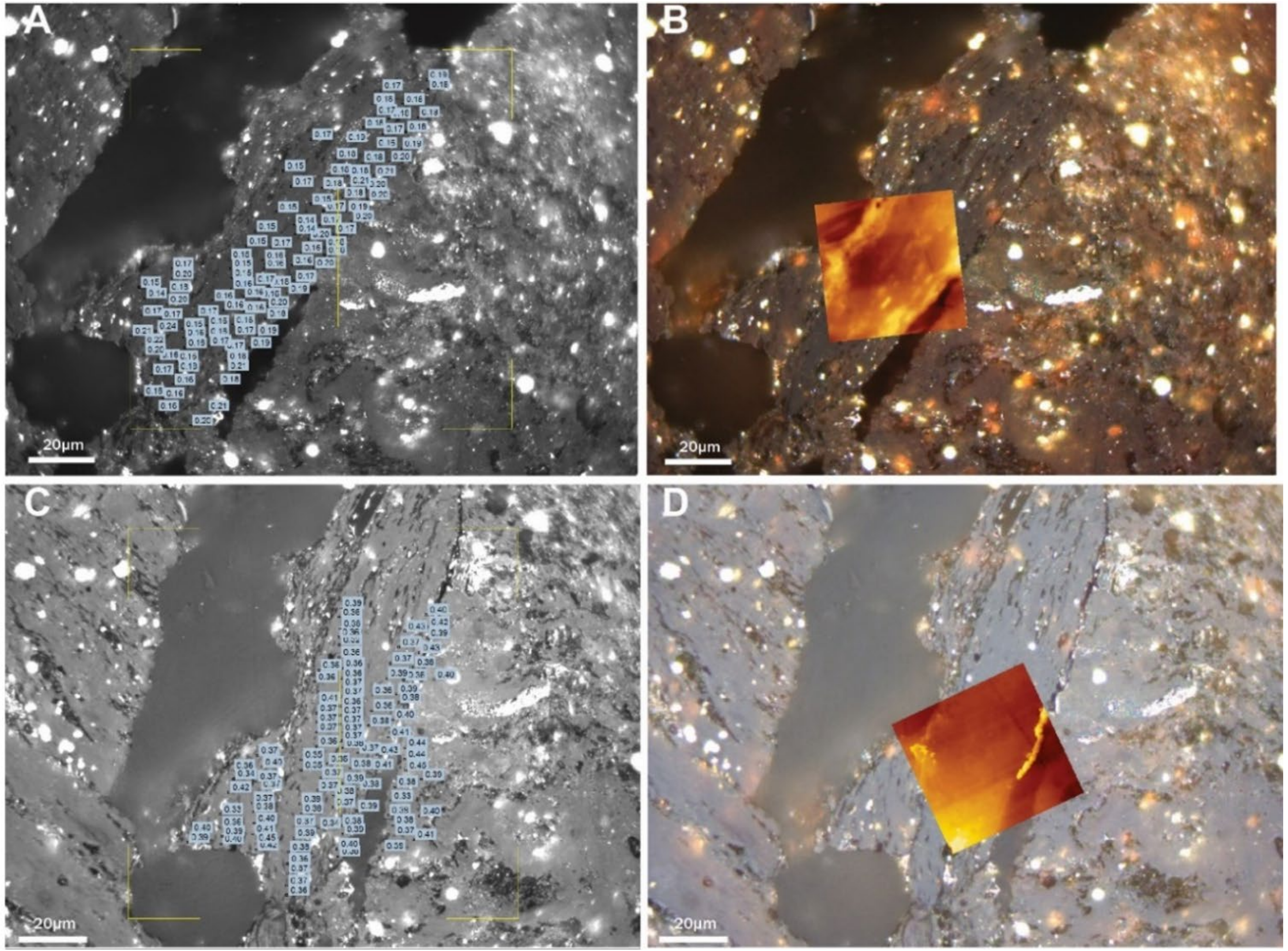
- Established Potsdam 2015
- Call for participants ICCP News No. 63 (Nov. 2015)
- Questionnaire February 2016
- Additional participants from 2016 GSA, 2016 ICCP (total 16)
- Kimmeridge Clay (KC) selected for ILS in 2017
- KC sample distributed April 2018
- Initial KC results presented Brisbane 2018
- Efforts to rejuvenate WG discussed The Hague 2019
- Covid 2020
- Video meetings of the convenors in 2021
- No interest from WG members to continue in 2021
- Decision to close WG 2021

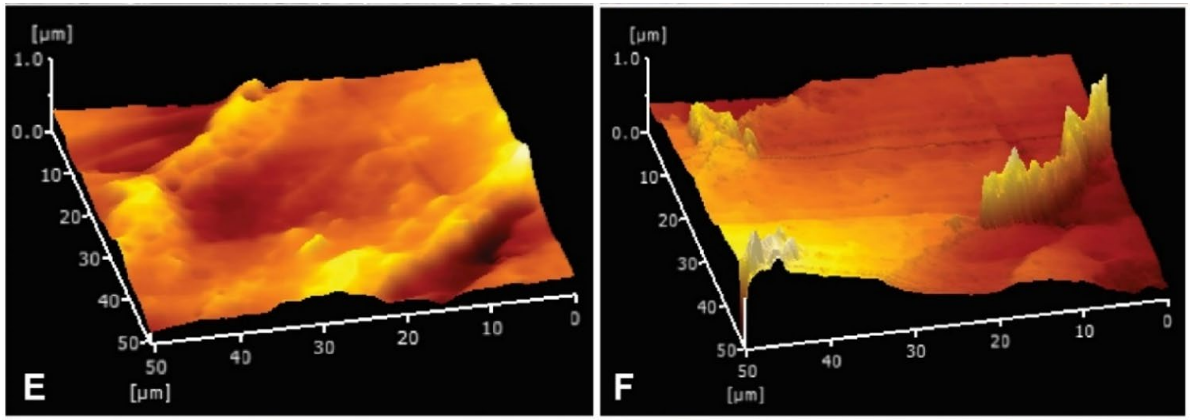
Sample used in CLSM WG

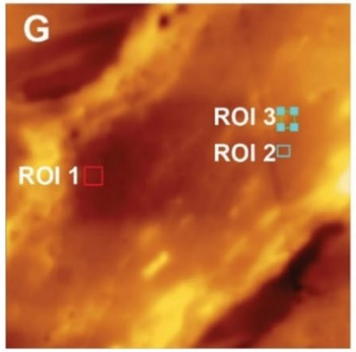


- Kimmeridge Clay Fm.
- 44 wt.% TOC, 0.42% VR_o
- 0.29% BR_o, 737 HI
- bituminite, vitrinite, solid bitumen, inertinite, micrinite, sulfides
- 0.035 S_{org}/C (6.63 wt.% org. S)

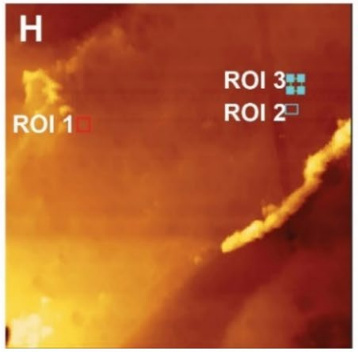
- Response to broad ion beam milling
- Reflectance increase
- Differential milling based on hardness
- BIB-induced surface flattening measured by AFM





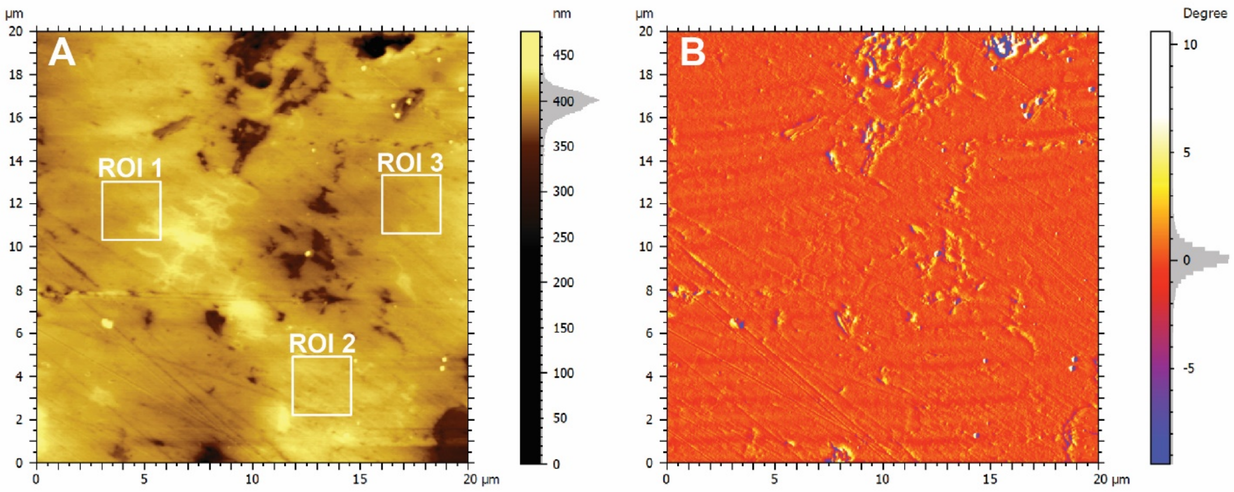


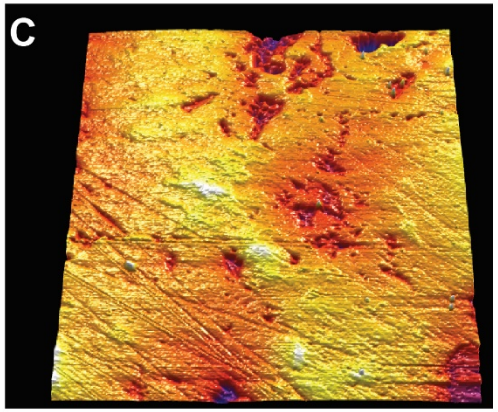
Location	S_q (nm)
Whole field	81.54
ROI 1	11.00
ROI 2	5.350
ROI 3	17.82
Avg. ROI	11.39



Location	S_q (nm)
Whole field	188.8
ROI 1	8.298
ROI 2	3.289
ROI 3	6.316
Avg. ROI	5.968

- Pre-milling: bituminite reflectance 0.18% (s.d. 0.02, n=100)
- Post-milling: bituminite reflectance 0.38% (s.d. 0.03, n=100)





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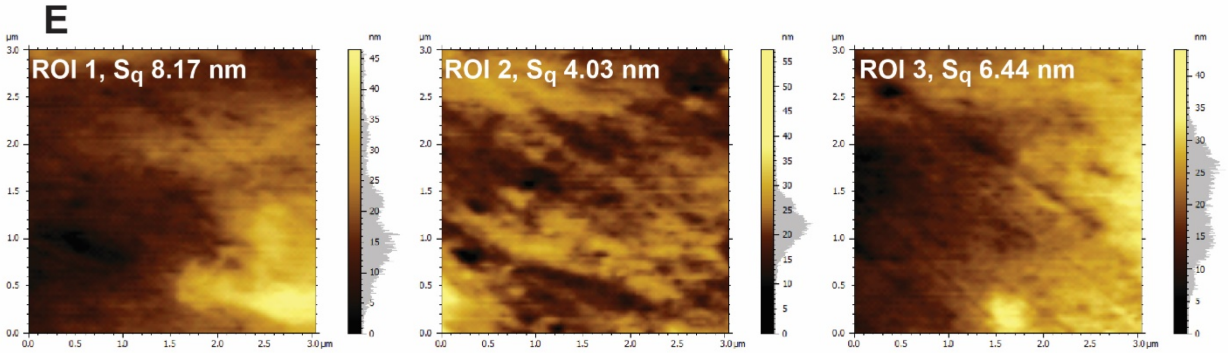
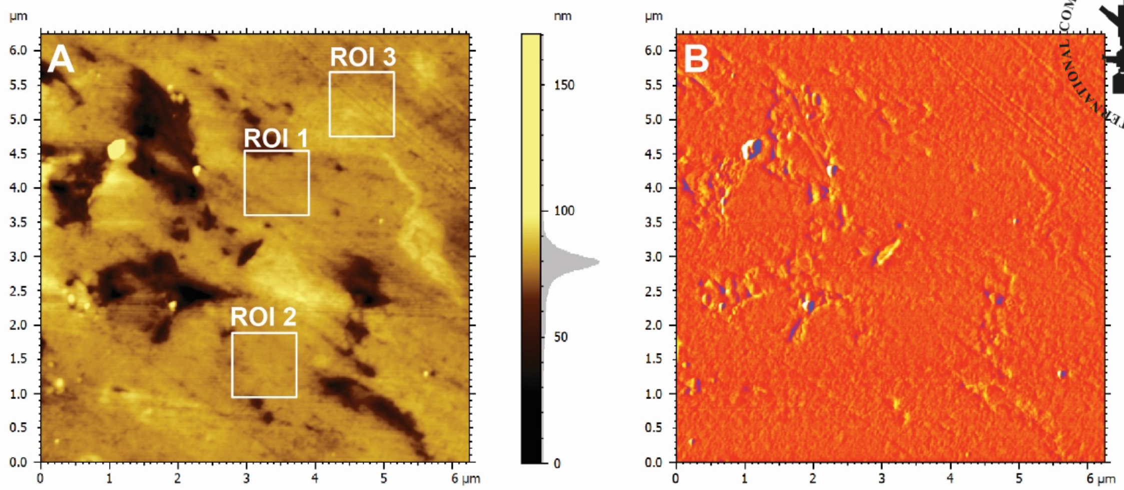
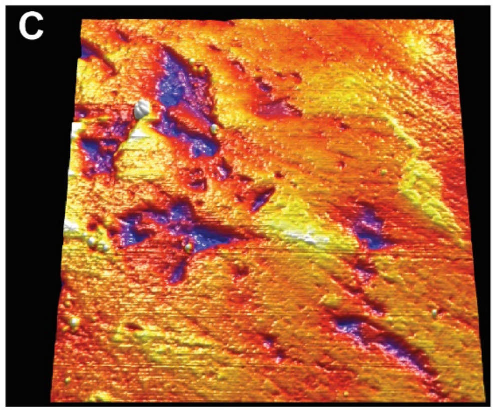
ISO 25178			
Height Parameters			
Sq	22.0	nm	*Root-mean-square height*
Ssk	-4.48		*Skewness*
Sku	46.9		*Kurtosis*
Sp	81.9	nm	*Maximum peak height*
Sv	395	nm	*Maximum pit height*
Sz	477	nm	*Maximum height*
Sa	13.3	nm	*Arithmetic mean height*







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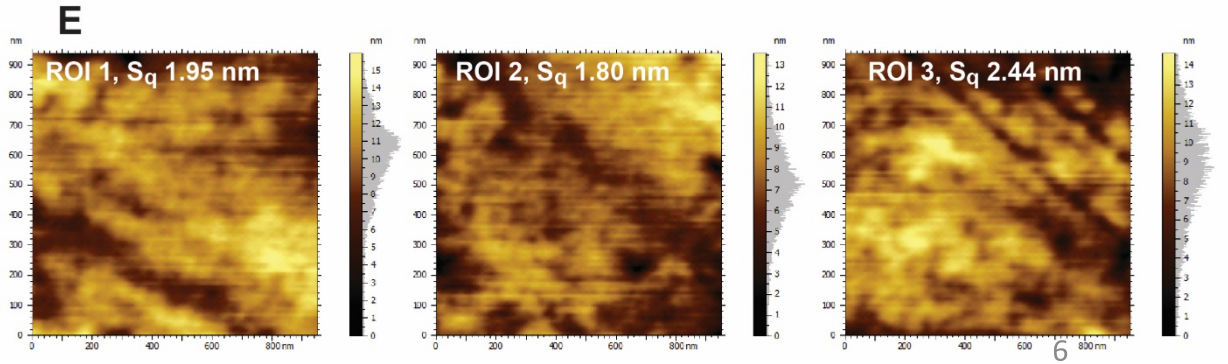
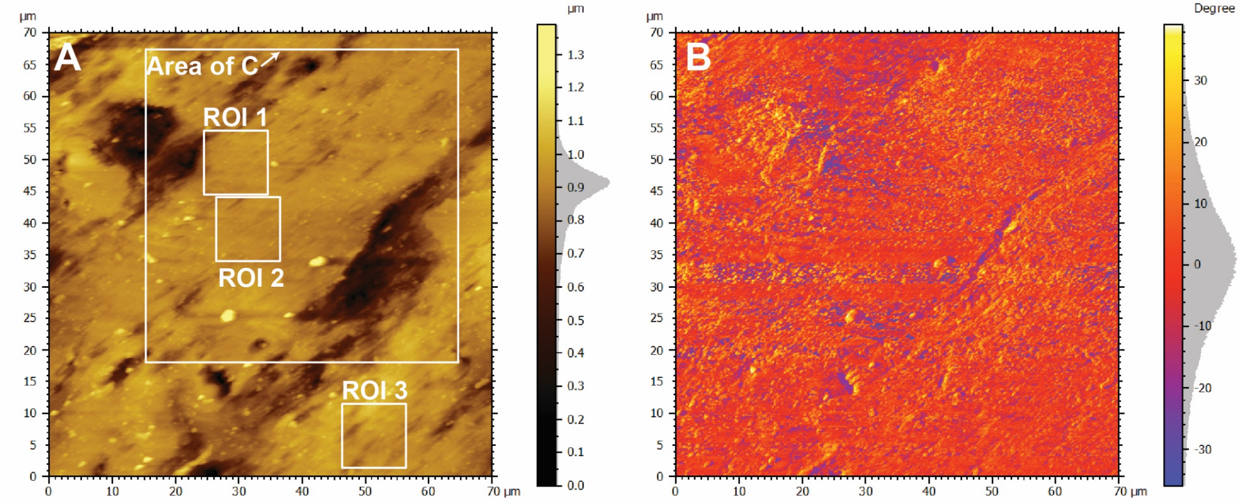
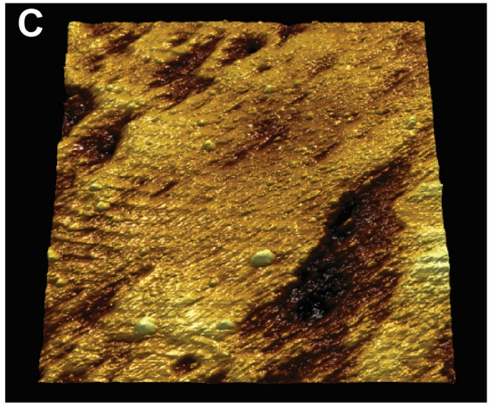
ISO 25178			
Height Parameters			
Sq	9.57	nm	*Root-mean-square height*
Ssk	-1.68		*Skewness*
Sku	12.2		*Kurtosis*
Sp	93.2	nm	*Maximum peak height*
Sv	77.0	nm	*Maximum pit height*
Sz	170	nm	*Maximum height*
Sa	6.08	nm	*Arithmetic mean height*







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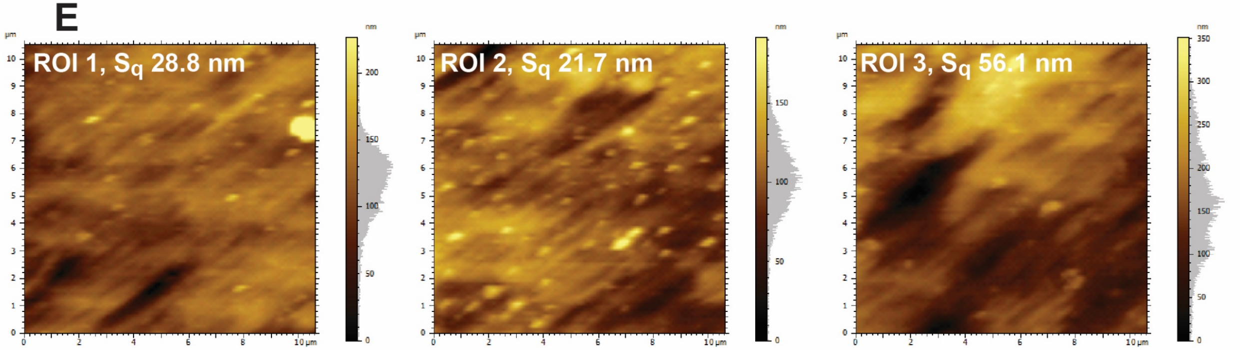
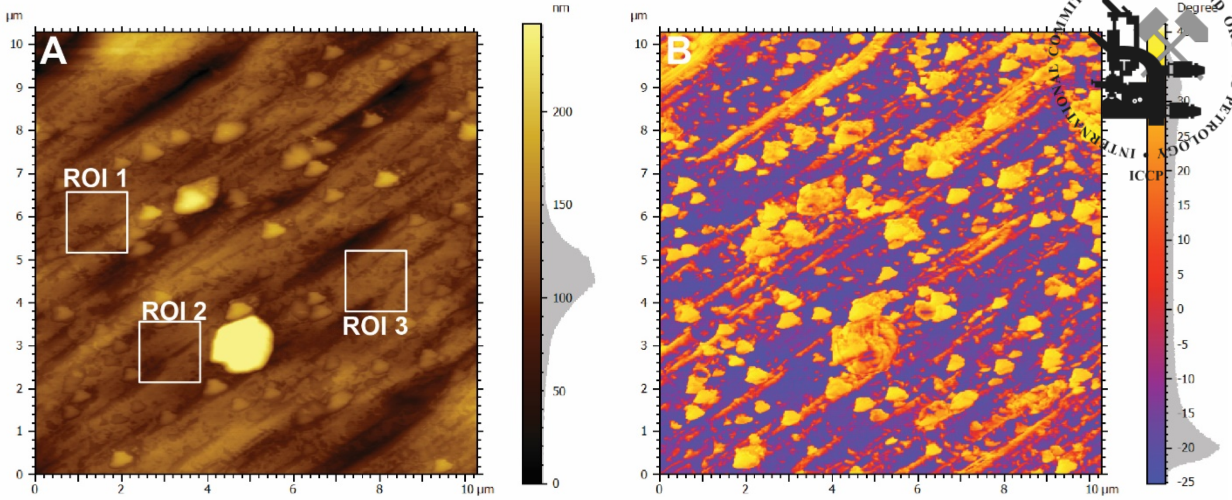
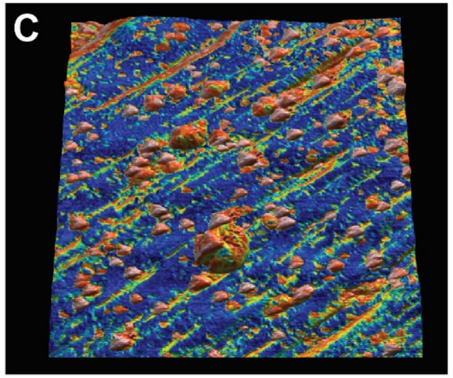
ISO 25178			
Height Parameters			
Sq	0.121	µm	*Root-mean-square height*
Ssk	-1.84		*Skewness*
Sku	8.07		*Kurtosis*
Sp	0.525	µm	*Maximum peak height*
Sv	0.865	µm	*Maximum pit height*
Sz	1.39	µm	*Maximum height*
Sa	0.0844	µm	*Arithmetic mean height*







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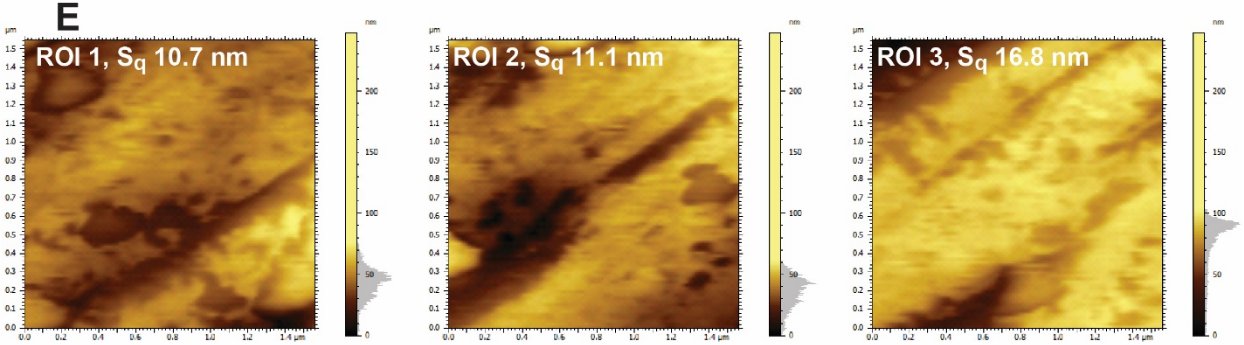
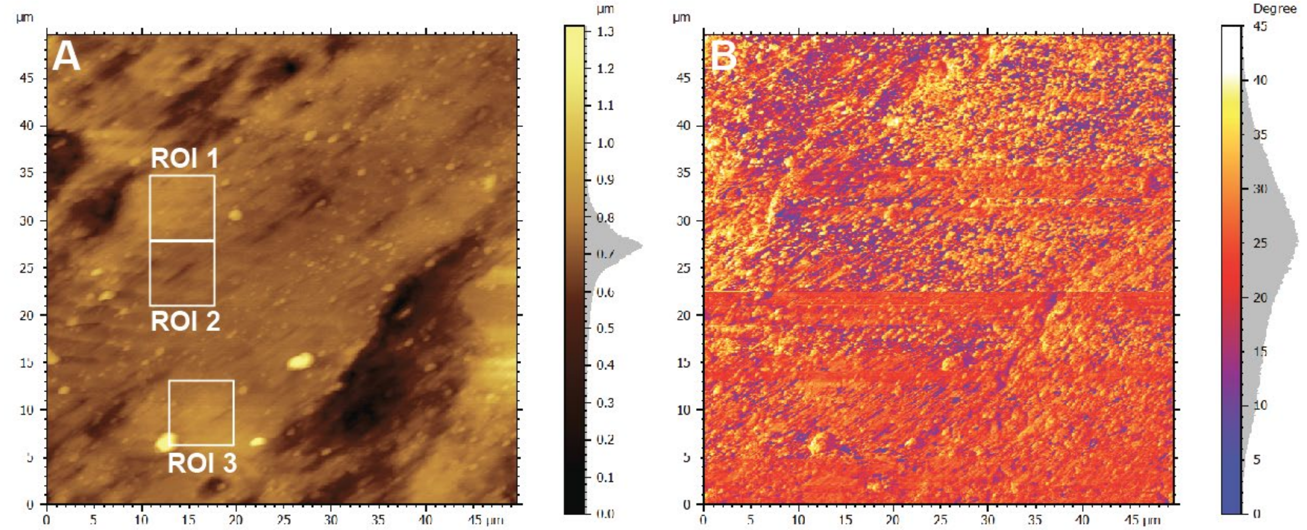
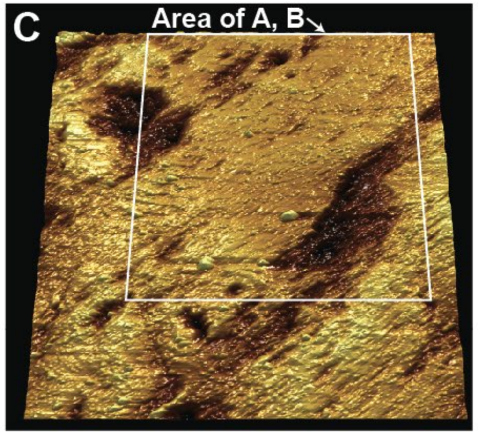
ISO 25178			
Height Parameters			
Sq	136	nm	*Root-mean-square height*
Ssk	0.011		*Skewness*
Sku	2.18		*Kurtosis*
Sp	331	nm	*Maximum peak height*
Sv	342	nm	*Maximum pit height*
Sz	672	nm	*Maximum height*
Sa	114	nm	*Arithmetic mean height*







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ISO 25178			
Height Parameters			
Sq	0.108	µm	*Root-mean-square height*
Ssk	-1.38		*Skewness*
Sku	8.77		*Kurtosis*
Sp	0.616	µm	*Maximum peak height*
Sv	0.698	µm	*Maximum pit height*
Sz	1.31	µm	*Maximum height*
Sa	0.0702	µm	*Arithmetic mean height*

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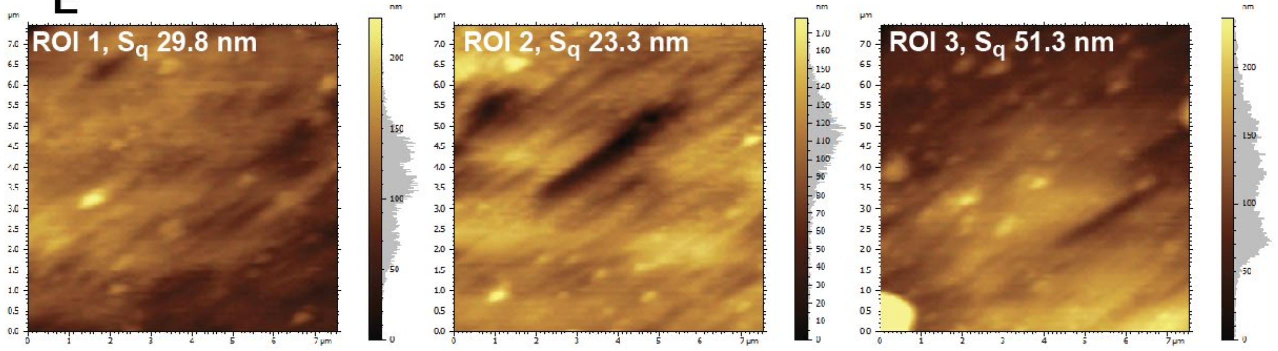

BIB/AFM Summary

- ❑Reflectance increase after BIB milling
- ❑Differential milling based on hardness – exposure of nano-sulfides, quartz
- ❑BIB-induced surface flattening of bituminite measured by correlative AFM
- ❑BIB-induced surface roughening caused by differential milling
- ❑Wider distribution of mechanical properties post-milling, exposure of nano-sulfides
- ❑Smearing of OM during mechanical polish disguises nano-sulfides(?)
- ❑Scale of observation matters: smaller fields give lower S_q
- ❑More work is required

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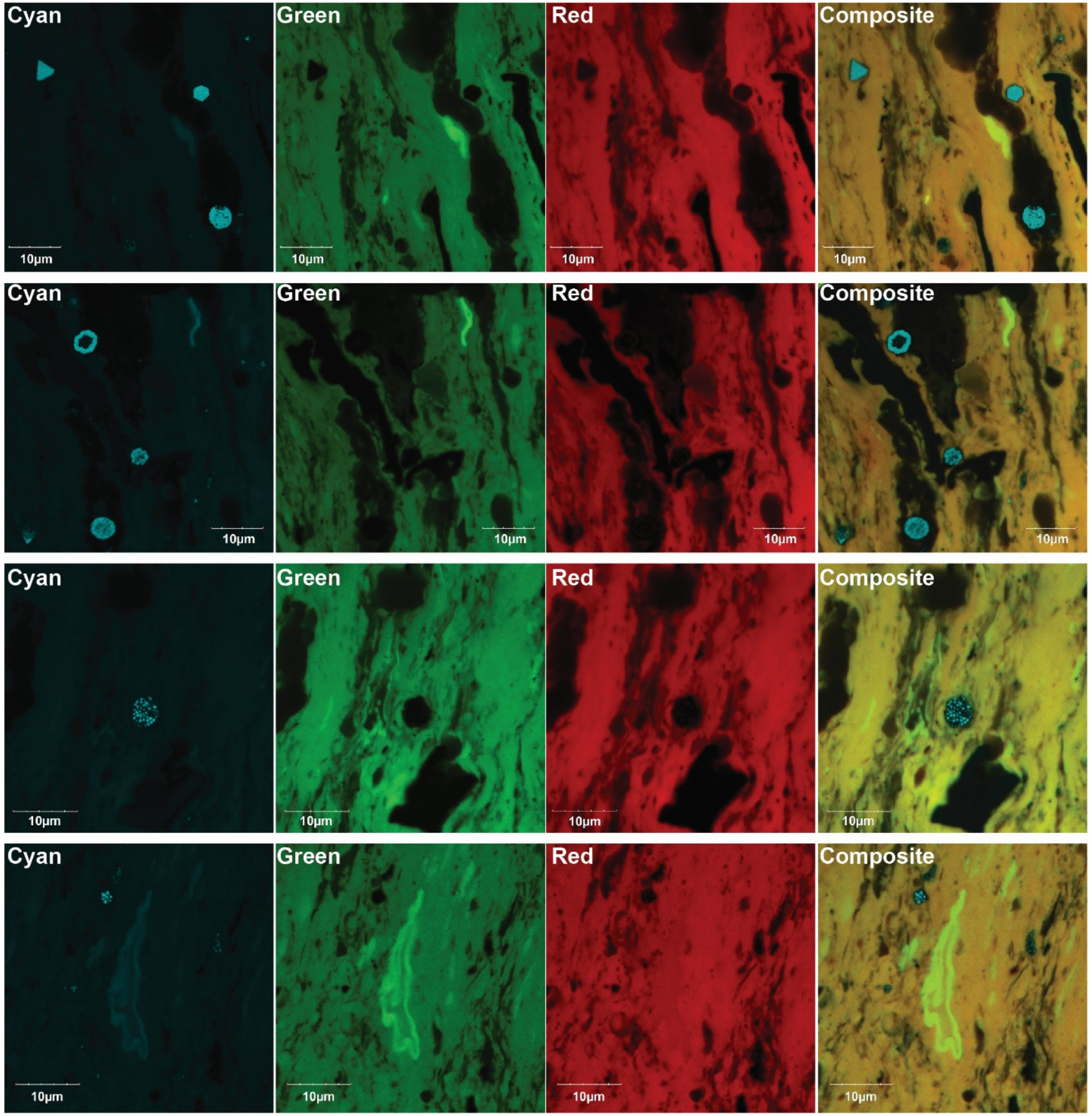
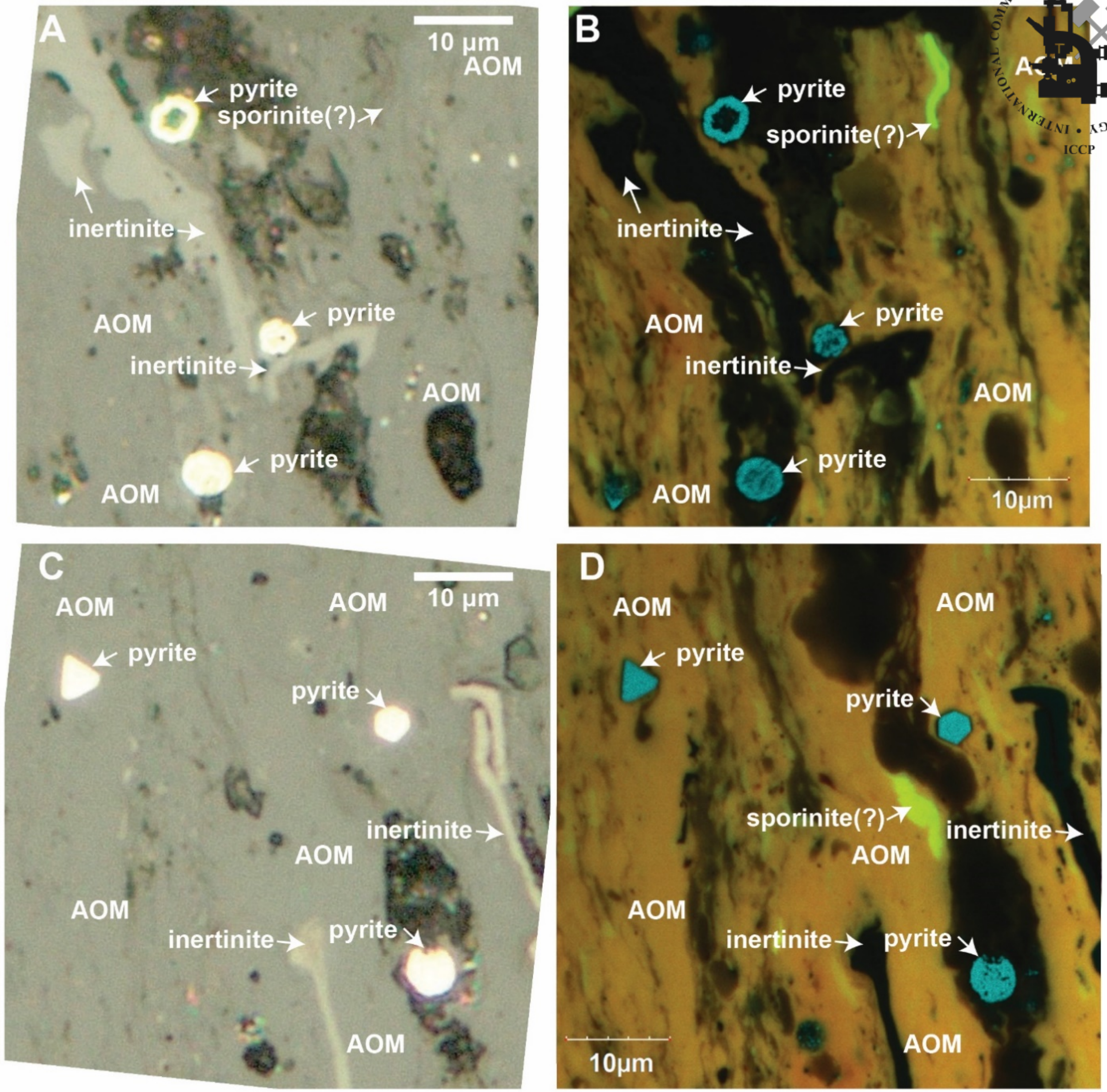
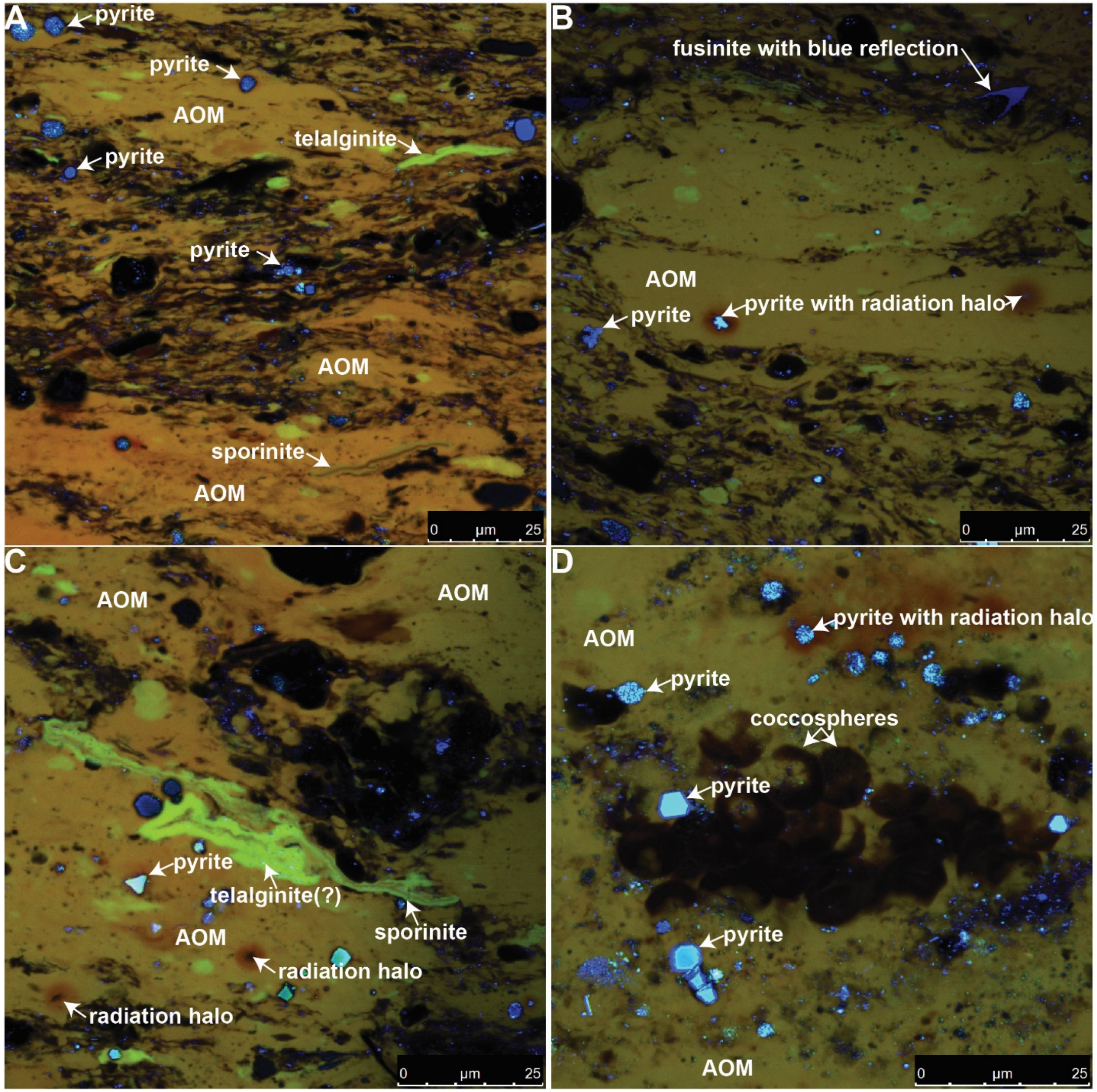
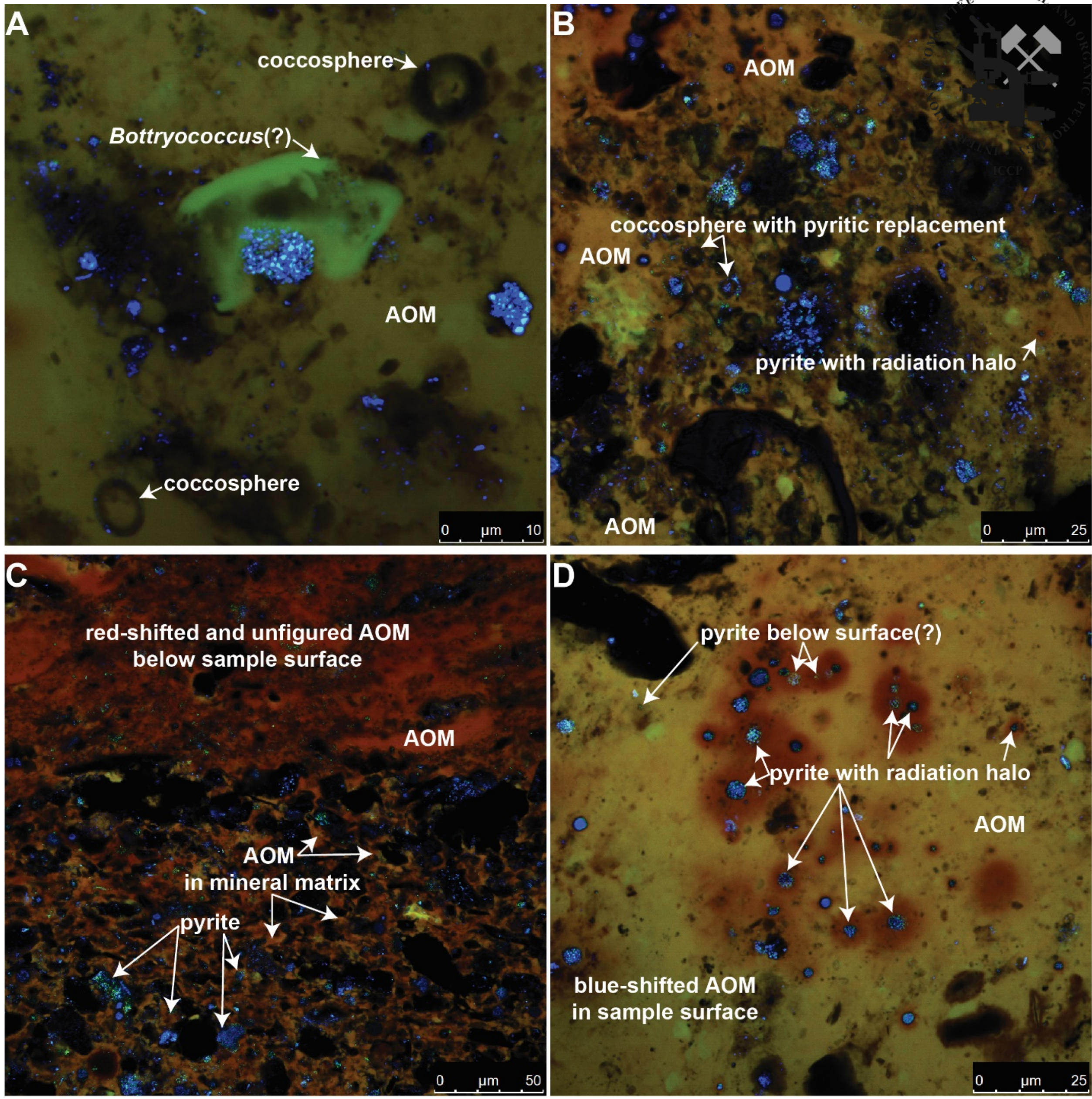




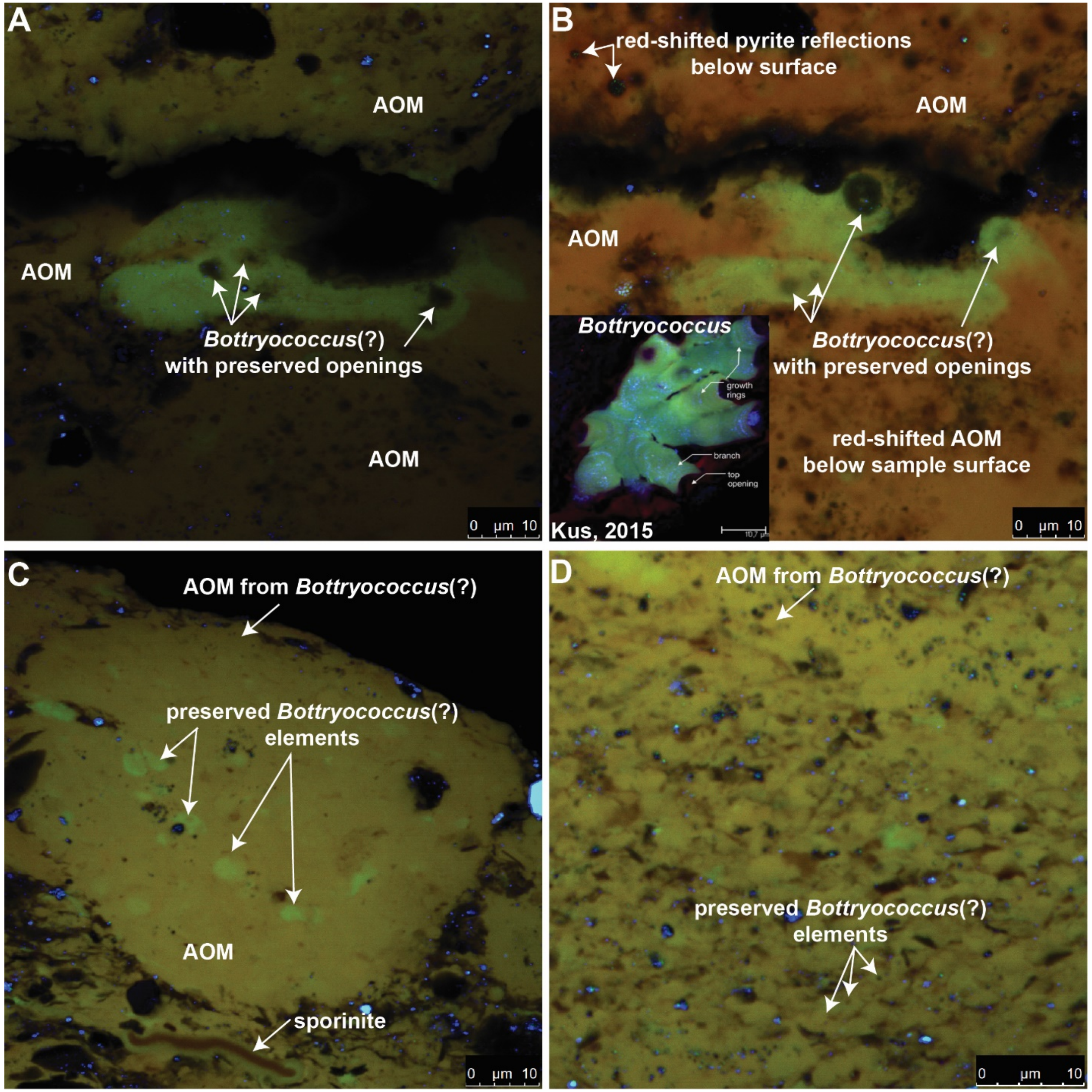
- ❑Construction of 3-channel false color images
- ❑Reflectance of 458 nm laser from sulfides





- ❑Reflectance of 458 nm laser from sulfides
- ❑U substitution for Fe: radiation halos

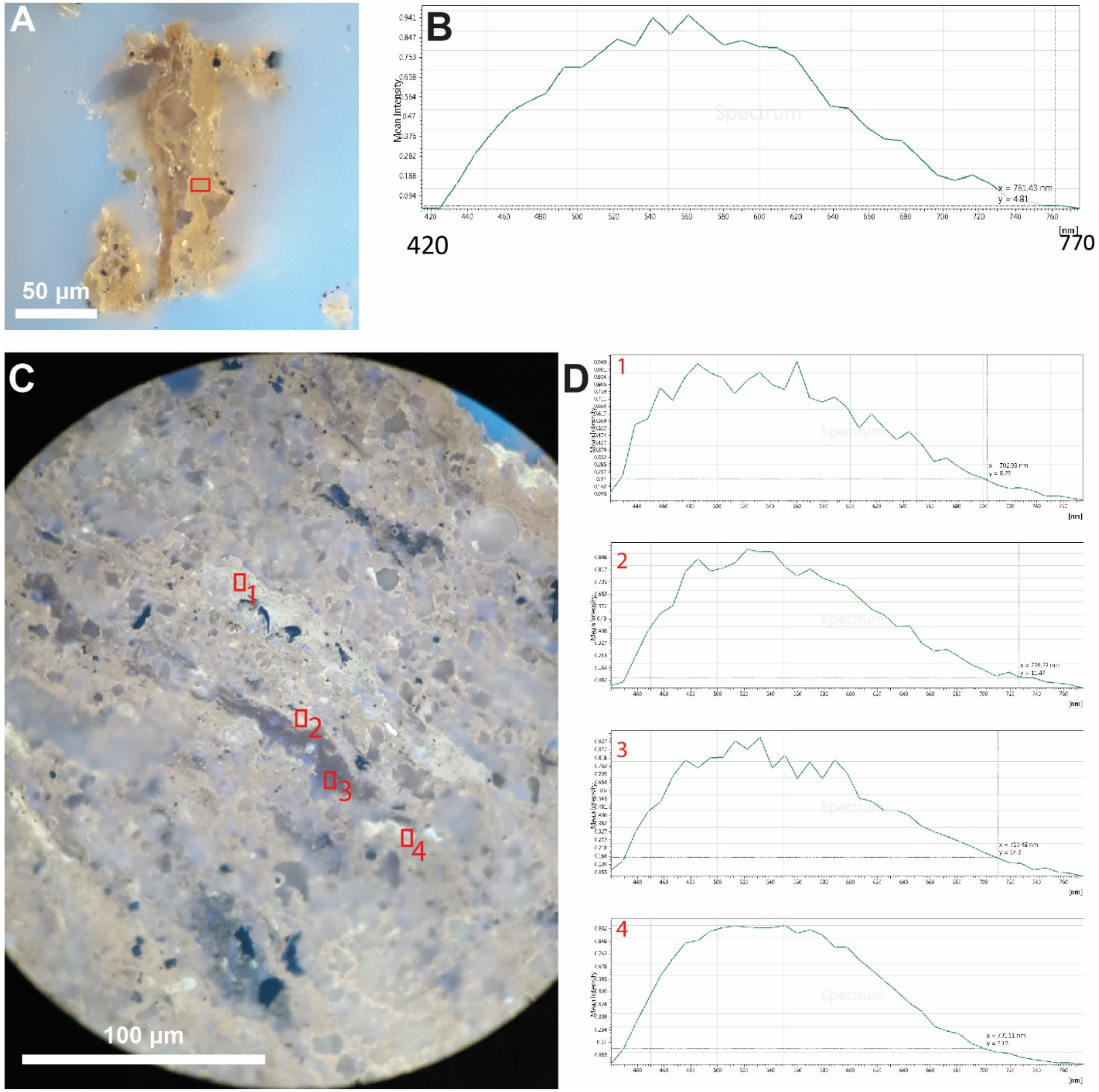
- ❑*Bottryococcus(?)*
- ❑Red-shifted fluorescence/reflectance below surface



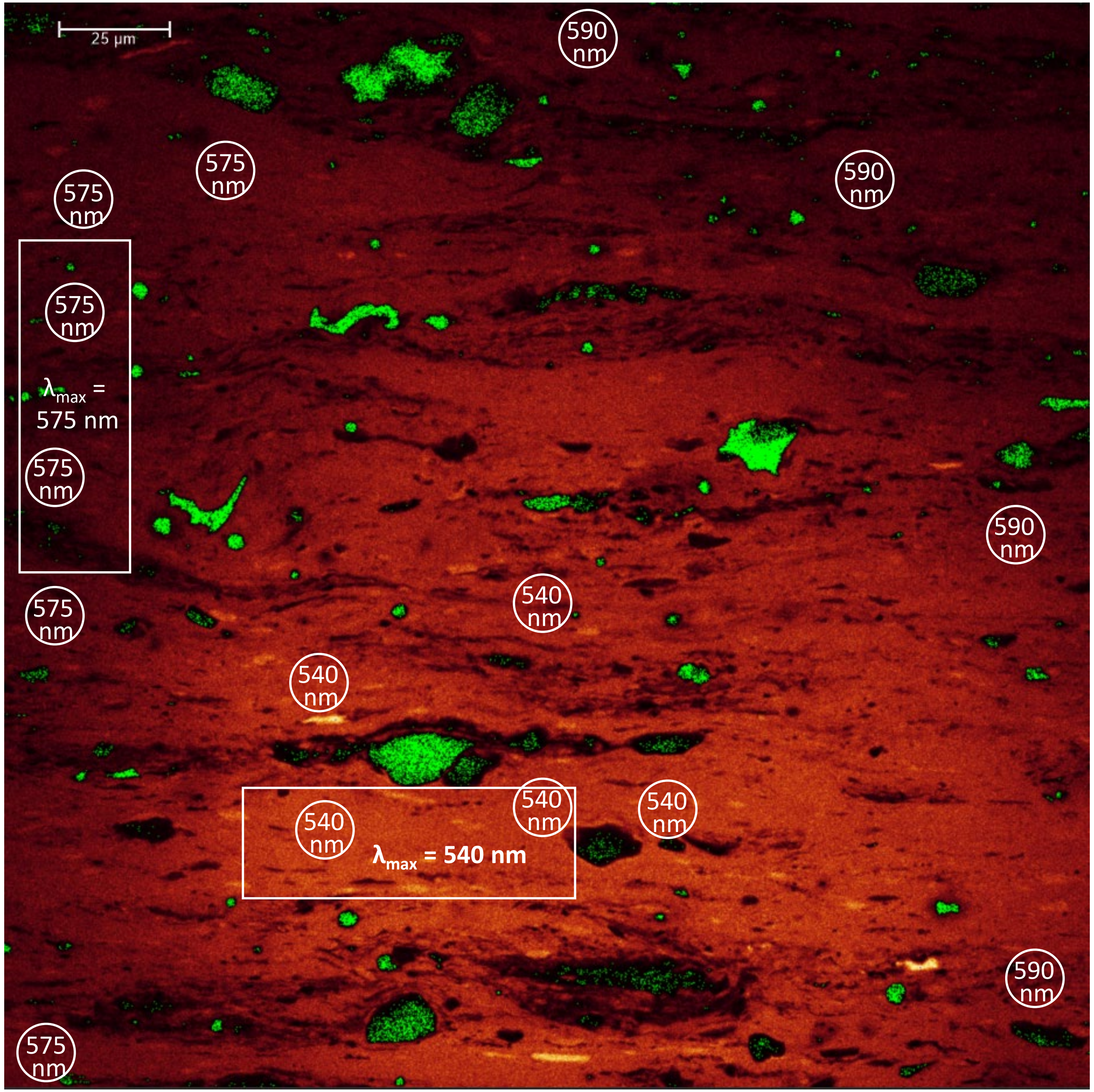
CLSM Imaging Summary

- ❑Reflectance of 458 nm laser
- ❑U substitution for Fe
- ❑*Bottryococcus(?)*
- ❑Red-shifted fluorescence and reflectance below sample surface

CLSM Spectroscopy

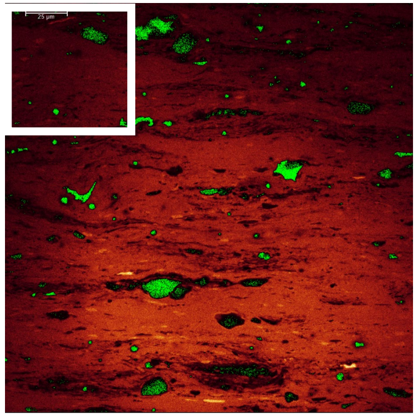


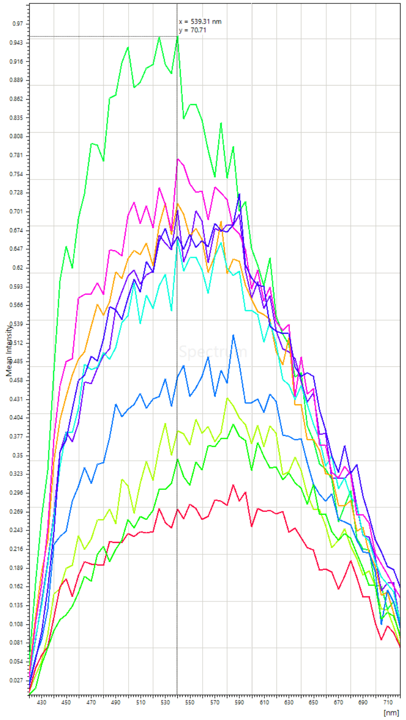
- B: λ_{max} 562 nm (405 nm diode)
- Calc. BR_o 0.29% (Stasiuk, 1994)
- B+D: Avg. λ_{max} 539 nm (n=5)
- Calc. BR_o 0.21% (Stasiuk, 1994)

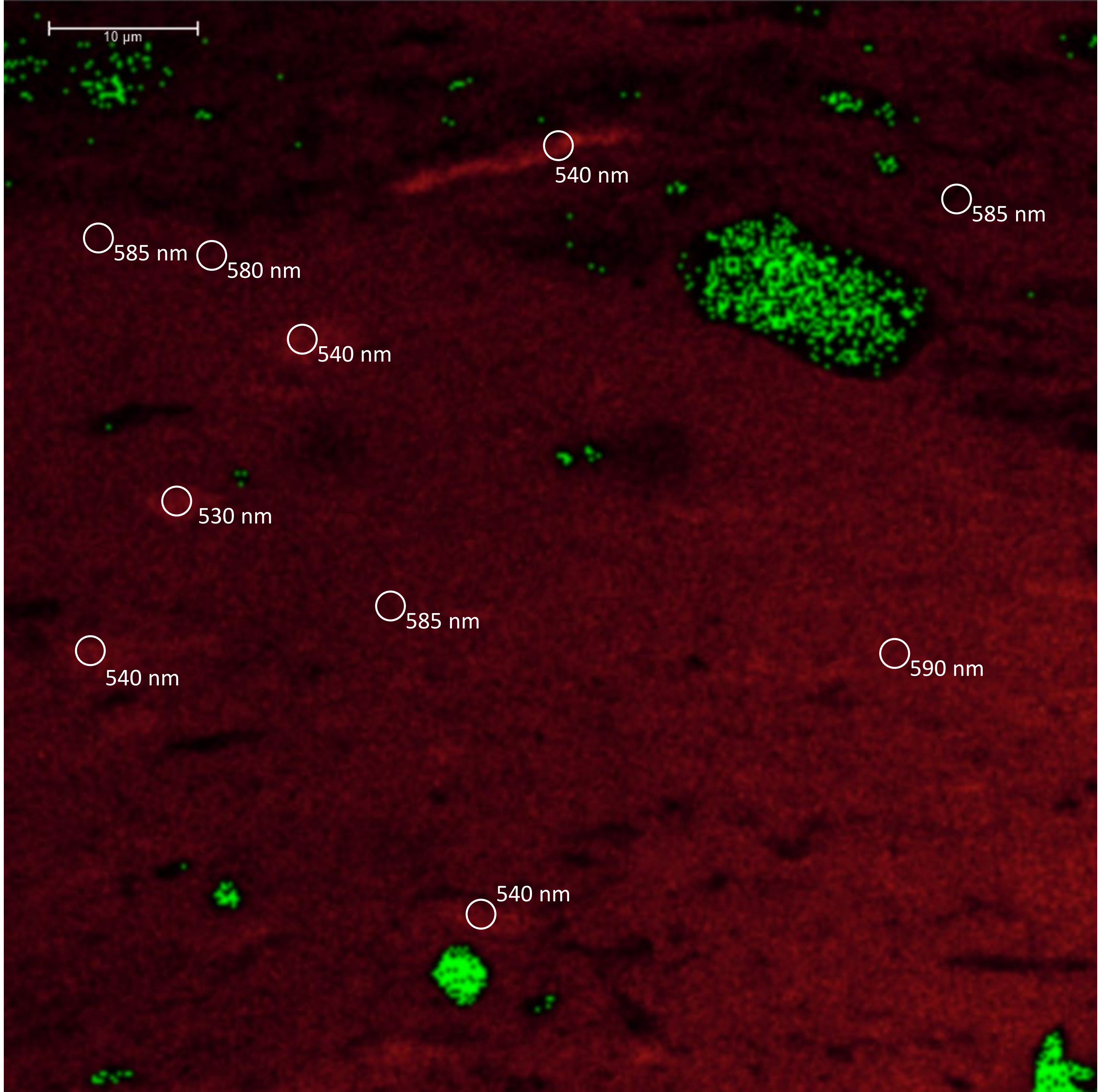


CLSM Spectroscopy

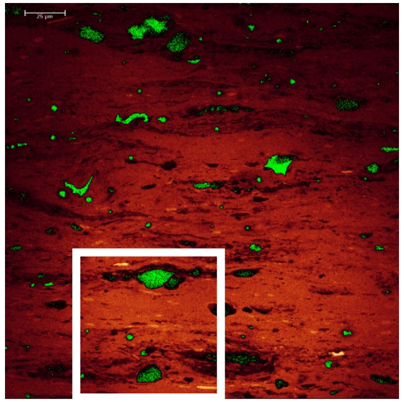
- ❑Unintentional photo-oxidation
- ❑Unaltered λ_{max} 575 nm
- ❑Altered λ_{max} 540 nm
- ❑Calc. BR_o 0.32% (Stasiuk, 1994) from unaltered area

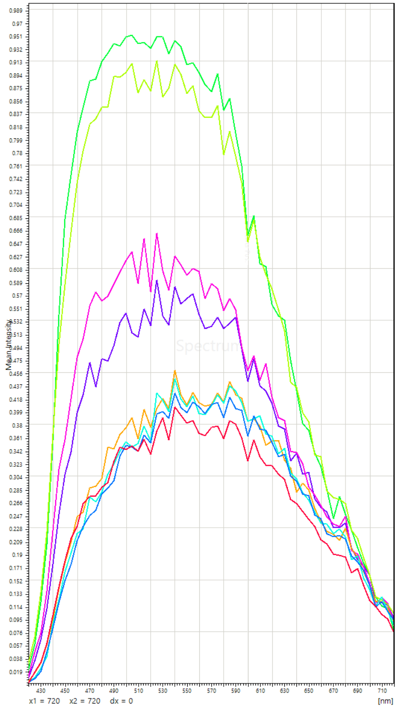


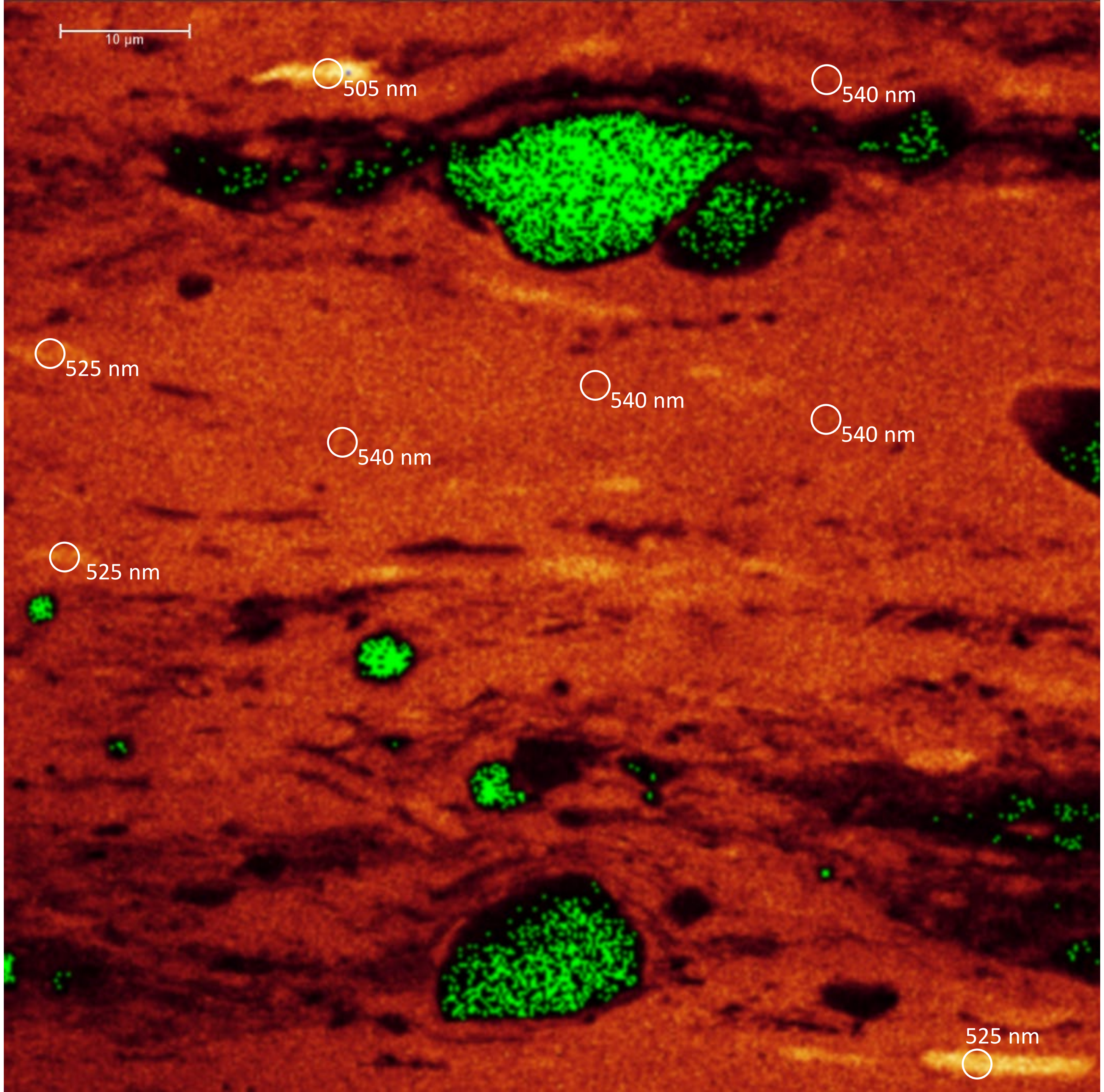




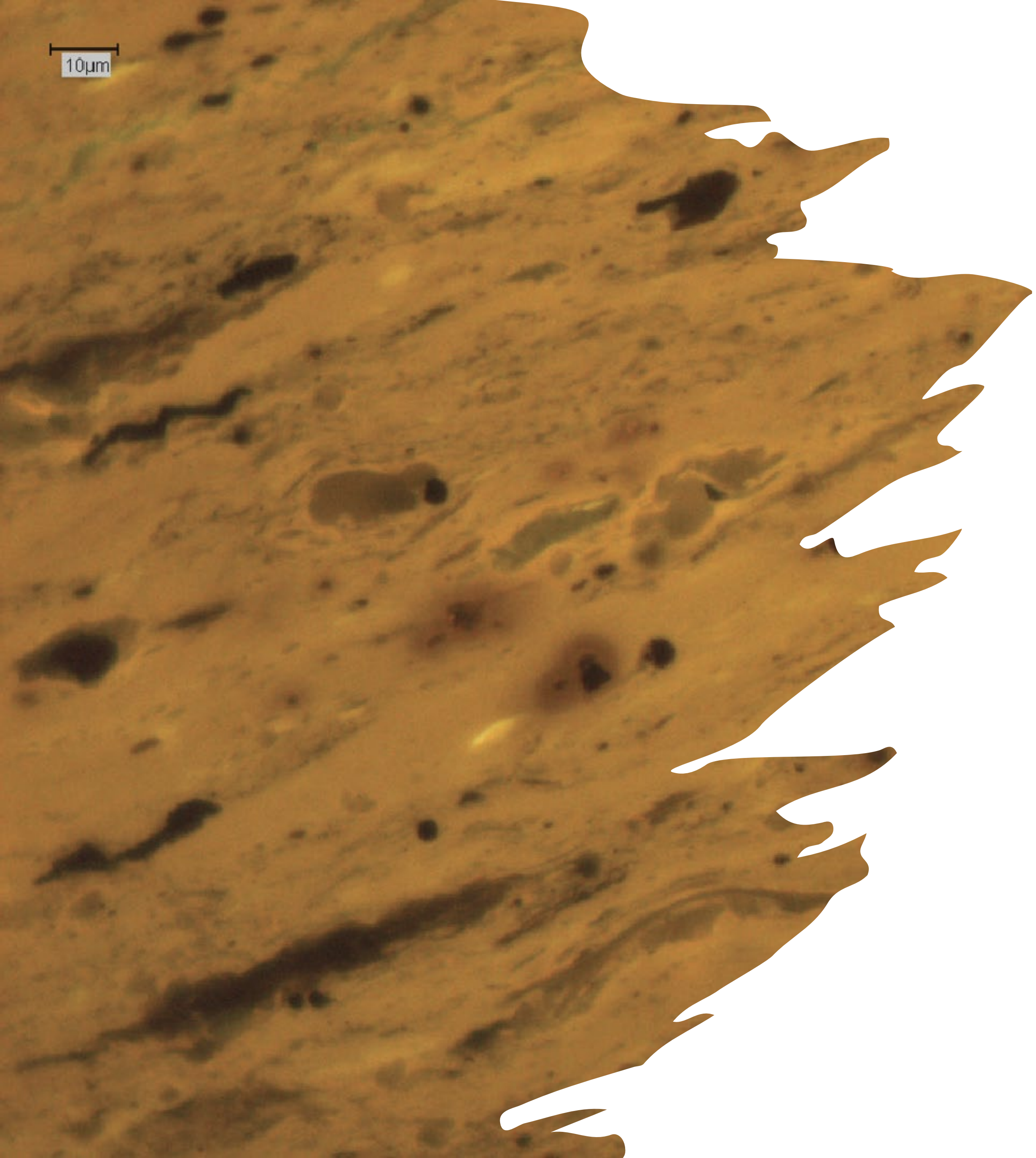
- Unaltered region
- Higher emission intensity blue-shift
- λ_{max} 538 nm, avg. high intensity
- λ_{max} 585 nm, avg. lower intensity







- Altered region
- Higher emission intensity blue-shift
- λ_{max} 520 nm, avg. high intensity
- λ_{max} 540 nm, avg. lower intensity

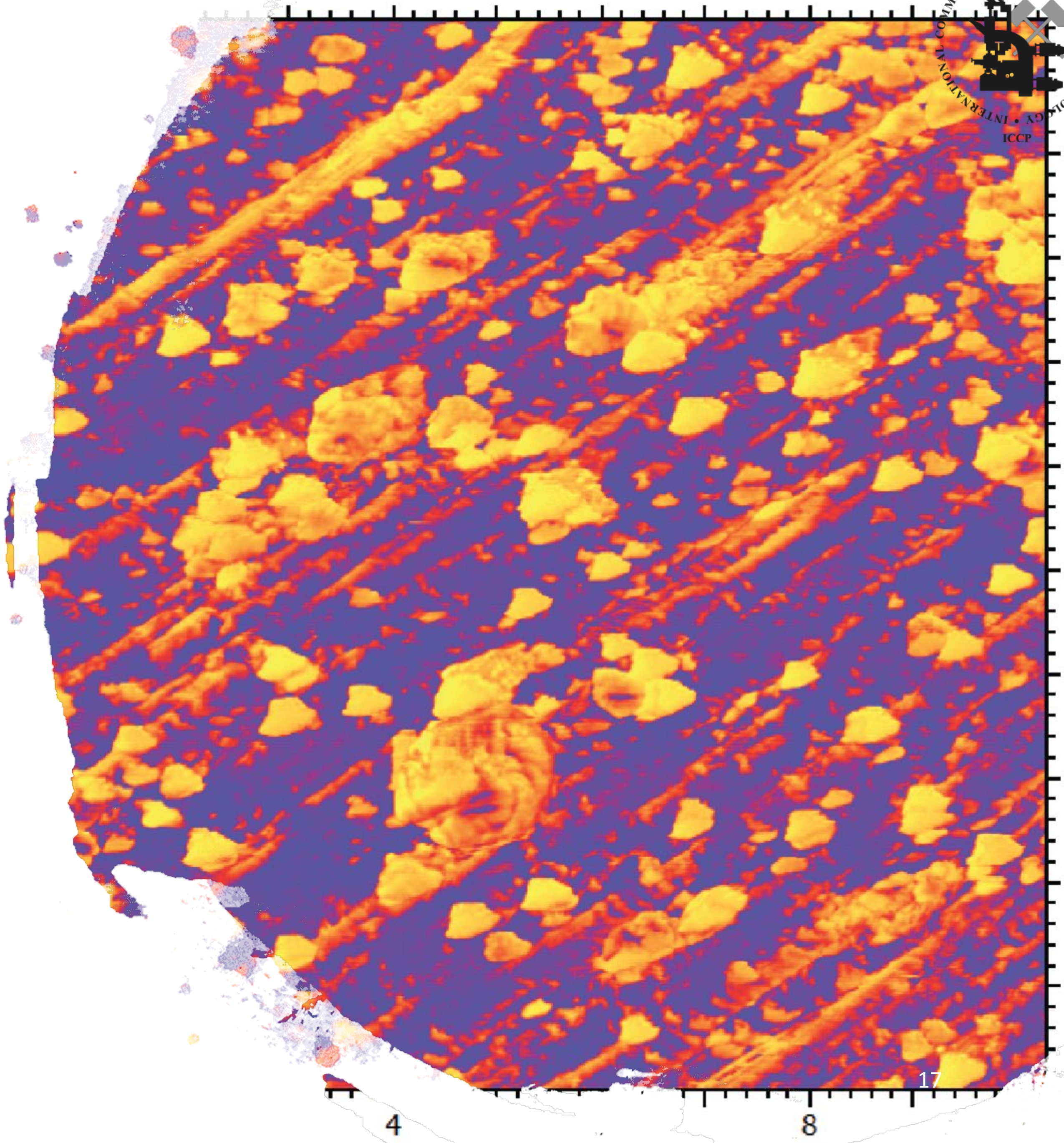


Summary

- KC sample characterized by AFM, CLSM
- Differential milling via BIB
- Reflectance increase via BIB
- CLSM identified *Bottryococcus(?)*
- Reflectance of 458 nm laser
- Red-shift of reflectance and fluorescence from below surface
- λ_{max} measurement requires standardization
- Empirical relation from Stasiuk matches measured BR_o
- Photooxidation causes positive alteration, color blue-shift

Next Steps

- ❑Provide final CLSM WG report
- ❑Update webpage
- ❑Notification to ICCP News
- ❑Peer-reviewed manuscript



Thanks ICCP!

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