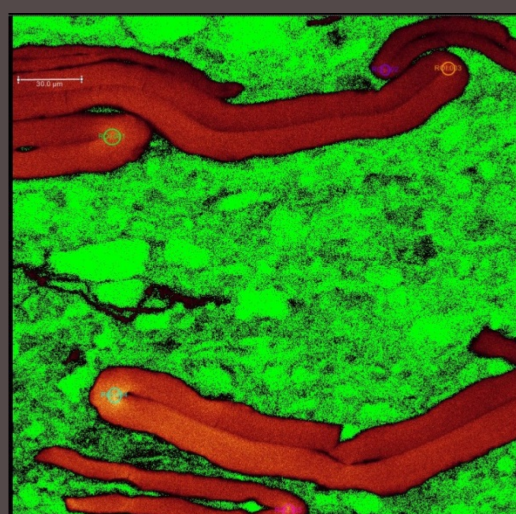
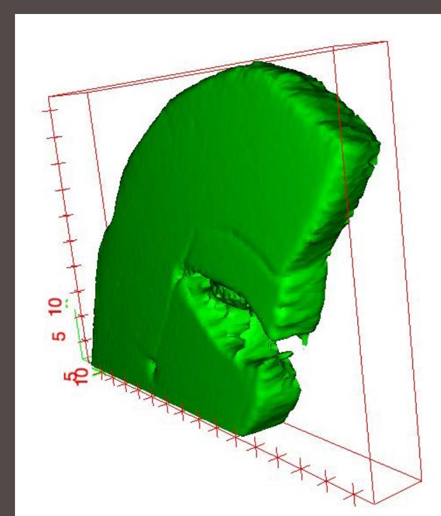
ICCP CONFOCAL LASER SCANNING MICROSCOPY (CLSM) WORKING GROUP 2018

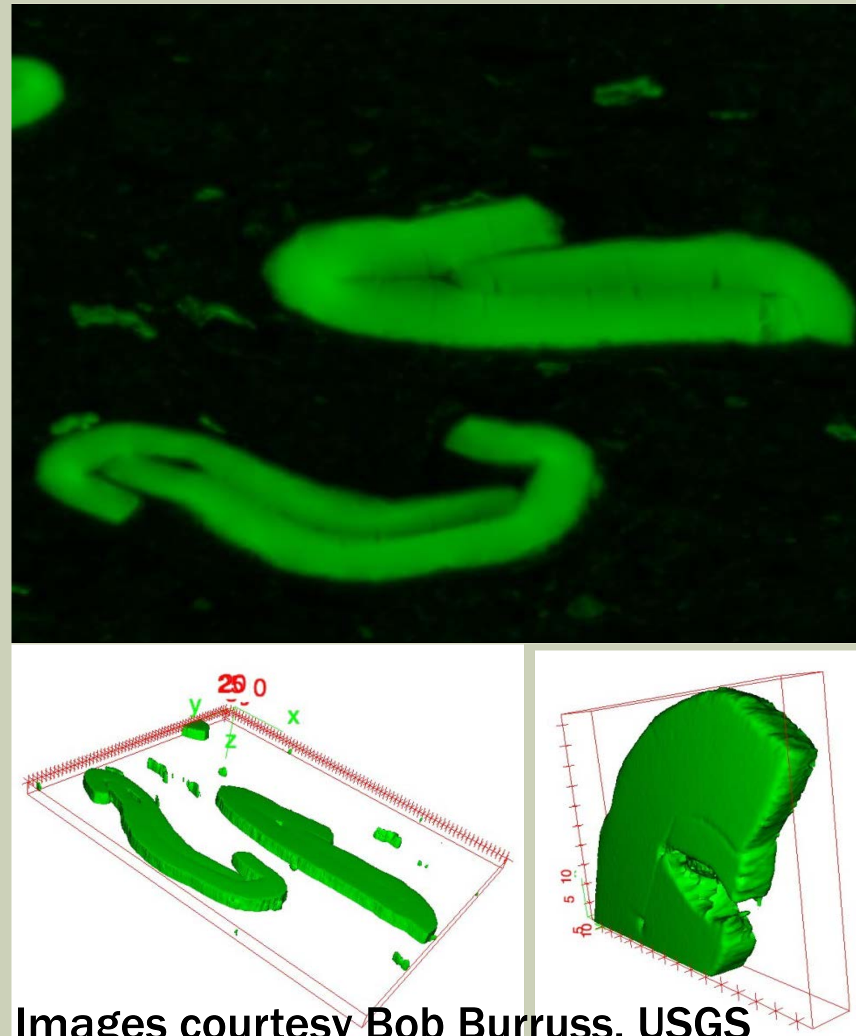
Paul C. Hackley
Jolanta Kus





WHY USE CLSM ON SEDIMENTARY ORGANIC MATTER?

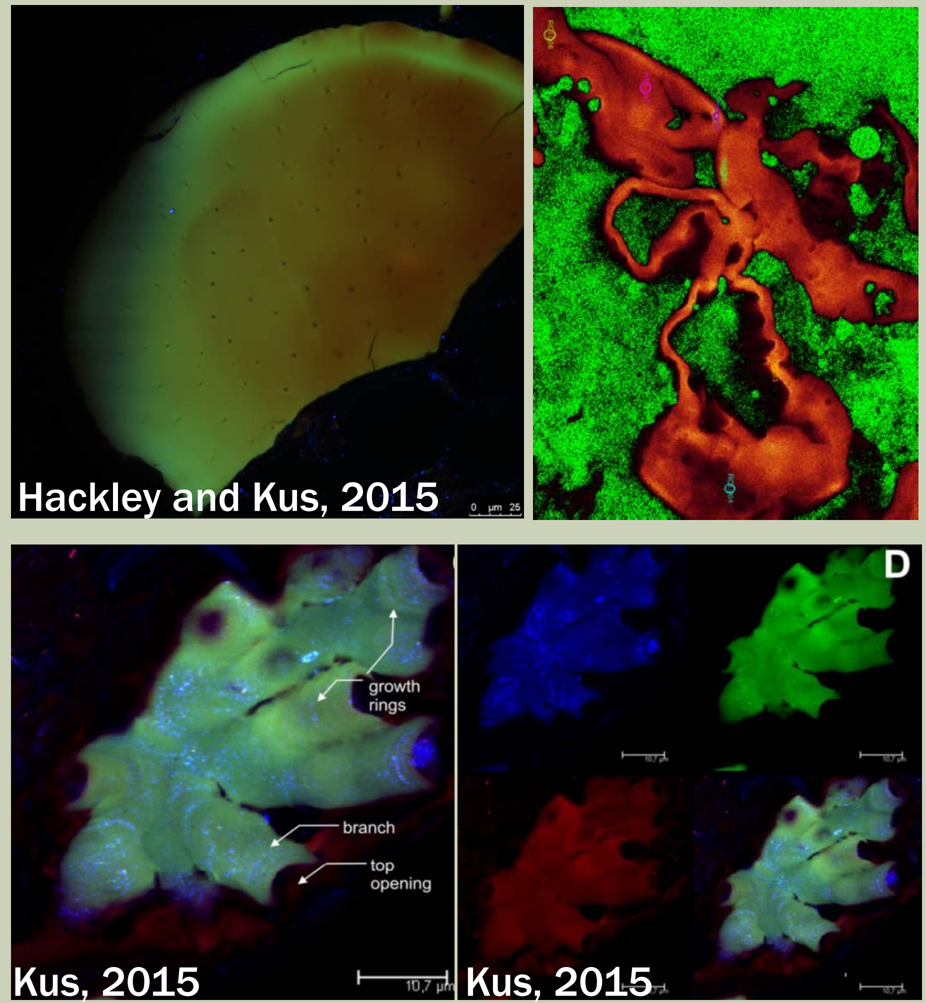
- Many applications
- Technique is broadly underutilized
- High resolution (~200 nm)
- 2-D and 3-D imaging
- 3-D reconstructions, video
- Non-destructive
- Fluorescence spectroscopy
- Useful for organic petrology!



Images courtesy Bob Burruss, USGS

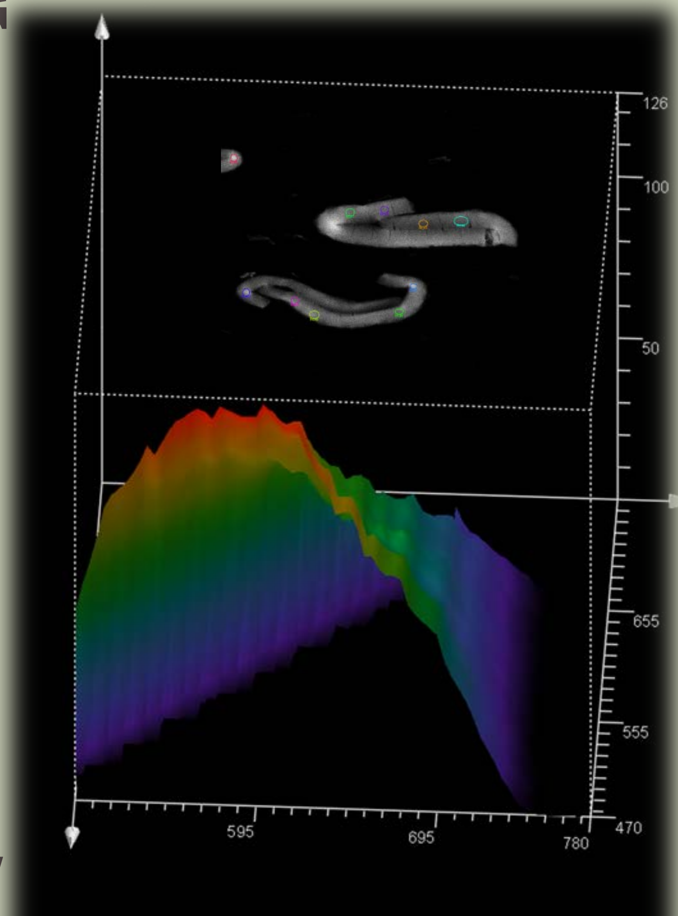
CLSM WG UPDATE 2018

- Working Group approved in Potsdam 2015
- Note in ICCP News 63, 2015
- Presentation at GSA, Baltimore Nov 2015
- User survey sent Feb 2016
- WG presentations Houston 2016, Bucharest 2017
- Webpage established October 2017 (thanks Stavros)



ACTIVITIES 2018

- Kimmeridge sample selected for WG study in late 2017
- Sample distributed April 2018 to 13 participants, with no instructions
- Results received from 2 ½ participants up to September 2018
- Outline from here: sample information, supporting work (ion milling), CLSM WG results up to now

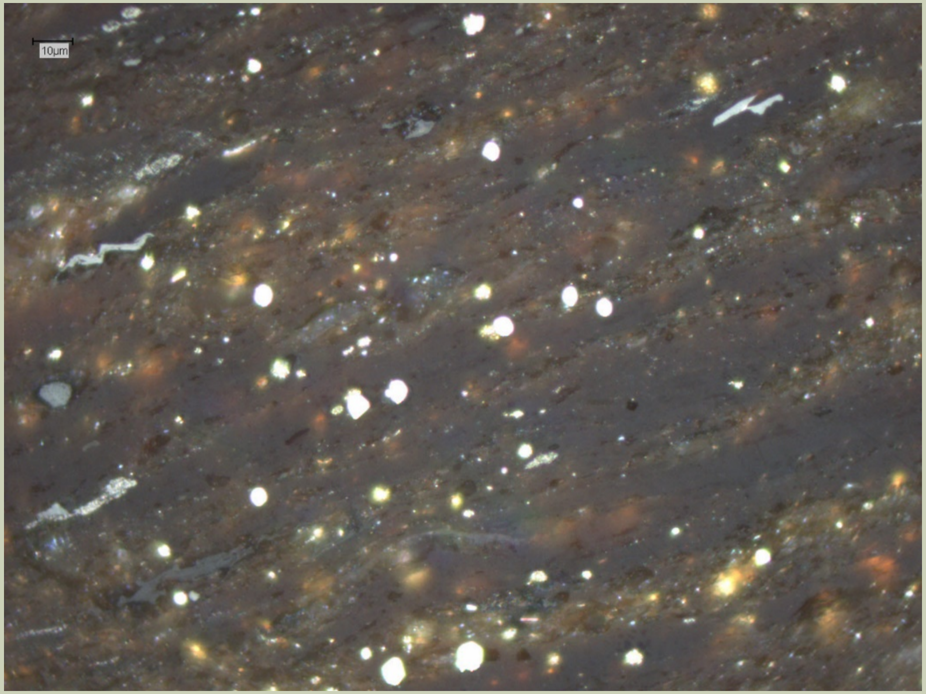


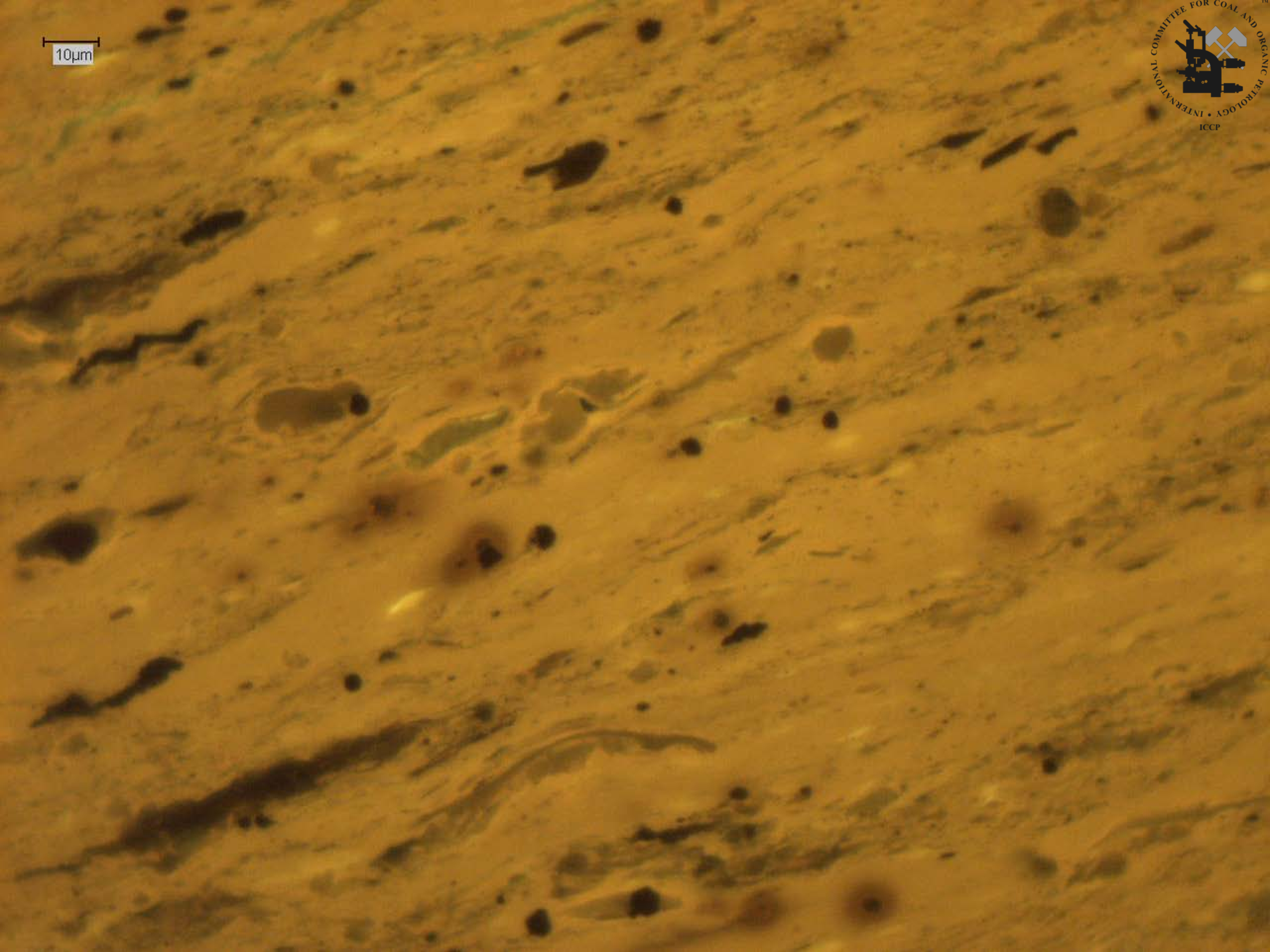
Hackley et al., 2013

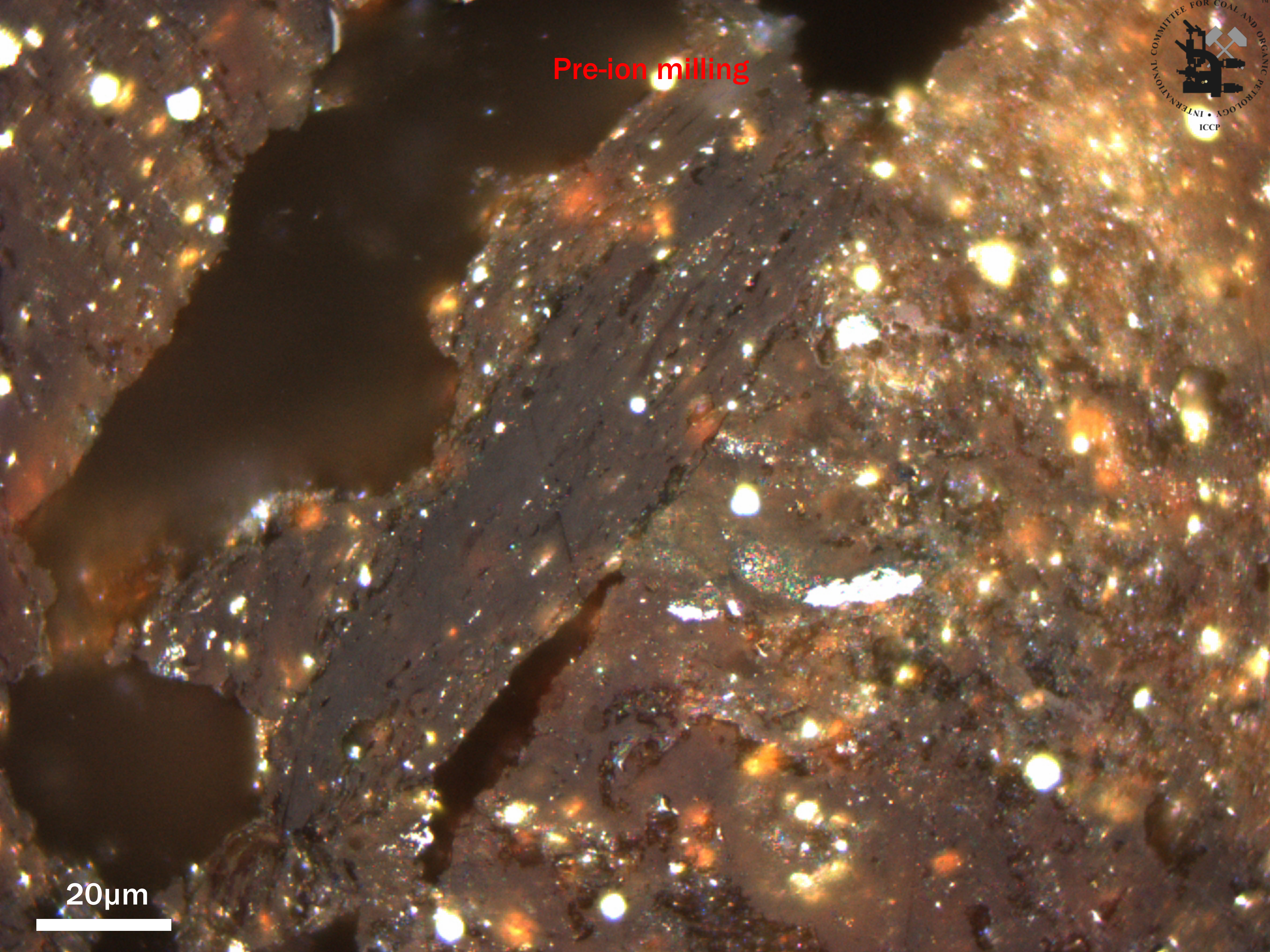
CLSM WG SAMPLE: KIMMERIDGE CLAY

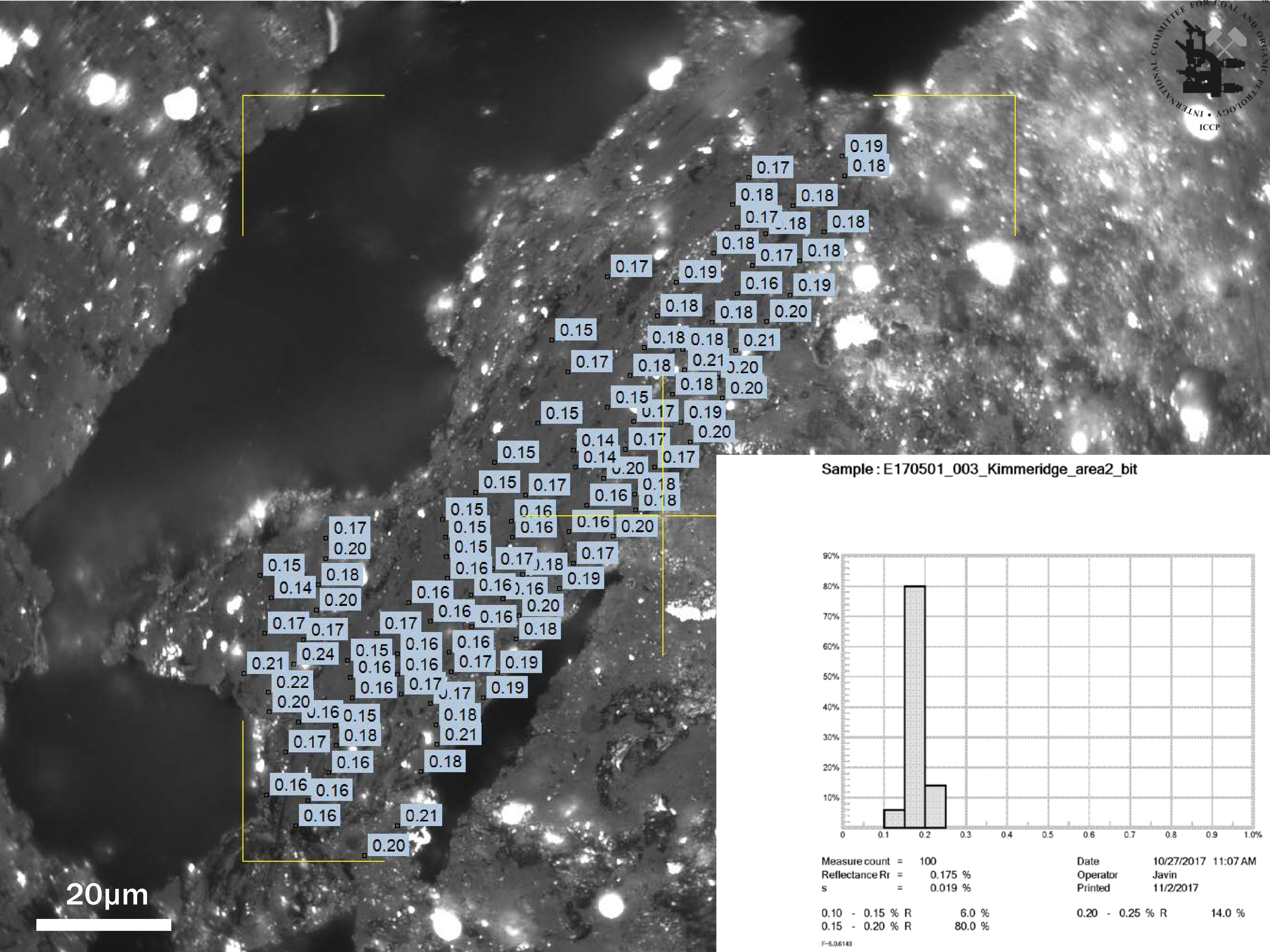
Kimmeridge Clay: KC-1

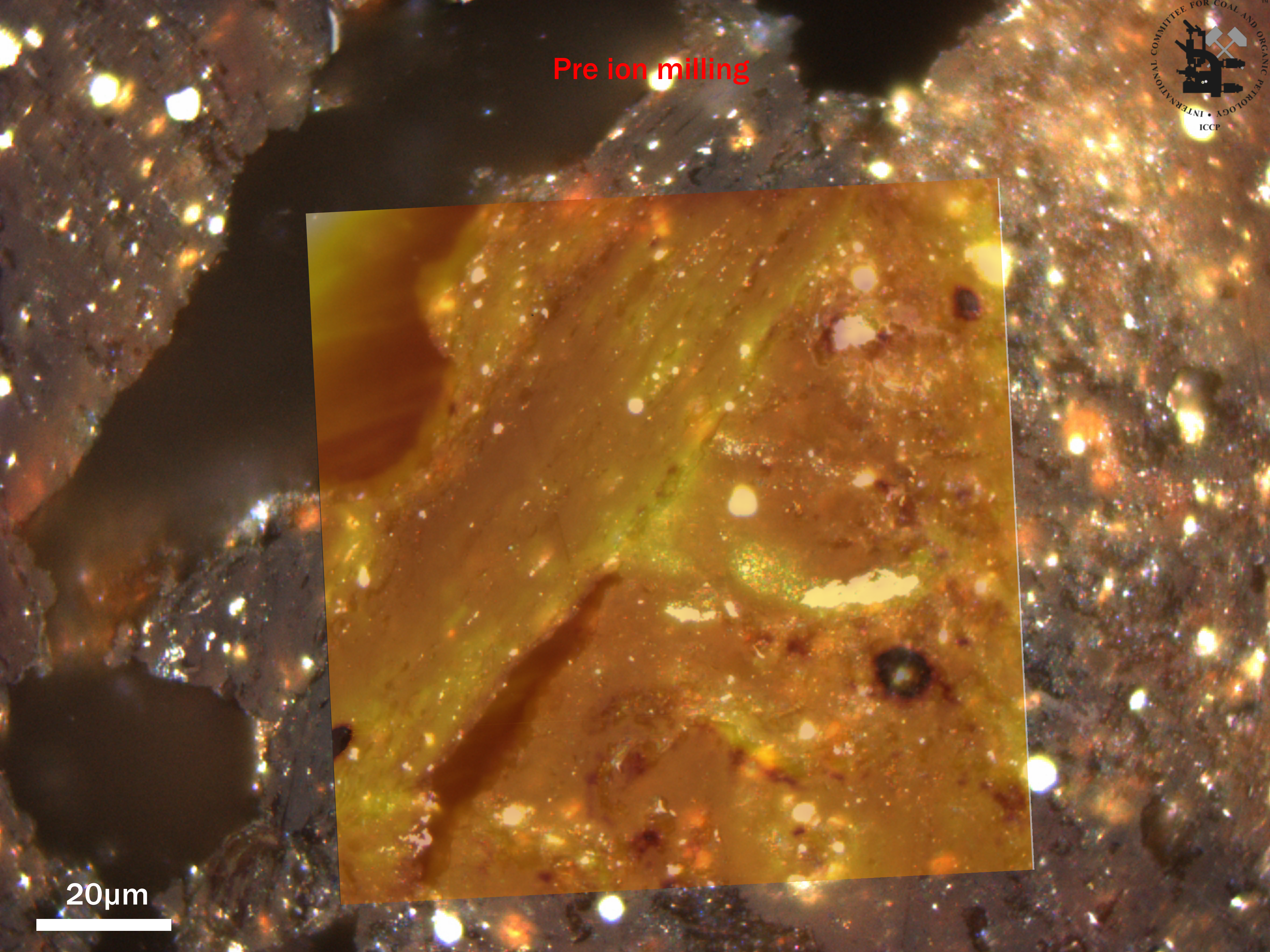
- Kimmeridge Blackstone
- BRo: 0.29%
- VRo: 0.42%
- Tmax: 409°C
- TOC: 44.1 wt.%
- HI: 737 mg HC/g TOC
- Fluorescent AOM, minor solid bitumen, vitrinite/inertinite, micrinite
- Other data ....

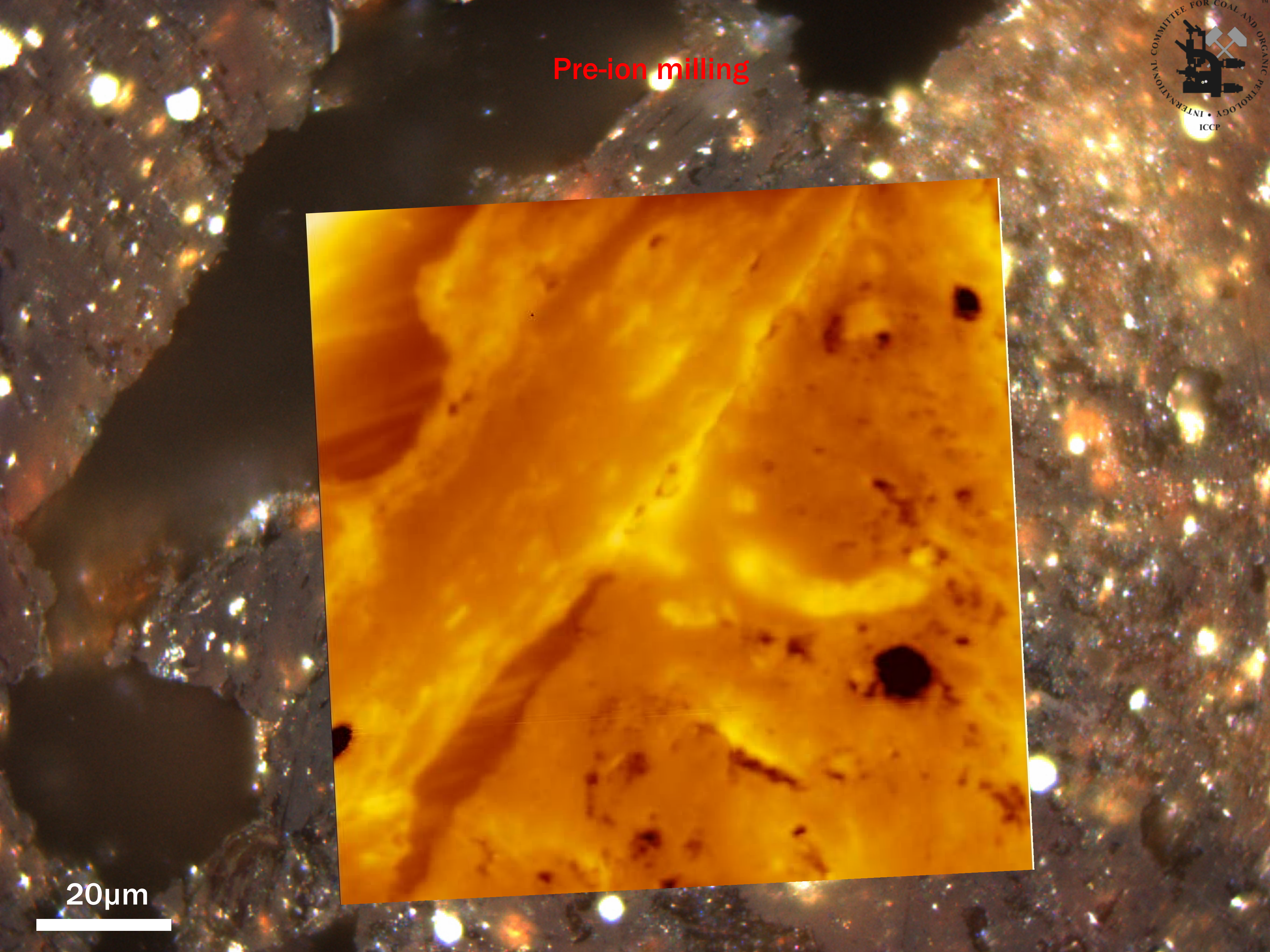



10µm

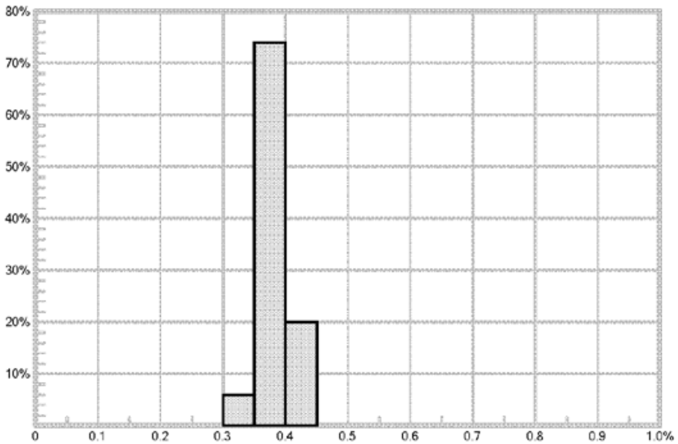

Pre-ion milling
20μm
INTERNATIONAL COMMITTEE FOR COAL AND ORGANIC PETROLOGY
ICCP


20µm
Sample : E170501_003_Kimmeridge_area2_bit
Measure count = 100
Reflectance Rr = 0.175 %
s = 0.019 %
Date 10/27/2017 11:07 AM
Operator Javin
Printed 11/2/2017
0.10 - 0.15 % R 6.0 %
0.15 - 0.20 % R 80.0 %
0.20 - 0.25 % R 14.0 %
F-6.0.6143


Pre ion milling
20µm


Pre-ion milling
20µm

Sample : E170501_003a_Kimmeridge_post_ion1_bit



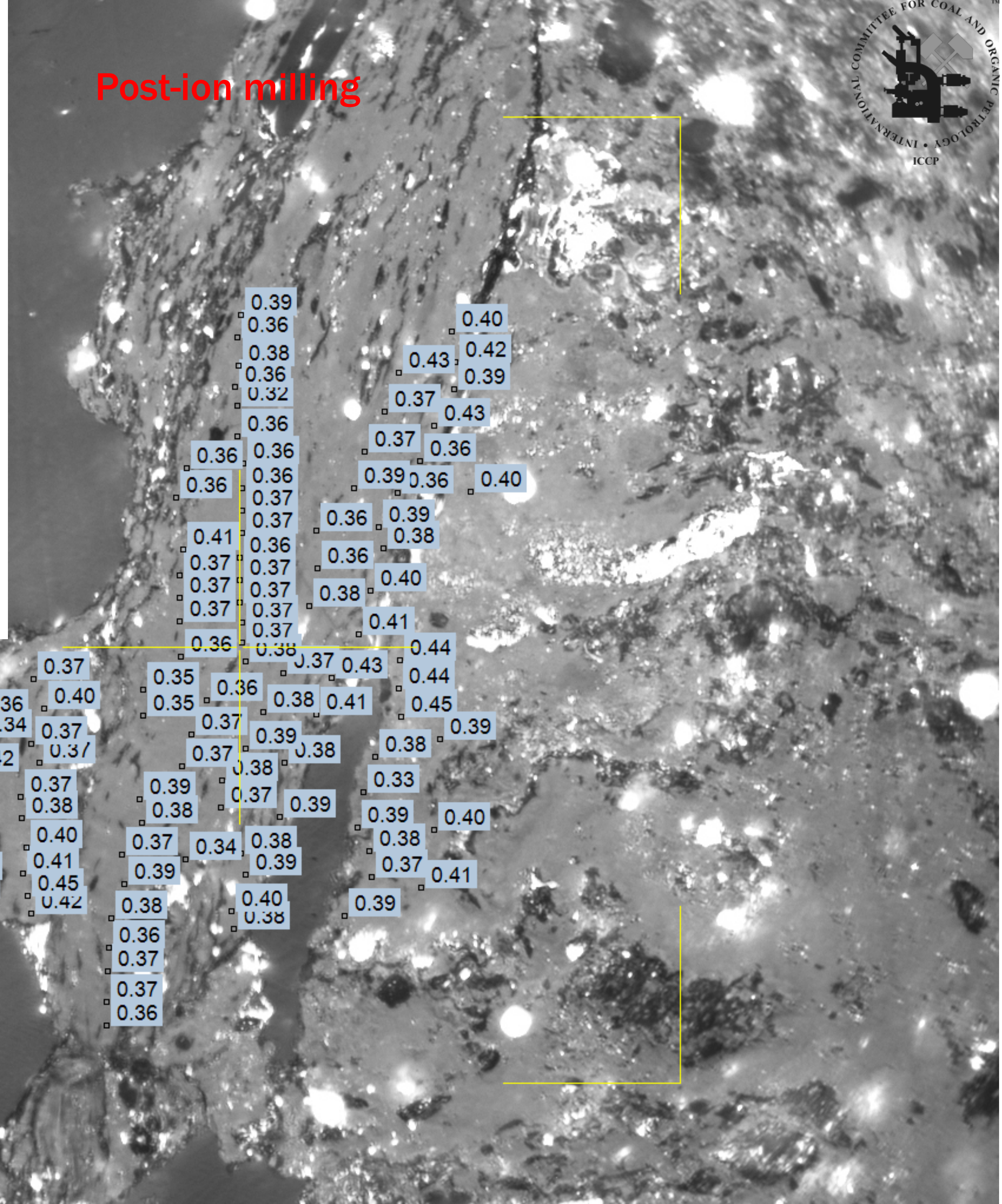
Measure count = 100
Reflectance Rr = 0.381 %
s = 0.025 %

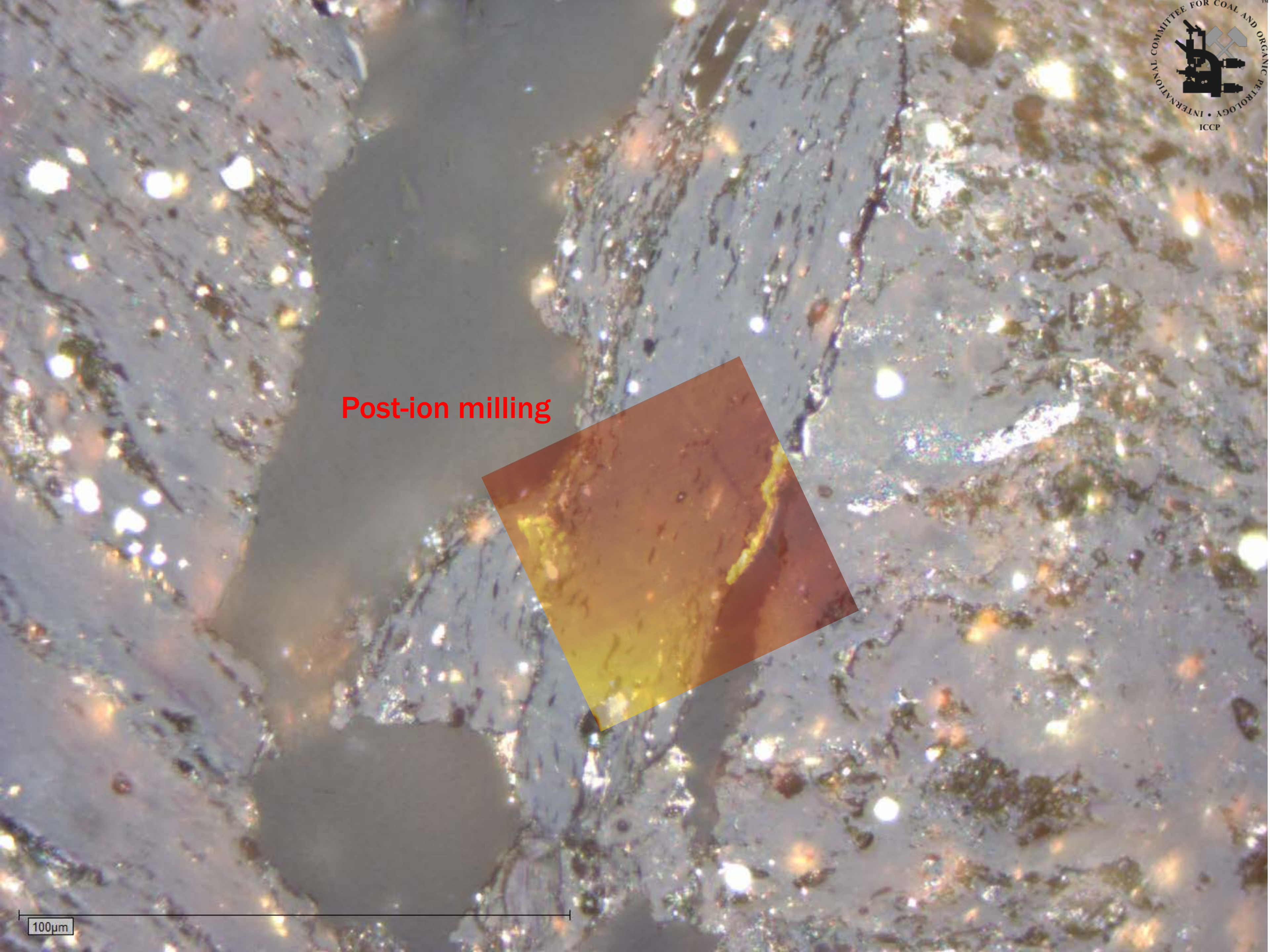
0.30 - 0.35 % R 6.0 %
0.35 - 0.40 % R 74.0 %

F=5.0.6143

Date 10/31/2017 1:49 PM
Operator Javin
Printed 10/31/2017

0.40 - 0.45 % R 20.0 %




Post-ion milling
100µm
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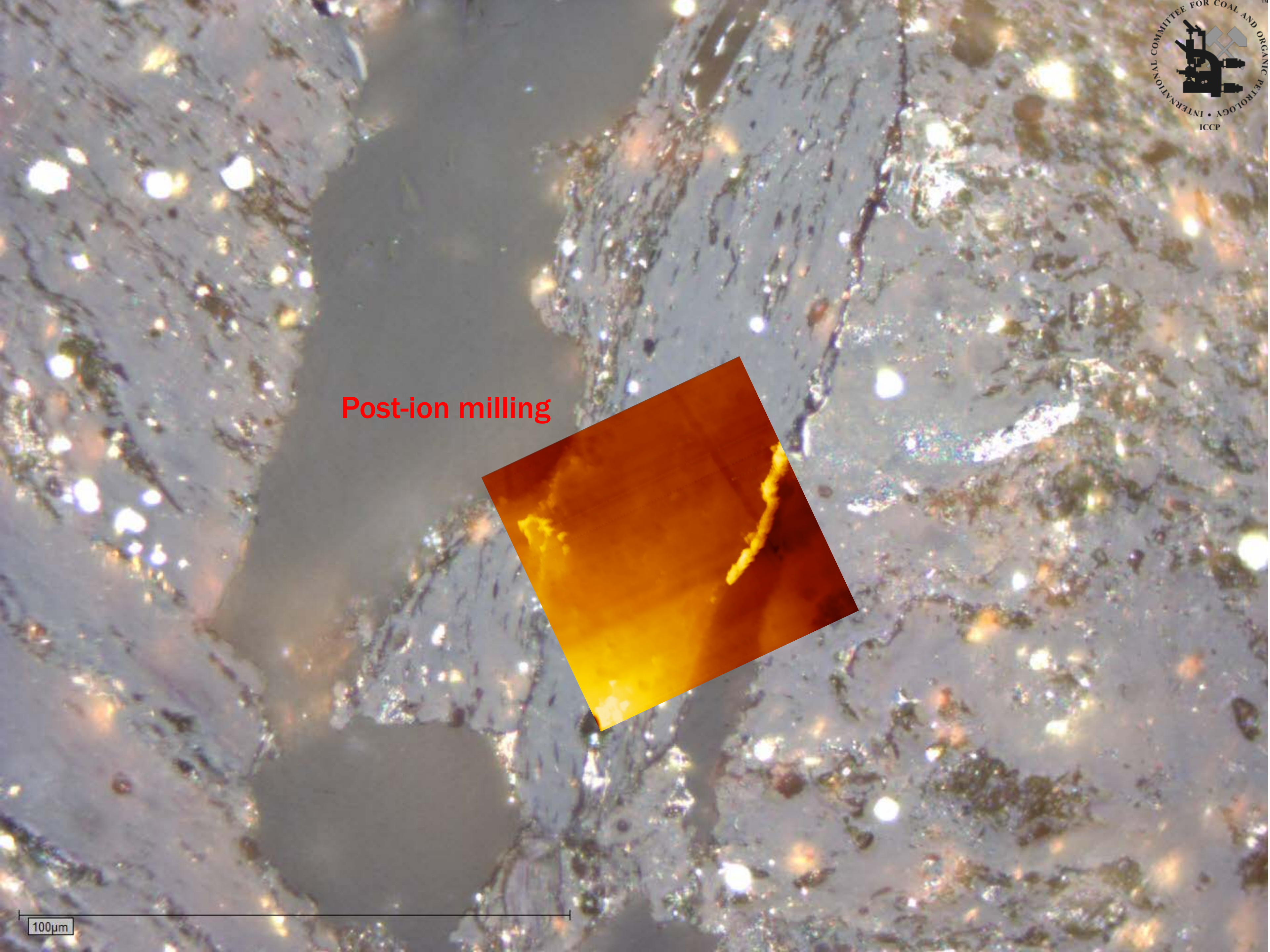
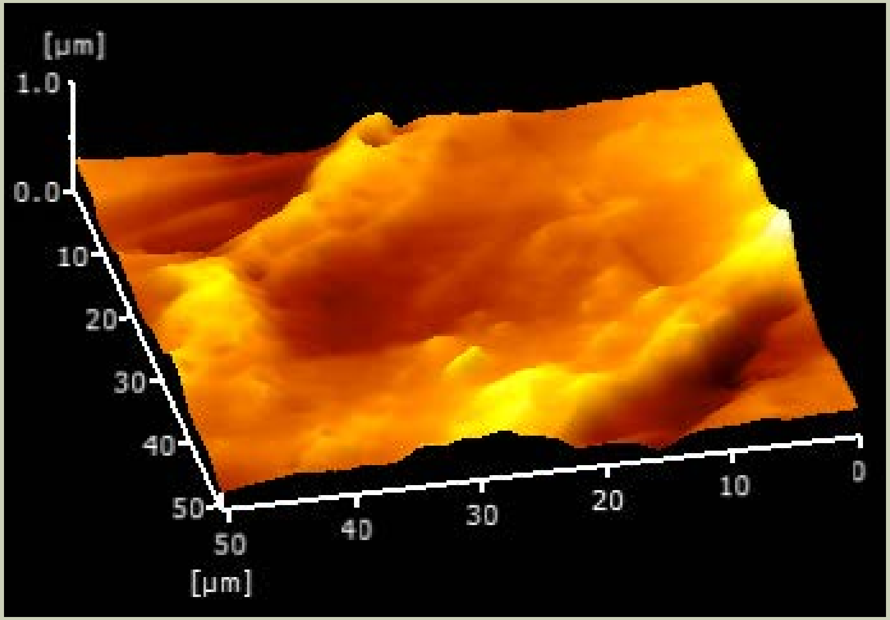
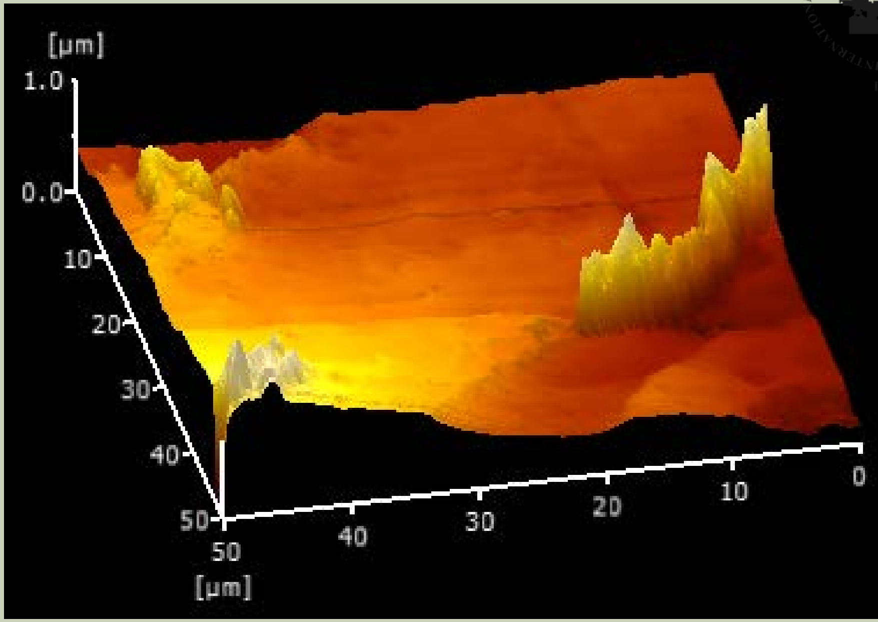
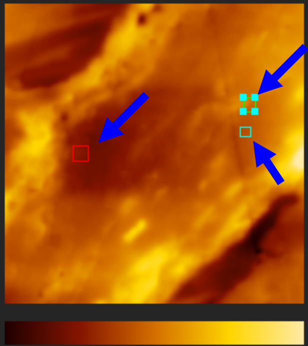

Post-ion milling
100µm

Pre-ion milling



Post-ion milling

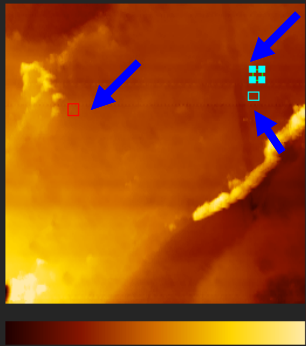




	Unit	Whole	1CH	2CH	3CH
Sa	nm	6.388E+01	9.687E+00	4.530E+00	1.477E+01
Sz/Smax	nm	6.470E+02	4.736E+01	2.188E+01	6.587E+01
Sp	nm	3.928E+02	2.027E+01	1.121E+01	4.025E+01
Sv	nm	2.542E+02	2.709E+01	1.067E+01	2.562E+01
Sq/RMS	nm	8.154E+01	1.100E+01	5.350E+00	1.782E+01
Δa	°	1.357E-01	9.670E-02	3.983E-02	7.741E-02
SzJIS/Sz	nm	2.946E+02	4.736E+01	2.188E+01	6.587E+01
S	nm²	2.565E+09	6.627E+06	2.823E+06	4.707E+06
Sratio		1.001E+00	1.000E+00	1.000E+00	1.001E+00
λc	nm	D.C	D.C	D.C	D.C

FileName 171030015.xqdx
Comm. 1 Kimmeridge area 1
Comm. 2 pre-ion mill
Comm. 3

Avg. RMS 11.39 nm

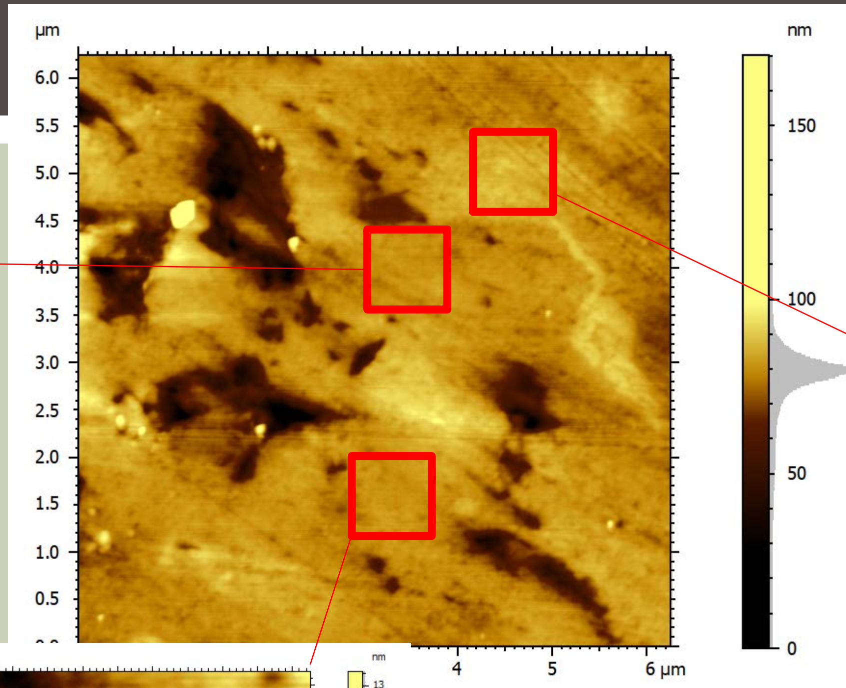


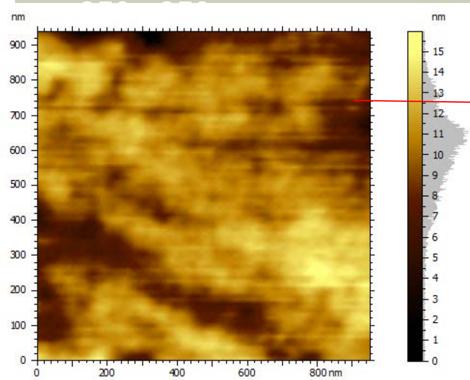
	Unit	Whole	1CH	2CH	3CH
Sa	nm	1.486E+02	6.301E+00	2.785E+00	4.894E+00
Sz/Smax	nm	1.592E+03	6.182E+01	1.589E+01	2.883E+01
Sp	nm	1.101E+03	2.685E+01	8.356E+00	1.558E+01
Sv	nm	4.911E+02	3.498E+01	7.530E+00	1.324E+01
Sq/RMS	nm	1.888E+02	8.298E+00	3.289E+00	6.316E+00
Δa	°	8.481E+00	2.156E-01	3.680E-02	6.124E-02
SzJIS/Sz	nm	5.931E+02	3.384E+01	1.589E+01	2.883E+01
S	nm²	2.547E+09	3.538E+06	2.425E+06	2.771E+06
Sratio		1.012E+00	1.022E+00	1.000E+00	1.000E+00
λc	nm	D.C	D.C	D.C	D.C

FileName 171030021.xqdx
Comm. 1 Kimmeridge area 1
Comm. 2 post-ion mill
Comm. 3

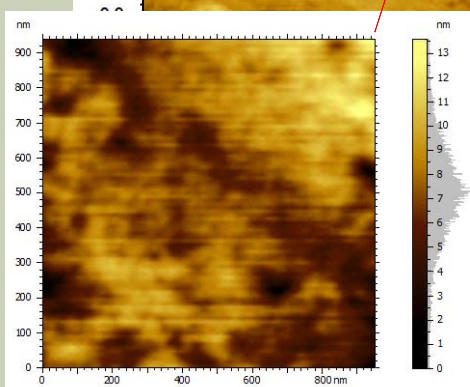
Avg. RMS 5.97 nm

PRE-ION MILLING

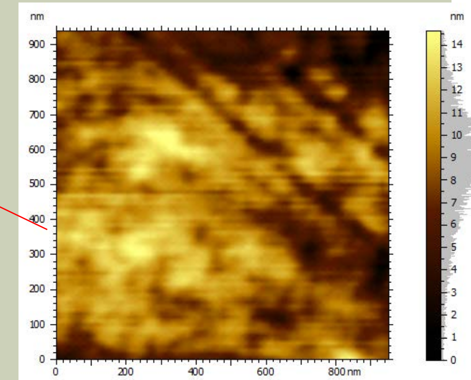




ISO 25178			
Height Parameters			
Sq	1.95	nm	*Root-mean-square height*
Ssk	-0.635		*Skewness*
Sku	4.40		*Kurtosis*
Sp	5.81	nm	*Maximumpeak height*
Sv	10.2	nm	*Maximumpit height*
Sz	16.0	nm	*Maximum height*
Sa	1.51	nm	*Arithmeticmeanheight*



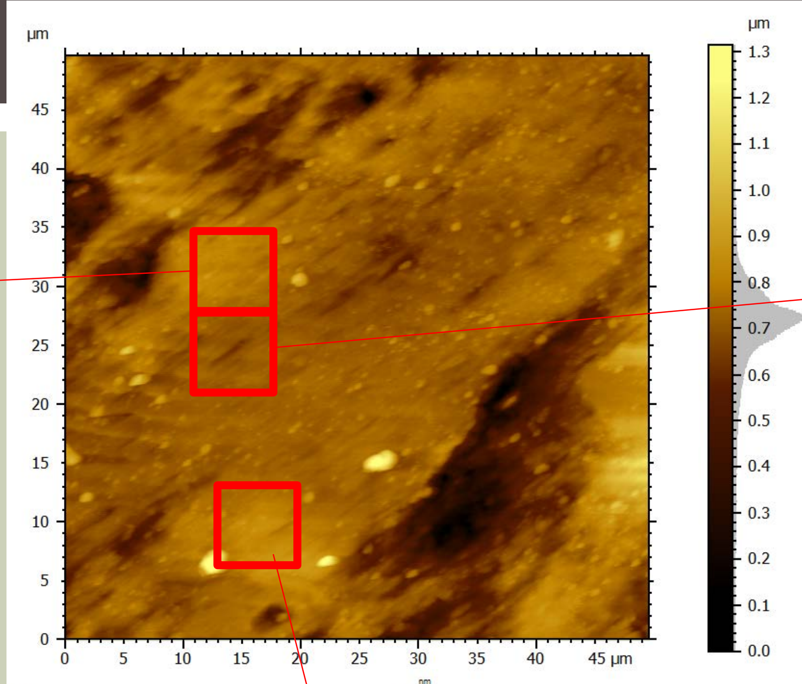
ISO 25178			
Height Parameters			
Sq	1.80	nm	*Root-mean-square height*
Ssk	-0.133		*Skewness*
Sku	3.41		*Kurtosis*
Sp	6.26	nm	*Maximumpeak height*
Sv	7.32	nm	*Maximumpit height*
Sz	13.6	nm	*Maximum height*
Sa	1.40	nm	*Arithmeticmeanheight*

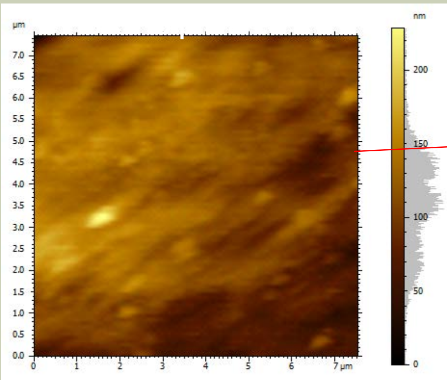


ISO 25178			
Height Parameters			
Sq	2.44	nm	*Root-mean-square height*
Ssk	-0.426		*Skewness*
Sku	3.08		*Kurtosis*
Sp	6.09	nm	*Maximumpeak height*
Sv	8.54	nm	*Maximumpit height*
Sz	14.6	nm	*Maximum height*
Sa	1.93	nm	*Arithmeticmeanheight*

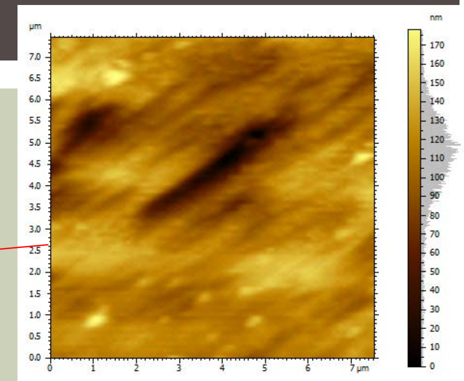
Courtesy Keysight Tech.

POST-ION MILLING

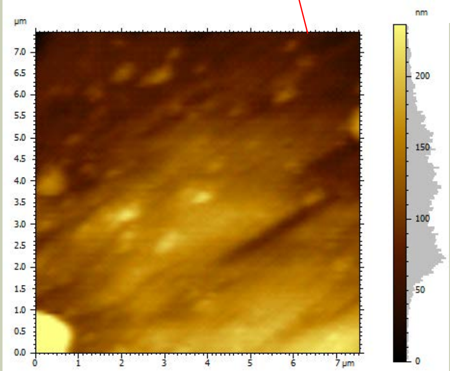




ISO 25178			
Height Parameters			
Sq	29.8	nm	*Root-mean-square height*
Ssk	-0.213		*Skewness*
Sku	2.64		*Kurtosis*
Sp	118	nm	*Maximum peak height*
Sv	110	nm	*Maximum pit height*
Sz	228	nm	*Maximum height*
Sa	24.2	nm	*Arithmetic mean height*



ISO 25178			
Height Parameters			
Sq	23.3	nm	*Root-mean-square height*
Ssk	-1.05		*Skewness*
Sku	5.56		*Kurtosis*
Sp	68.6	nm	*Maximum peak height*
Sv	109	nm	*Maximum pit height*
Sz	178	nm	*Maximum height*
Sa	17.1	nm	*Arithmetic mean height*



ISO 25178			
Height Parameters			
Sq	51.3	nm	*Root-mean-square height*
Ssk	2.69		*Skewness*
Sku	19.5		*Kurtosis*
Sp	459	nm	*Maximum peak height*
Sv	118	nm	*Maximum pit height*
Sz	577	nm	*Maximum height*
Sa	36.4	nm	*Arithmetic mean height*

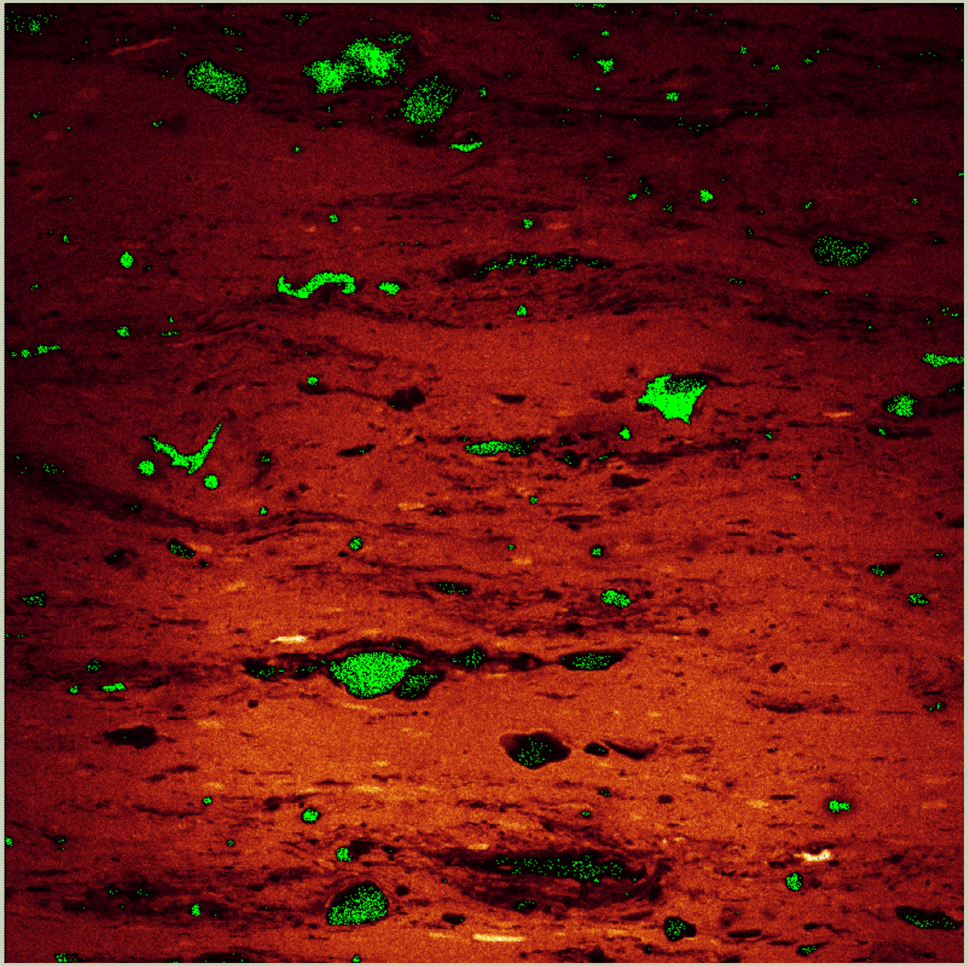
Courtesy Keysight Tech.

Not on correlative areas – exposure of nano-sulfides causes increase in RMS

CLSM WG RESULTS 2018

Contributors to-date:

- Paul Hackley (1/2 result)
- Jolanta Kus, BGR
- Andy Czaja, Univ. Cincinnati
- Isabel Suarez-Ruiz (hint of result), INCAR



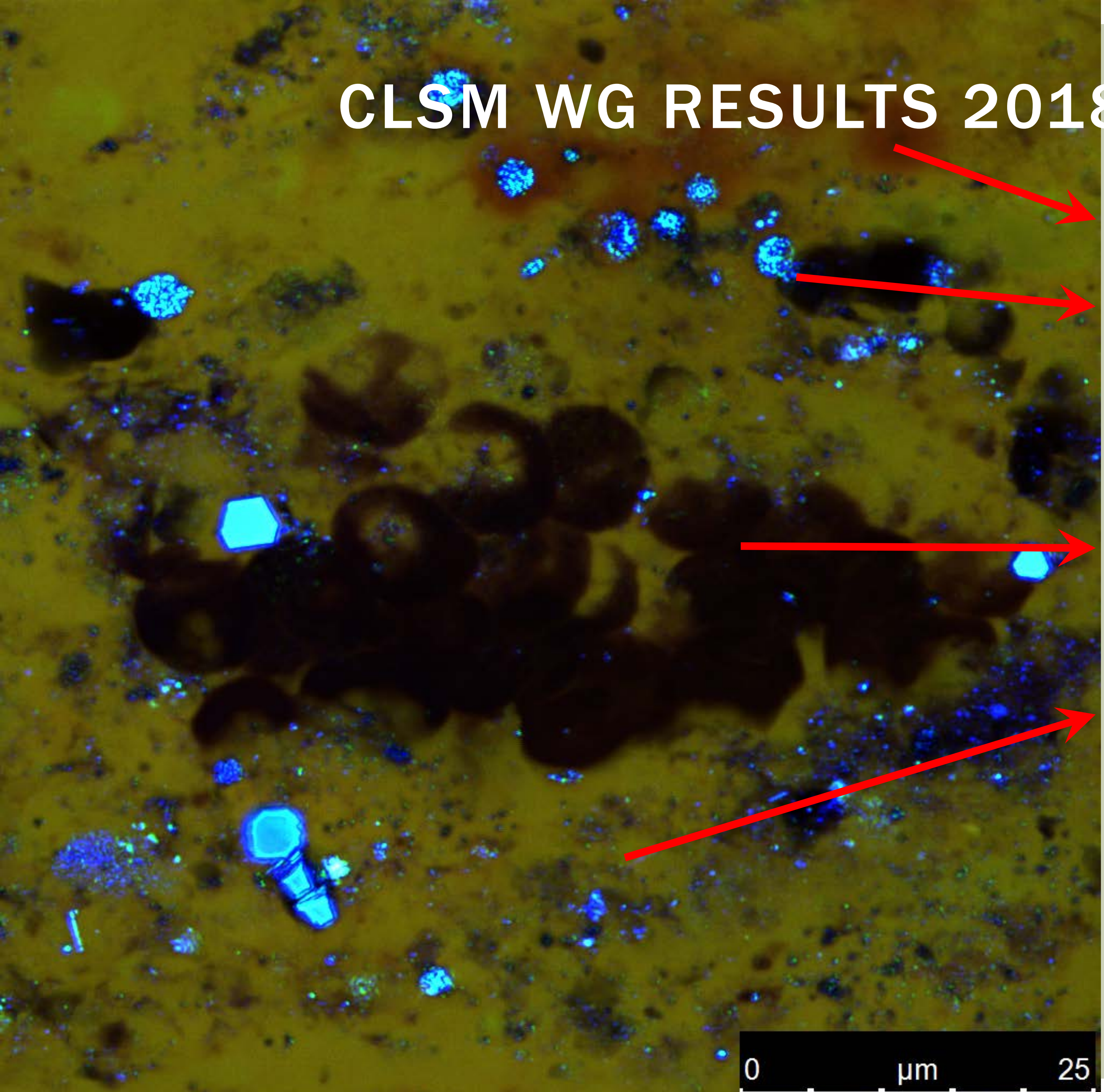
CLSM WG RESULTS 2018

Kimmeridge Clay: KC-1

- Image stack (128)
- 141 x 141 x 11 µm
- 405 nm diode laser
- 470 nm HyD detection
- 63x oil, 1.4NA

- Image shows highest intensity response in volume

CLSM WG RESULTS 2018



Bituminite with halos

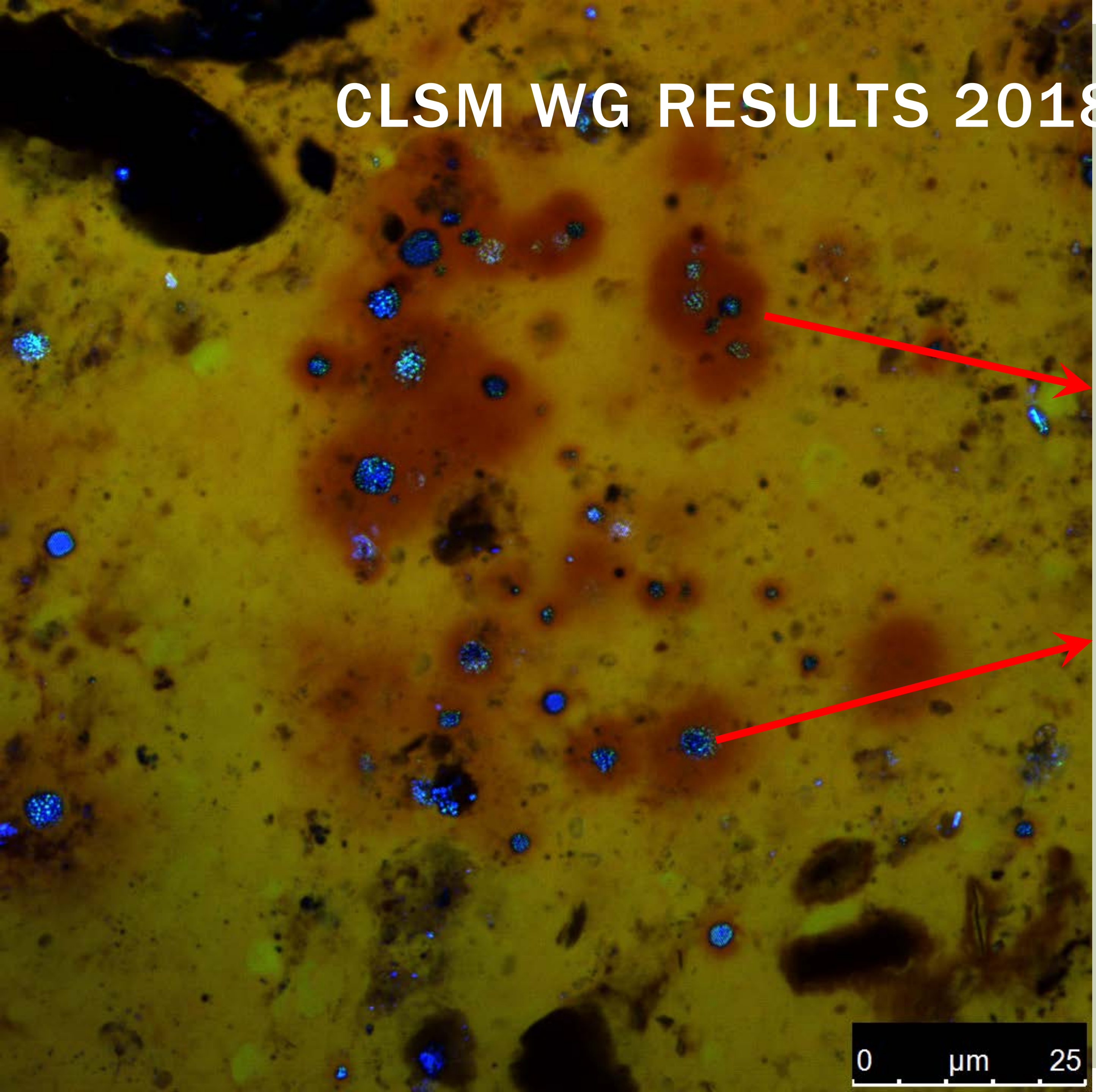
Framboidal pyrite

Coccoliths

Bituminite, pitted appearance, due to pyrite

Jolanta Kus

CLSM WG RESULTS 2018

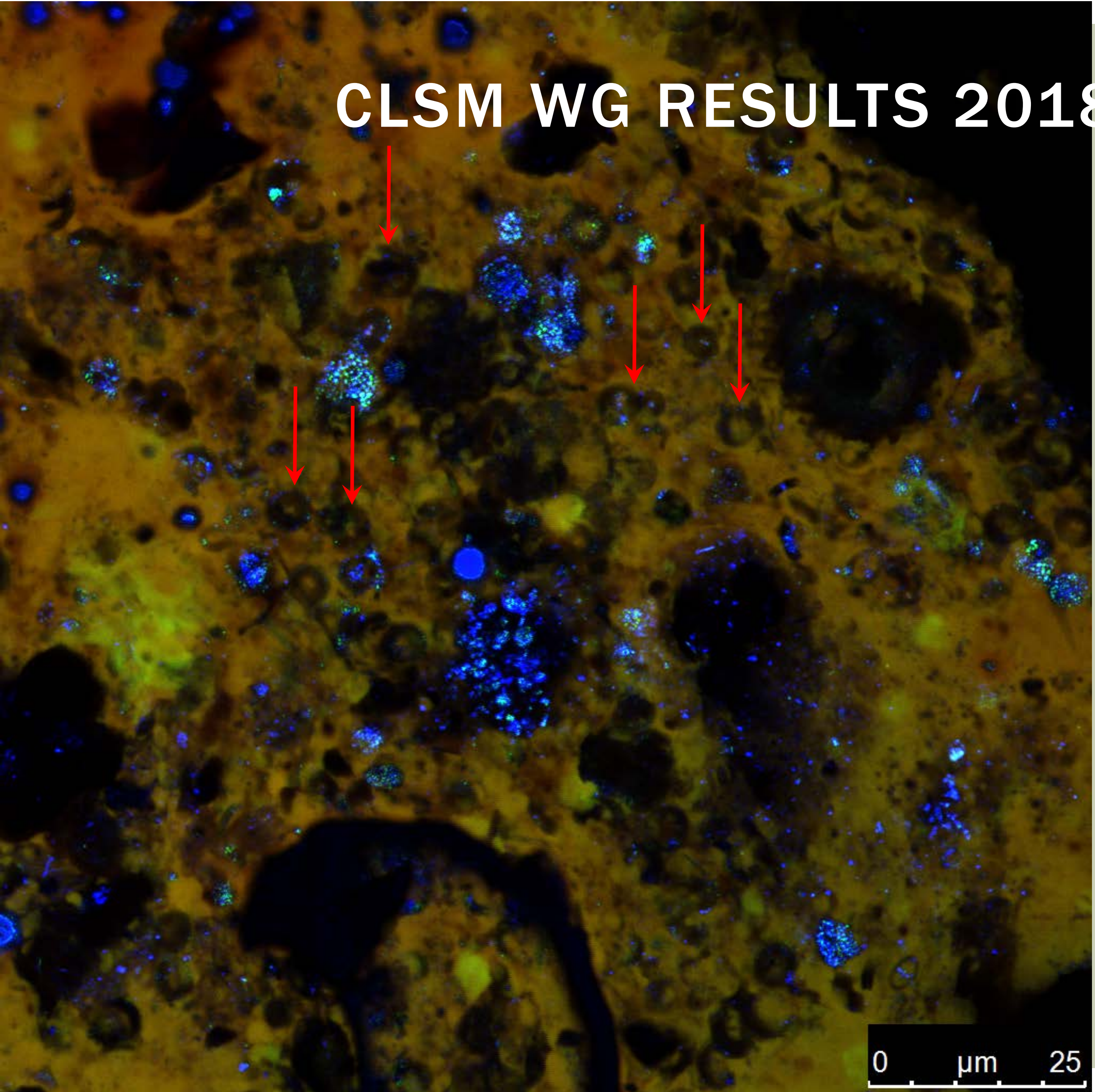


Bituminite with halos

Framboidal pyrite

Jolanta Kus

CLSM WG RESULTS 2018



Cocoliths, replacement with framboidal pyrite (red arrows)

Framboidal pyrite (blue colour)

Jolanta Kus

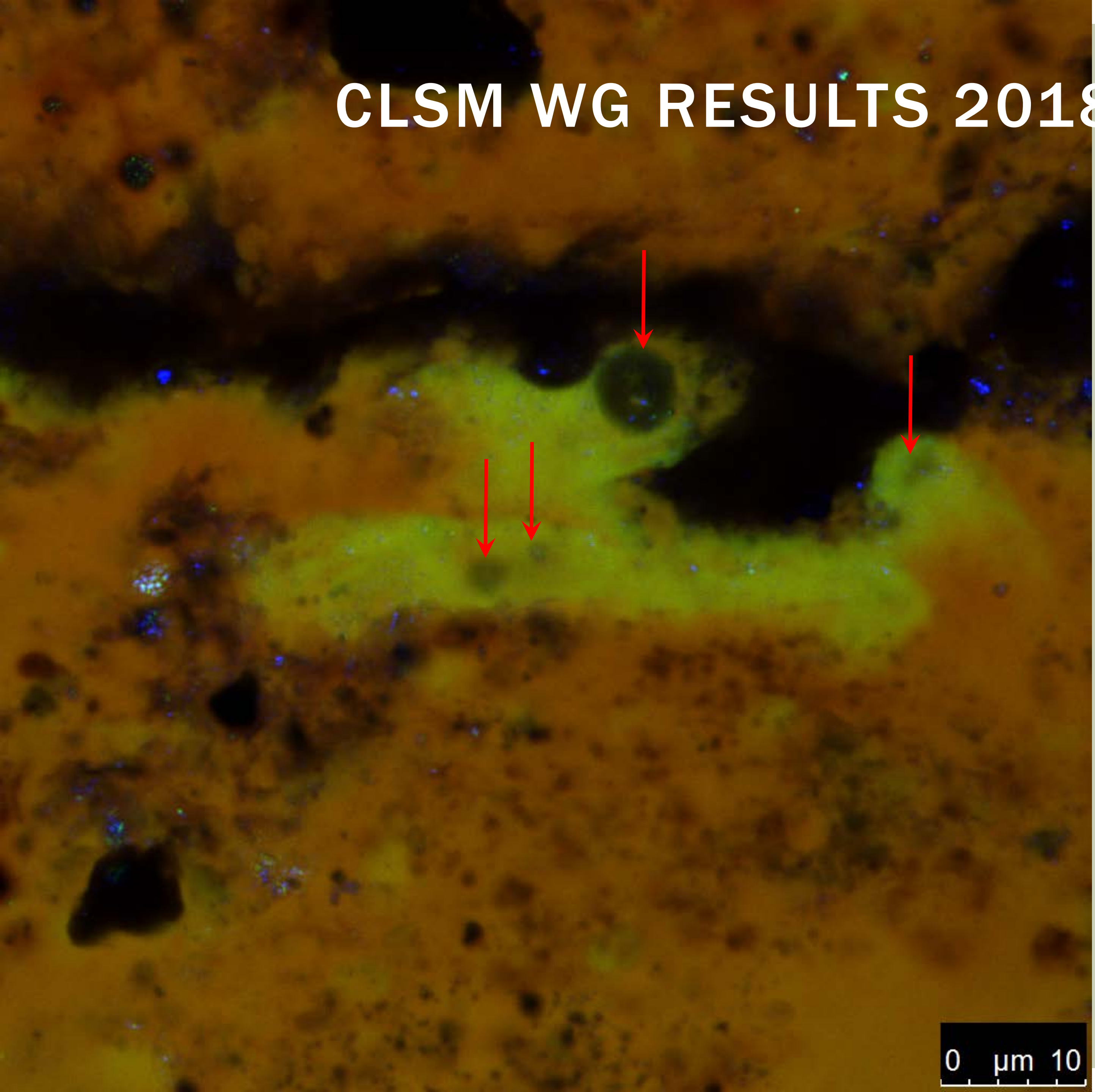
CLSM WG RESULTS 2018

Botryococcus: Branch with four openings (red arrows)

Framboidal pyrite (blue colour)

Jolanta Kus

CLSM WG RESULTS 2018



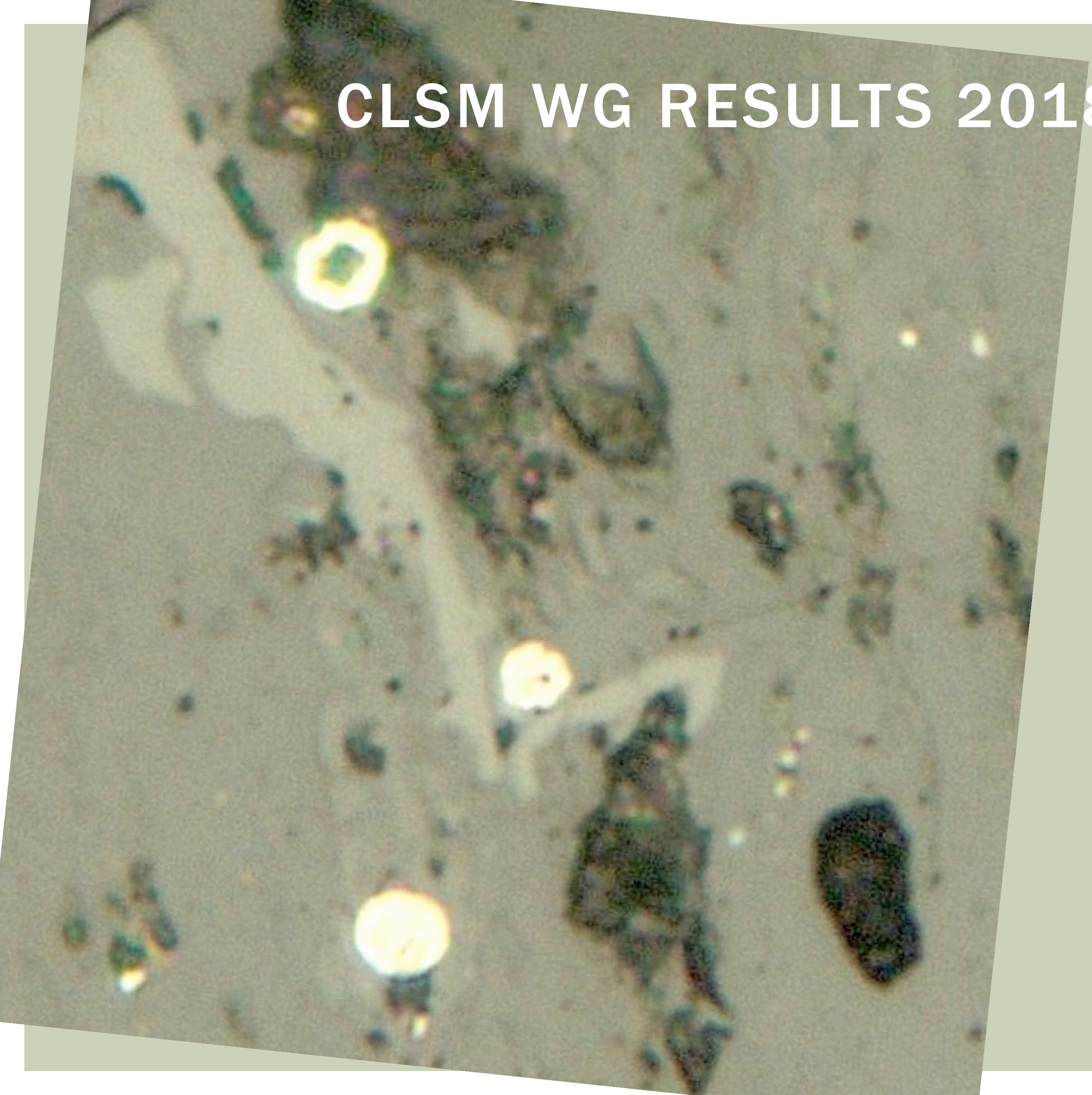
The same CLSM-image as previous slide but at a depth of 2-3 µm below surface of the pellet

Botryococcus: Branch with four openings (red arrows)

Framboidal pyrite (blue colour)

Jolanta Kus

CLSM WG RESULTS 2018

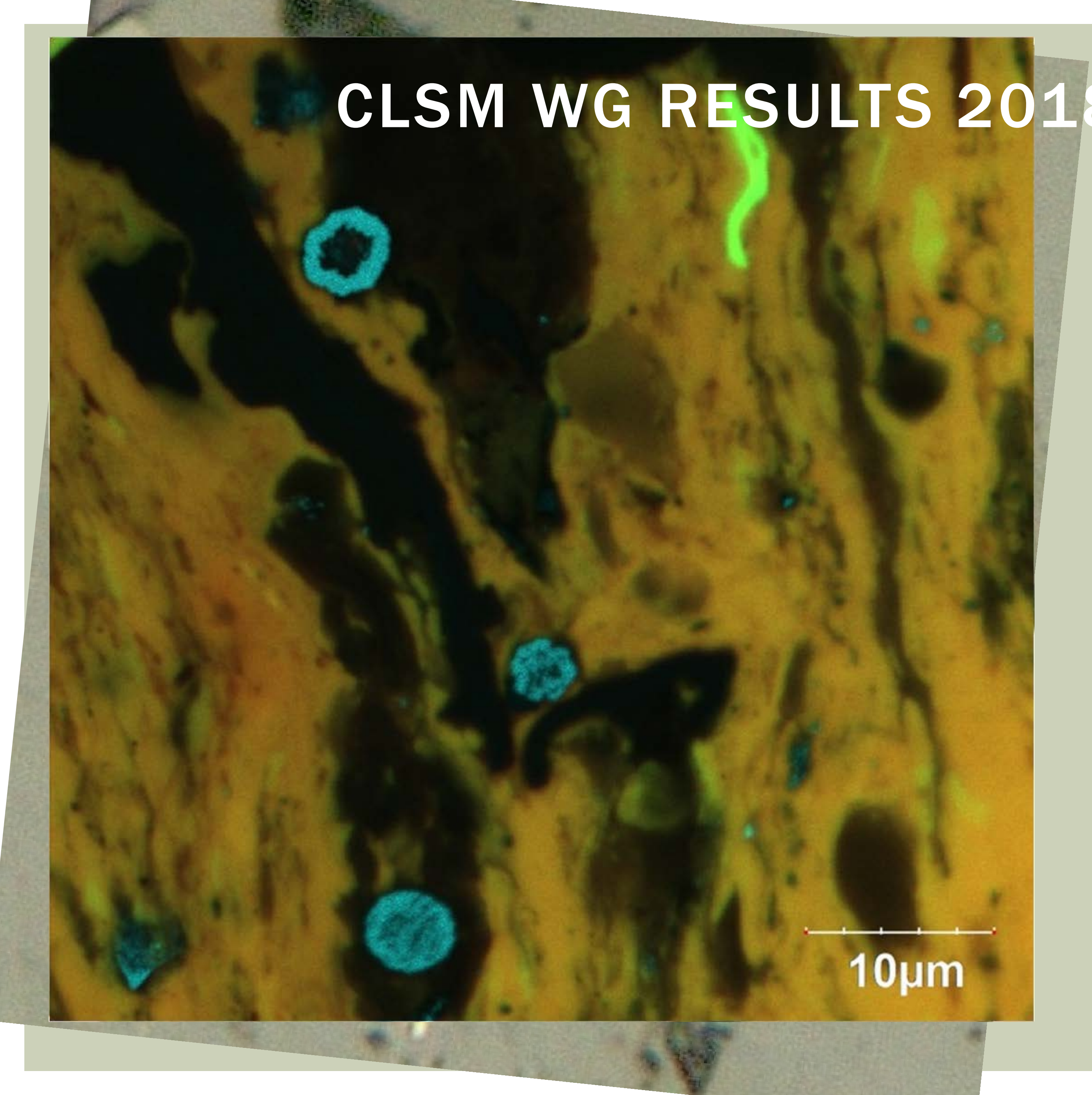


Andy Czaja

CLSM WG RESULTS 2018

Andy Czaja

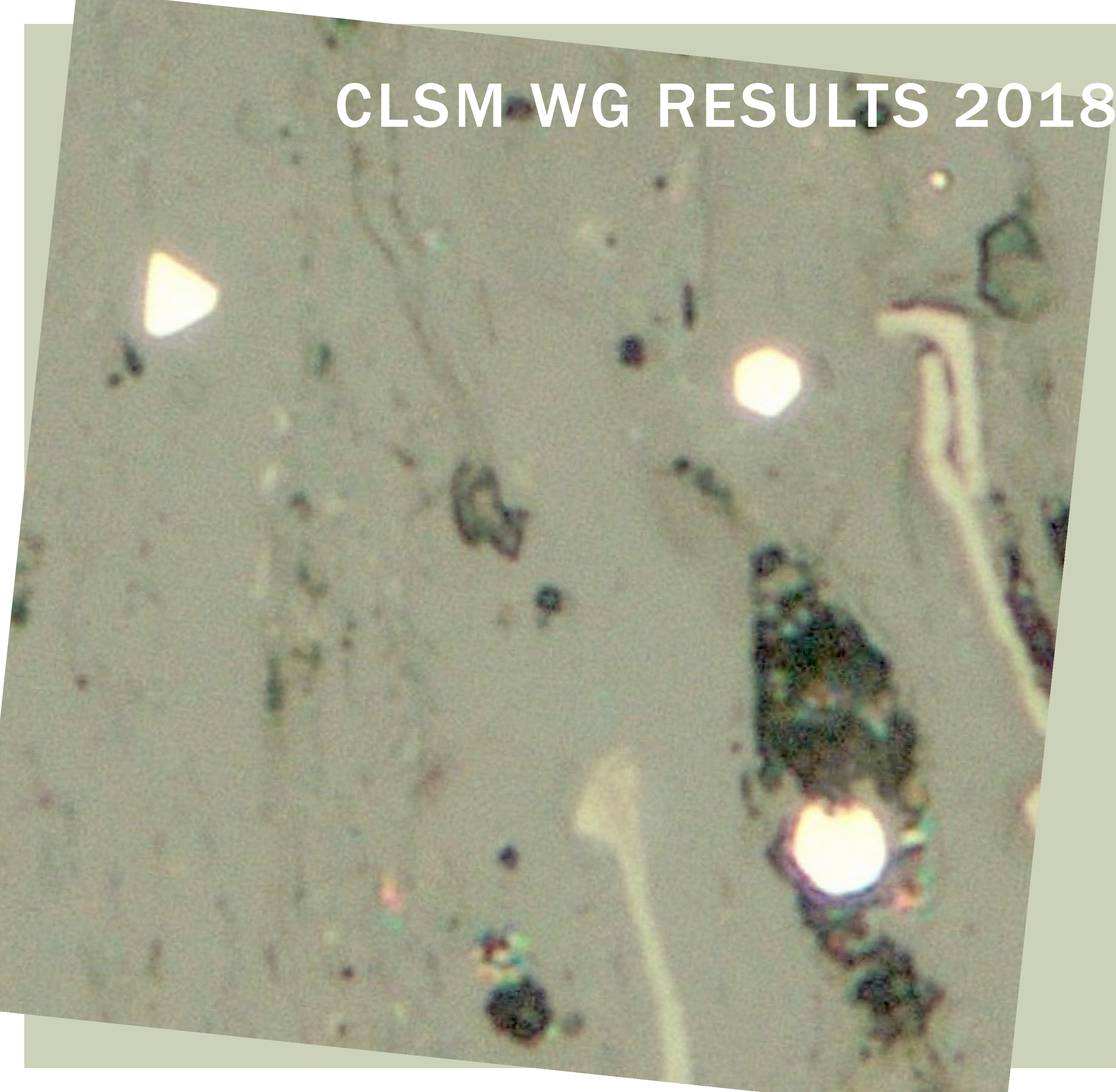
CLSM WG RESULTS 2018



Pyrite fluorescence imaged with 458 nm excitation, 480-495 bandpass

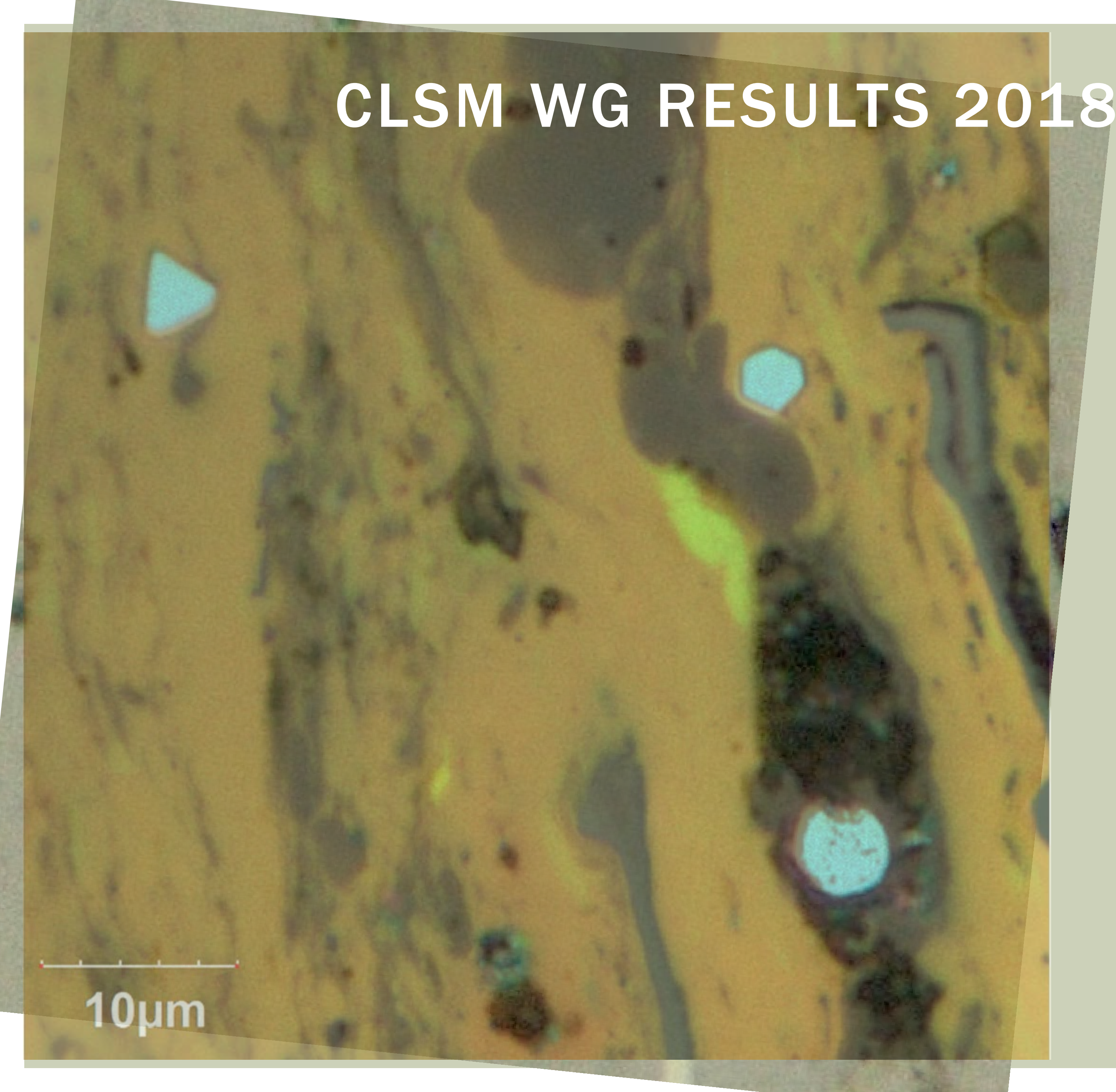
Andy Czaja

CLSM WG RESULTS 2018



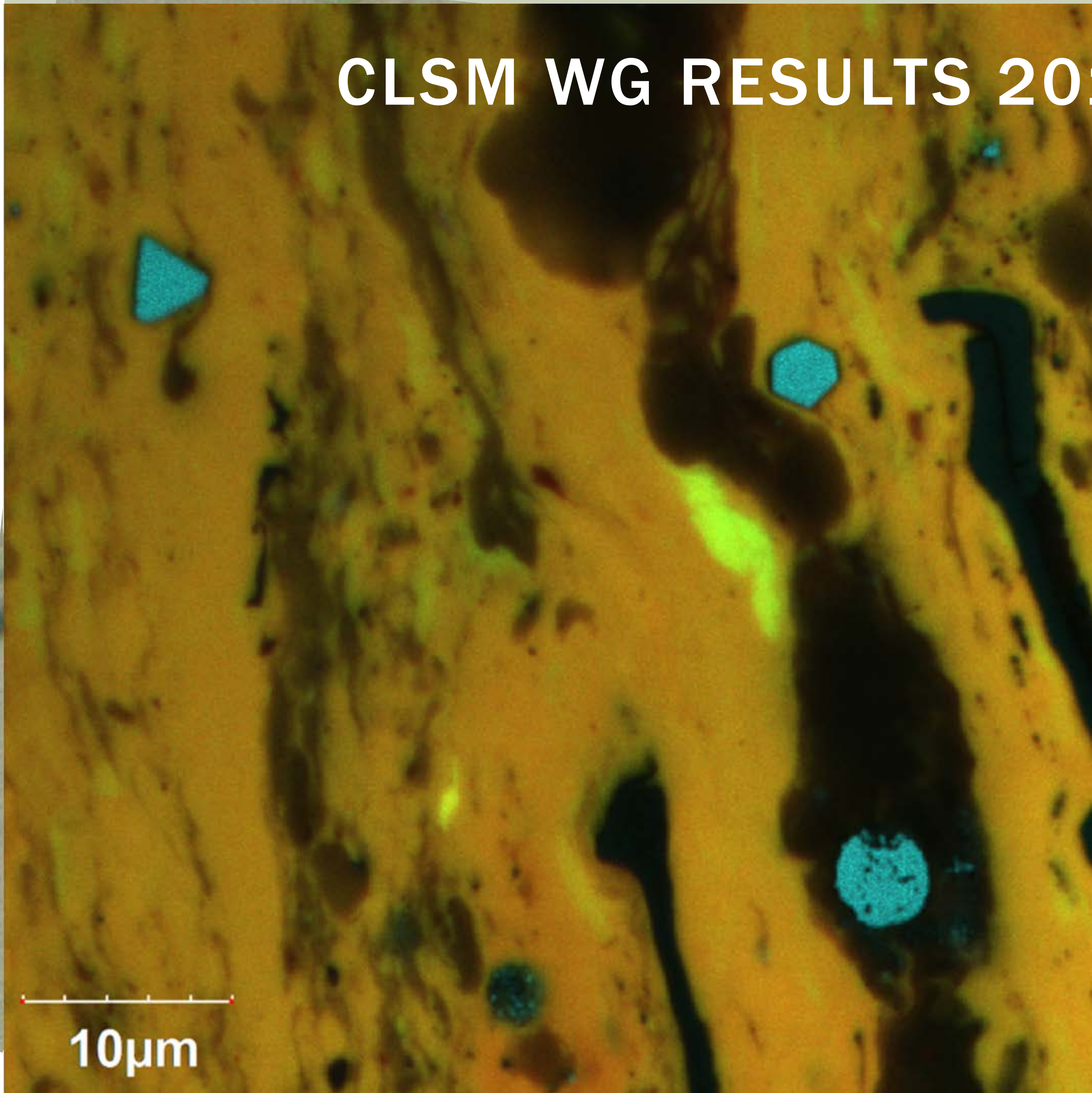
Andy Czaja

CLSM WG RESULTS 2018



Andy Czaja

CLSM WG RESULTS 2018



Andy Czaja

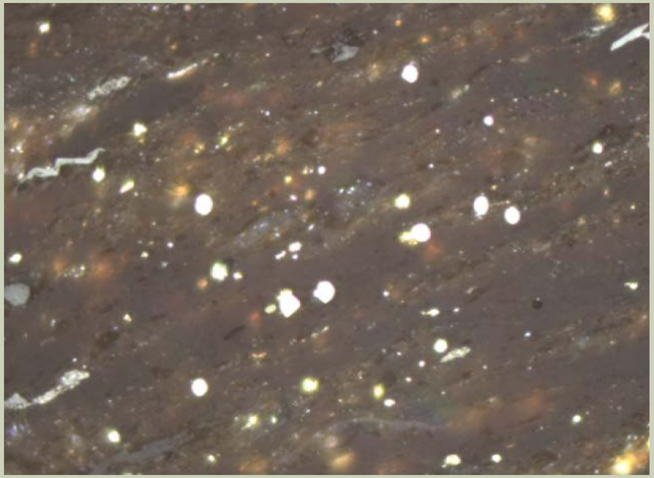
CLSM WG 2018 SUMMARY

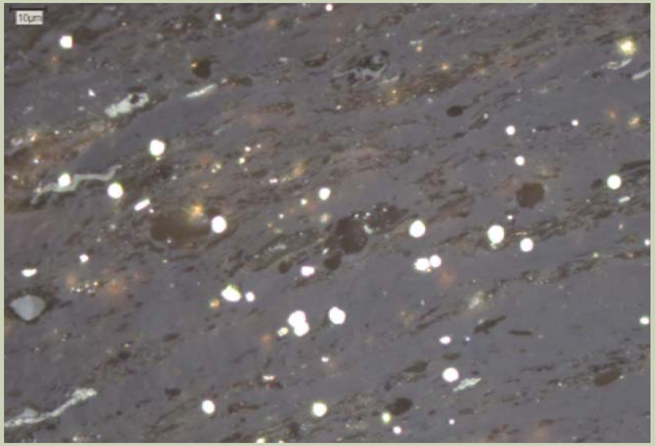
INTERNATIONAL COMMITTEE FOR COAL AND ORGANIC PETROLOGY • ICCP

Summary:

- Kimmeridge Clay sample selected
- Distributed to 13 persons
- Results received from 2.5 persons
- Ion milling causes Ro increase to AOM
- AFM suggests this is due to flattening

- 3-D rendering from image stacks
- Pyrite fluorescence(?)
- Botryococcus(?)
- Halos around pyrite framboids(?)
- Awaiting results from others






THANKS!