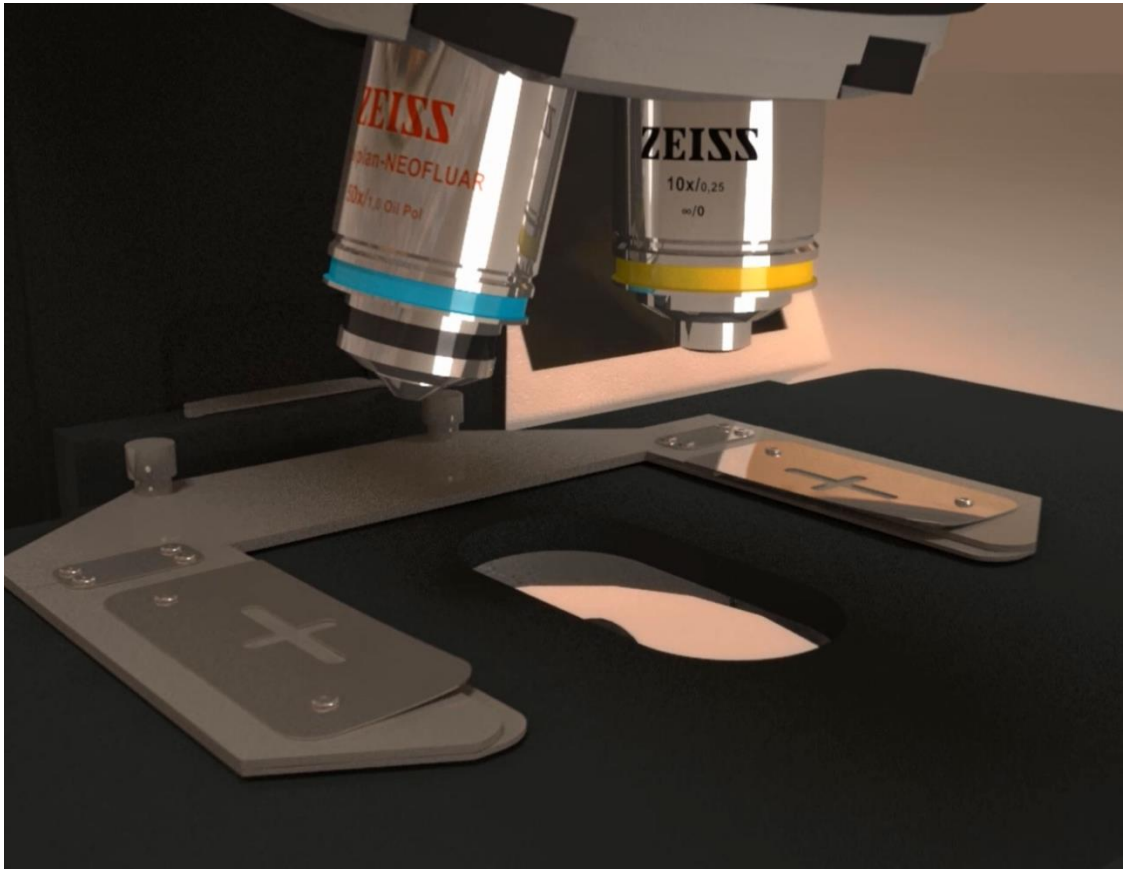
The End of Oil Immersion Microscopy?



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EngD Project Background

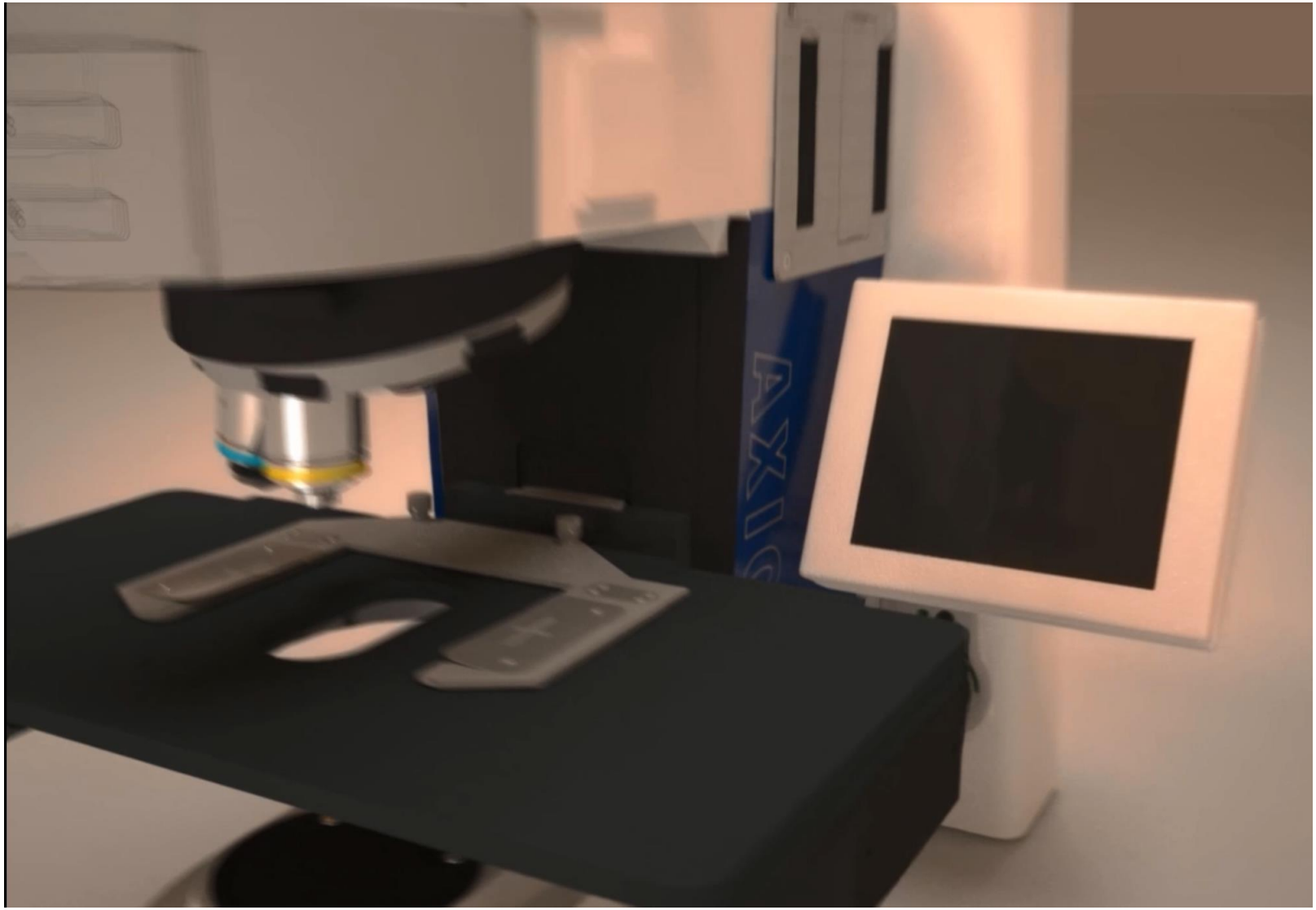
- MSci Chemistry - University of Nottingham
- October 2015 - EPSRC Centre for Doctoral Training in Carbon Capture and Storage and Cleaner Fossil Energy
- Using image analysis to predict how fuel types might perform in a pulverised fuel boiler. Burnout efficiency, slagging, fouling and NO_x /SO_x emissions.

Image Analysis

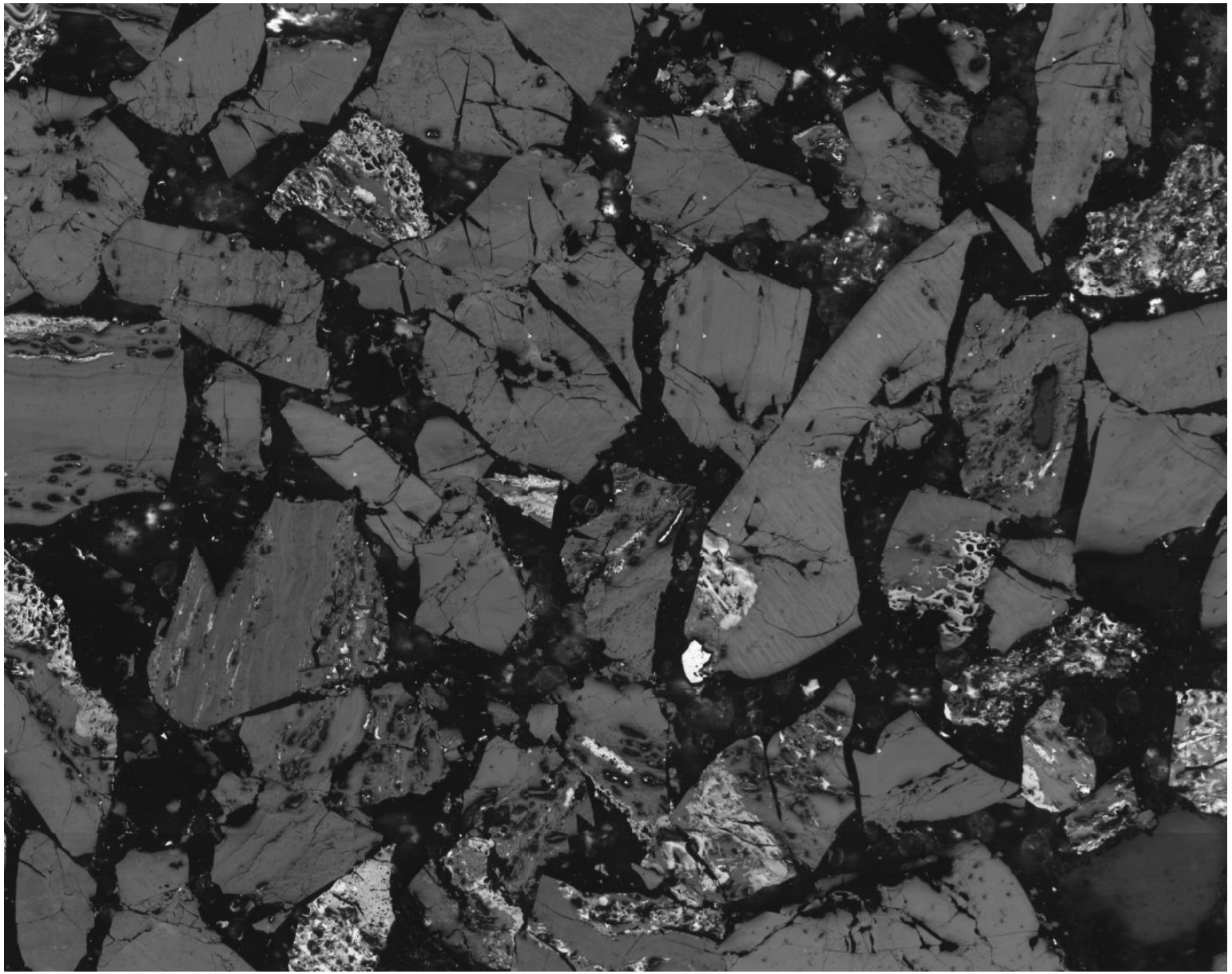
- Rapidly characterise fuels to predict boiler performance
- A fully automated tool for plant operators
- Blend or as a single fuel source
- Many behavioural aspects of coals can be ‘seen’ under an oil immersion microscope, there is no current technique for collating this information together.

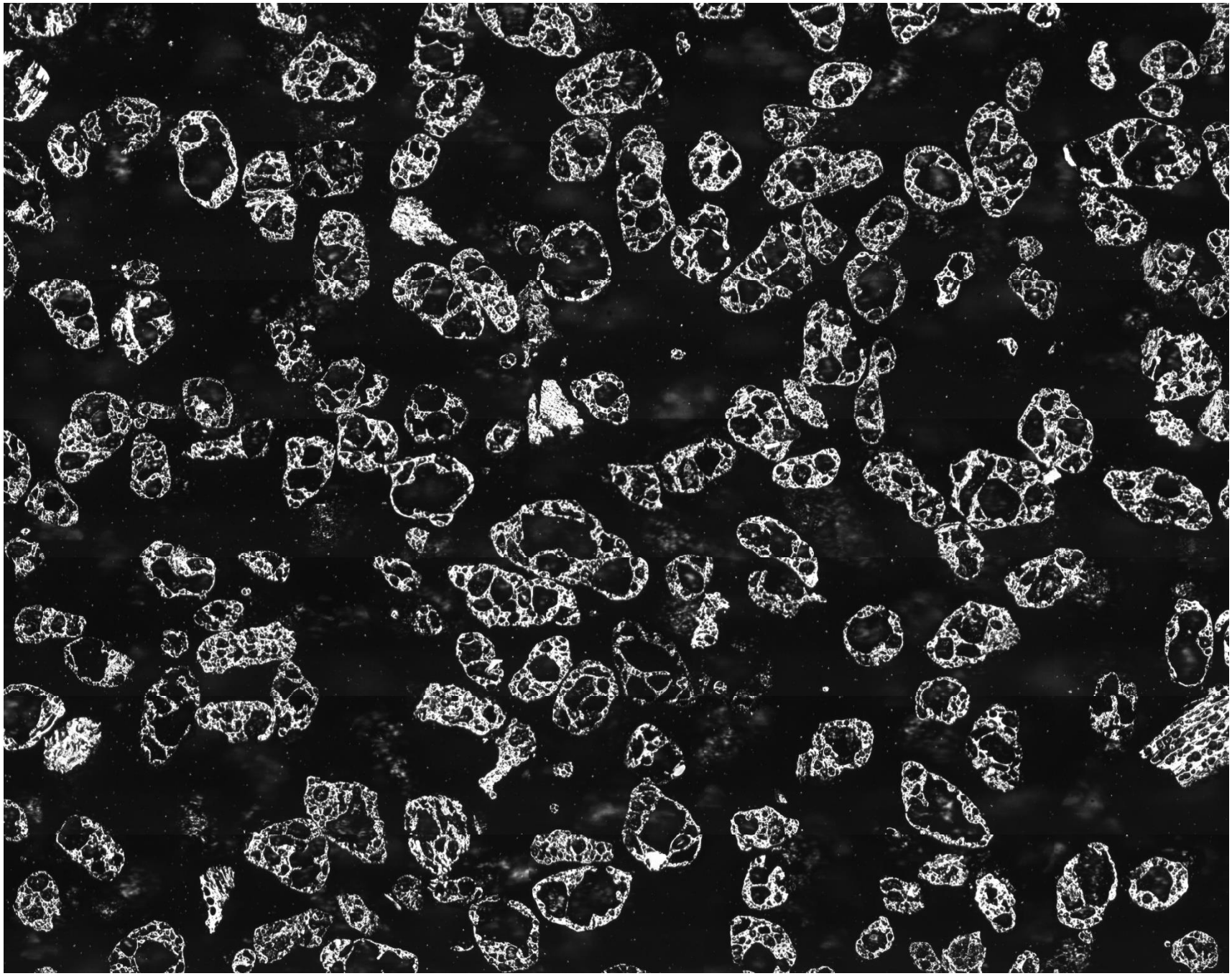
To develop several new image analysis methods to measure coal, char, mineral and ash materials resulting in a simple method that can characterise fuel in a way that enables power generators to understand the consequences of fuel choices.

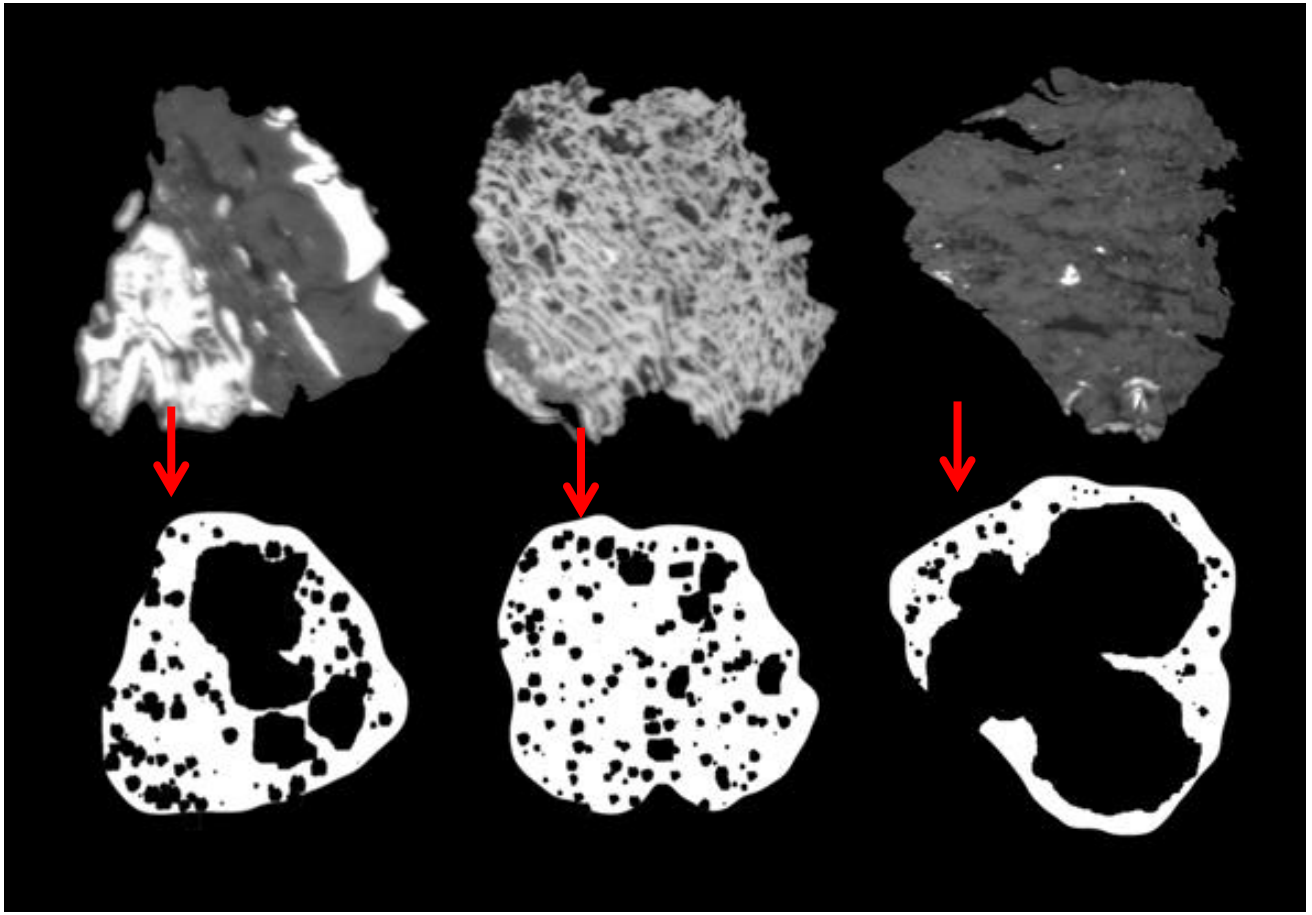
Oil Immersion Microscopy



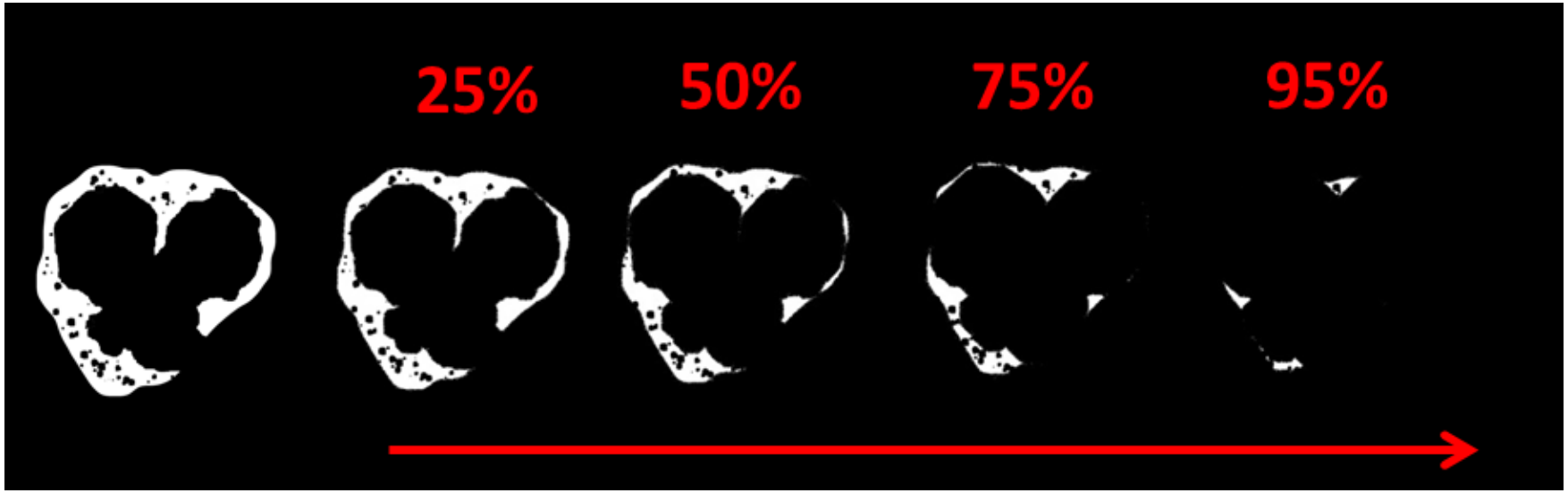
Zeiss_imaging_system.mp4



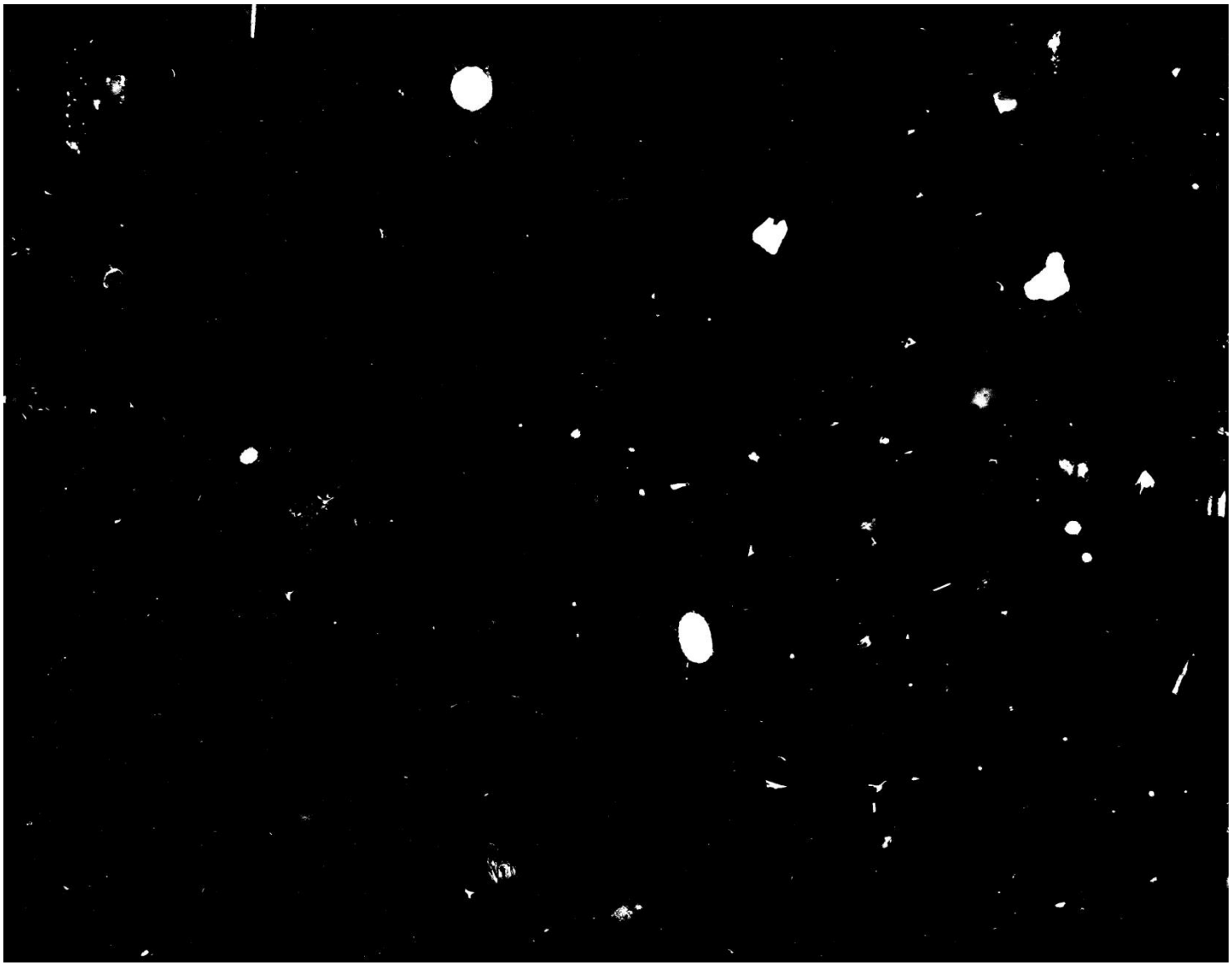




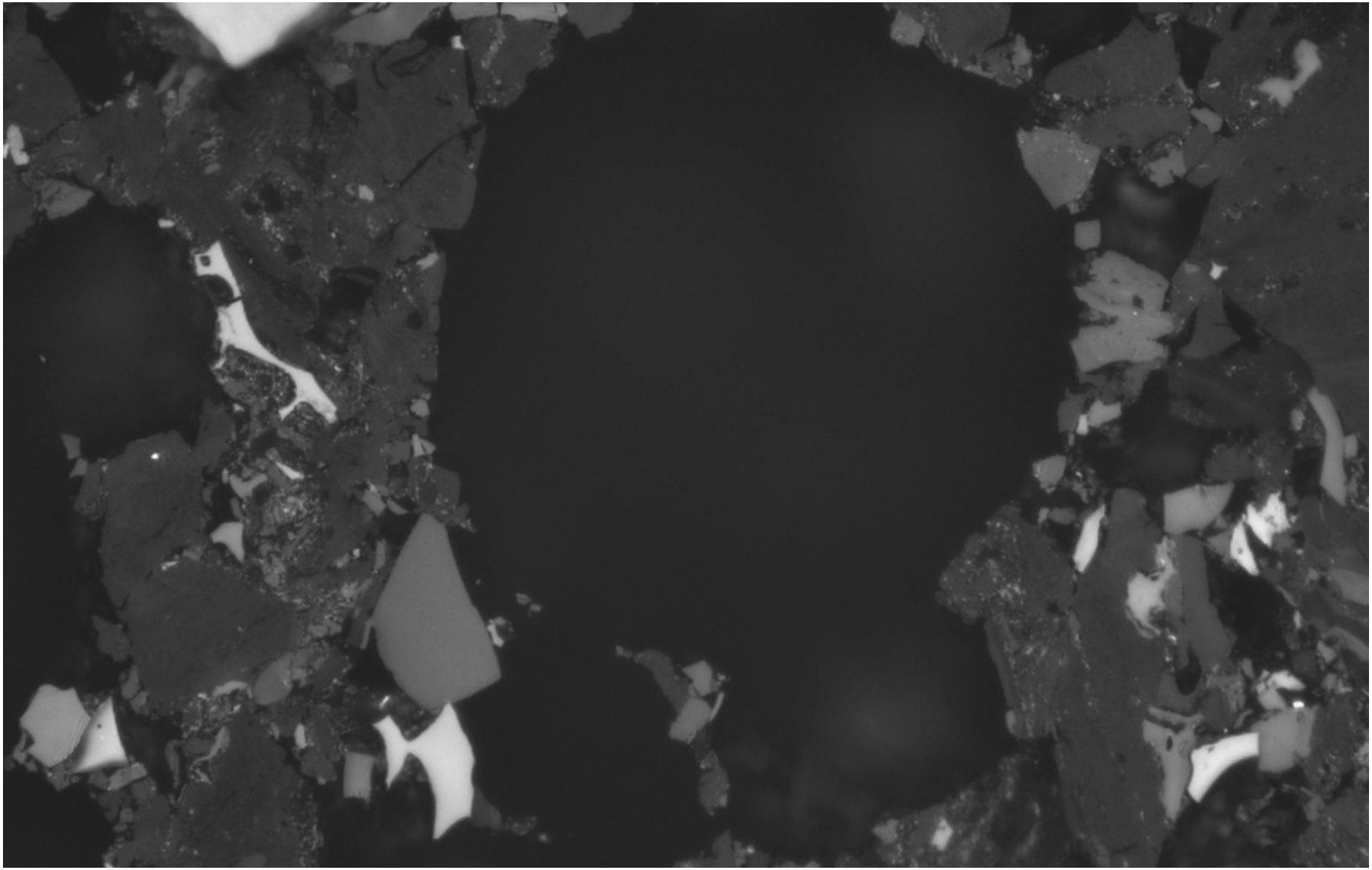
Kinetics Free Combustion Simulations…?



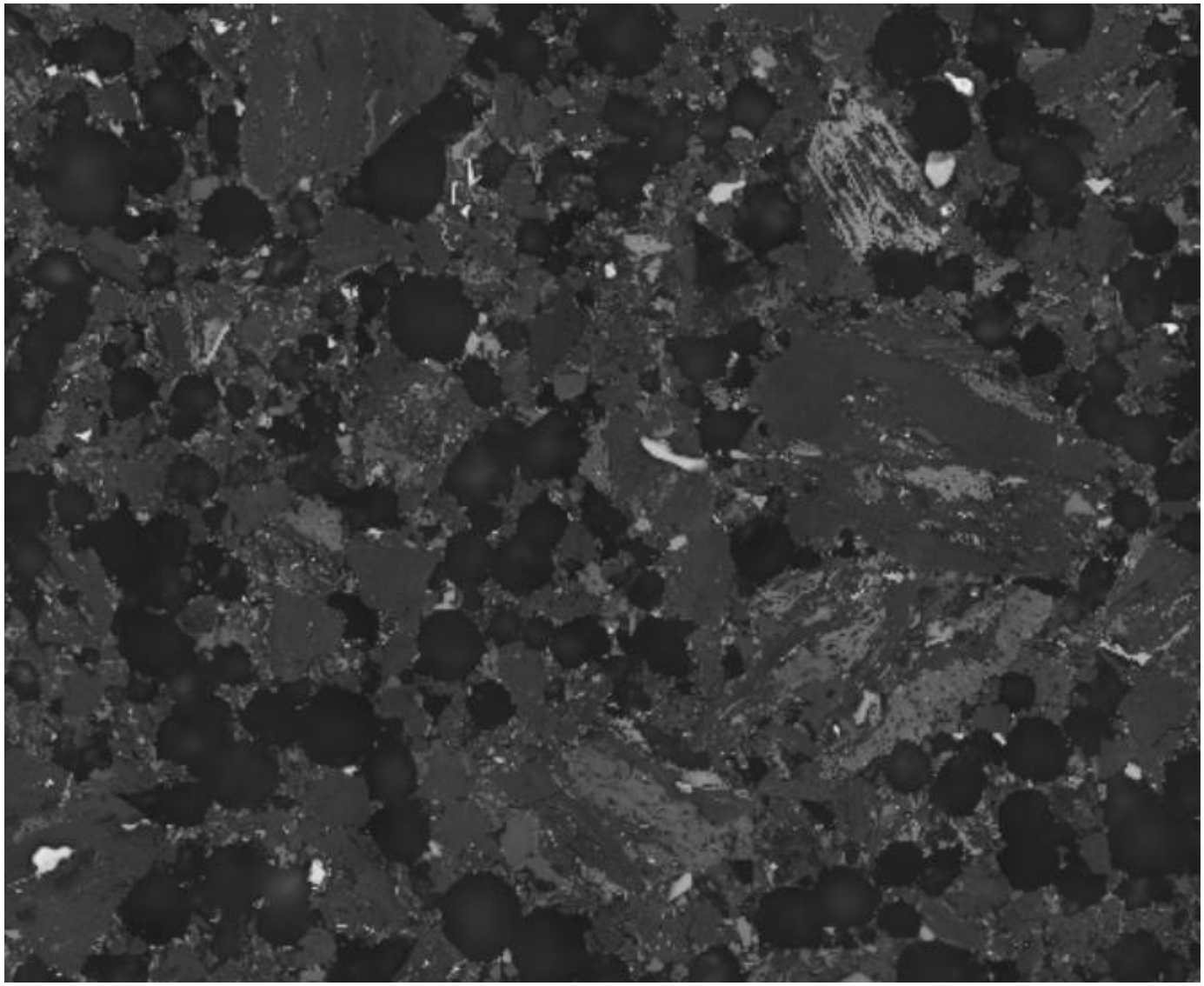
Pyrite Detection



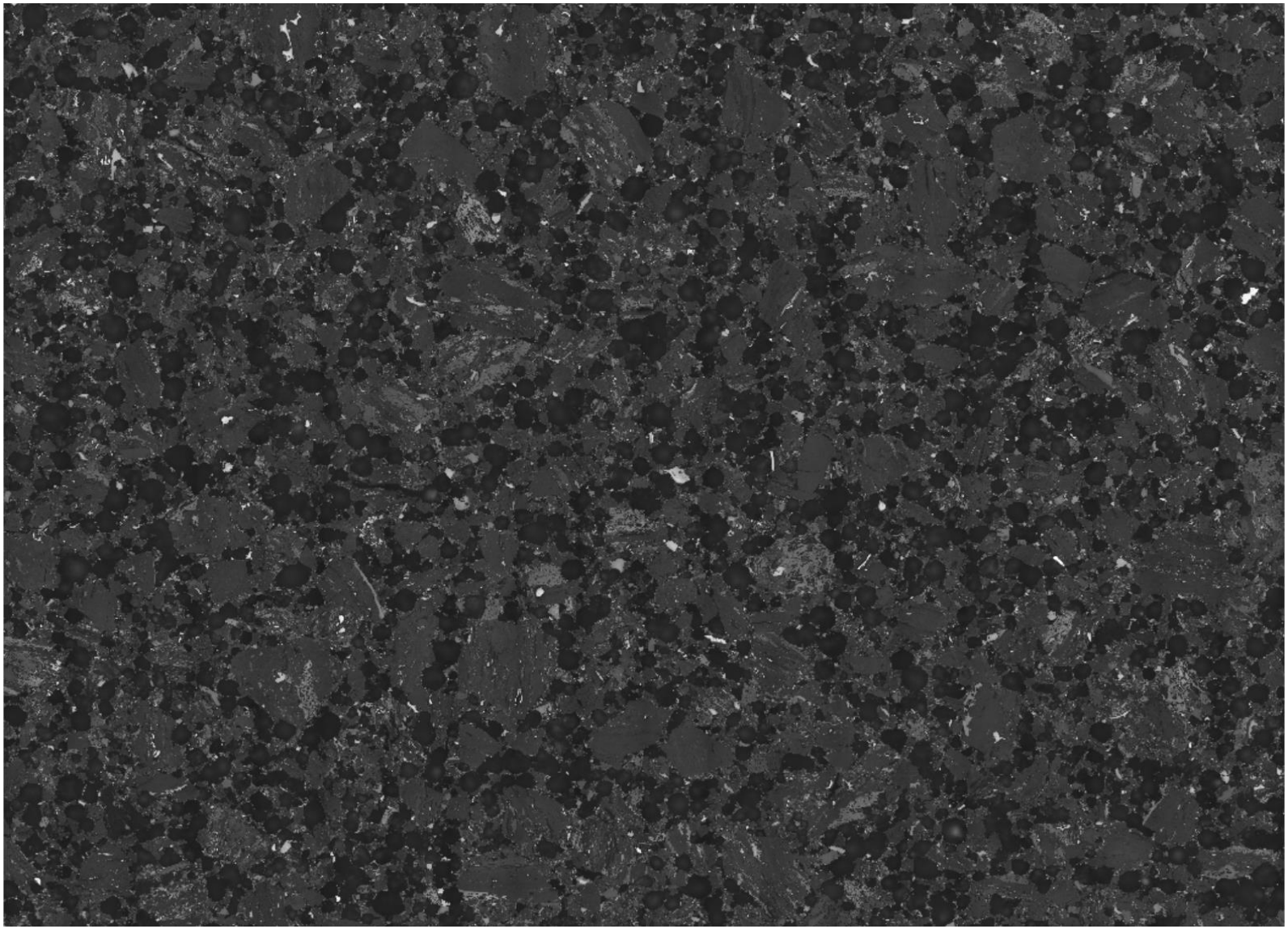
Single O.I Image



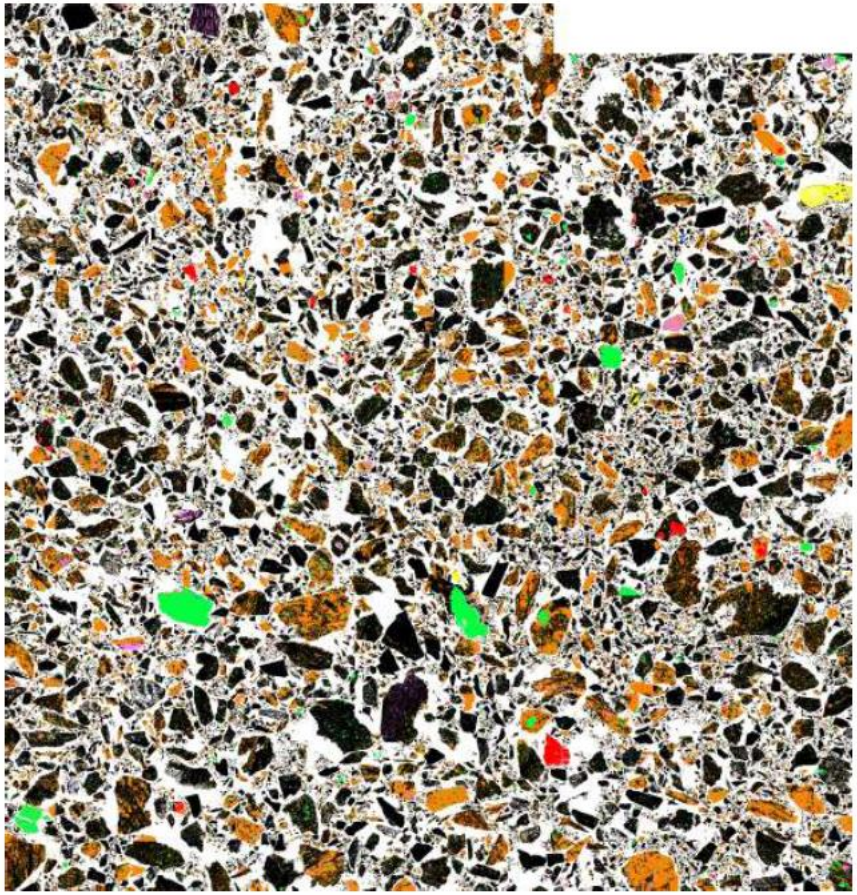
2x2 O.I Image



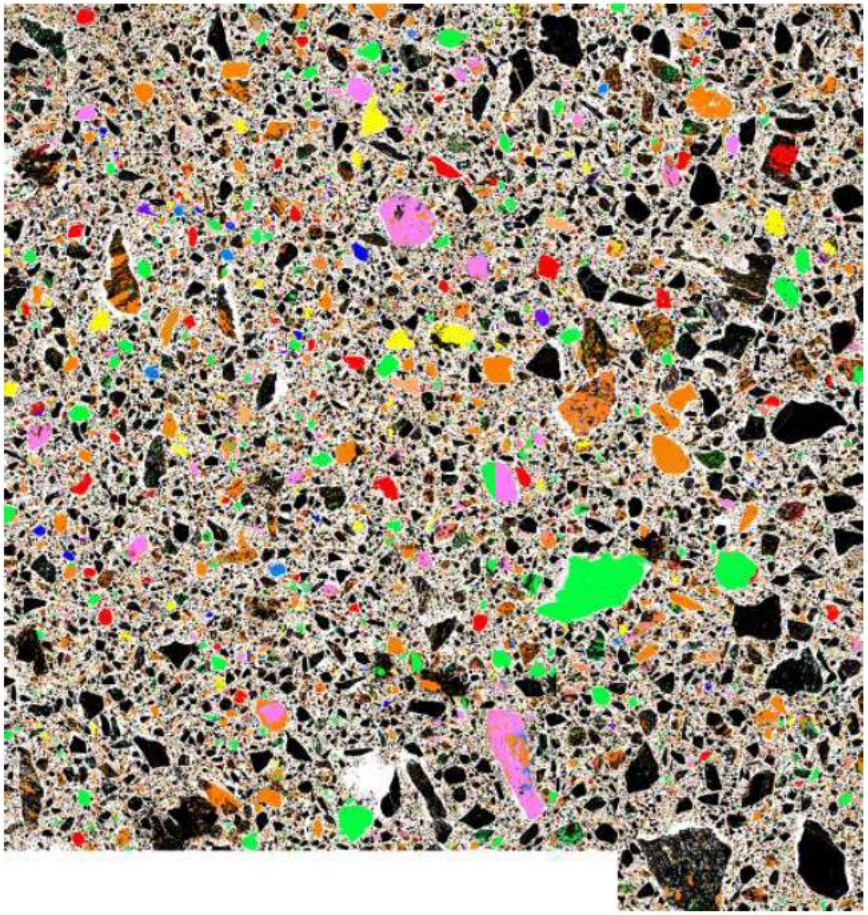
30 x 30 O.I image



Mineral Liberation Analysis

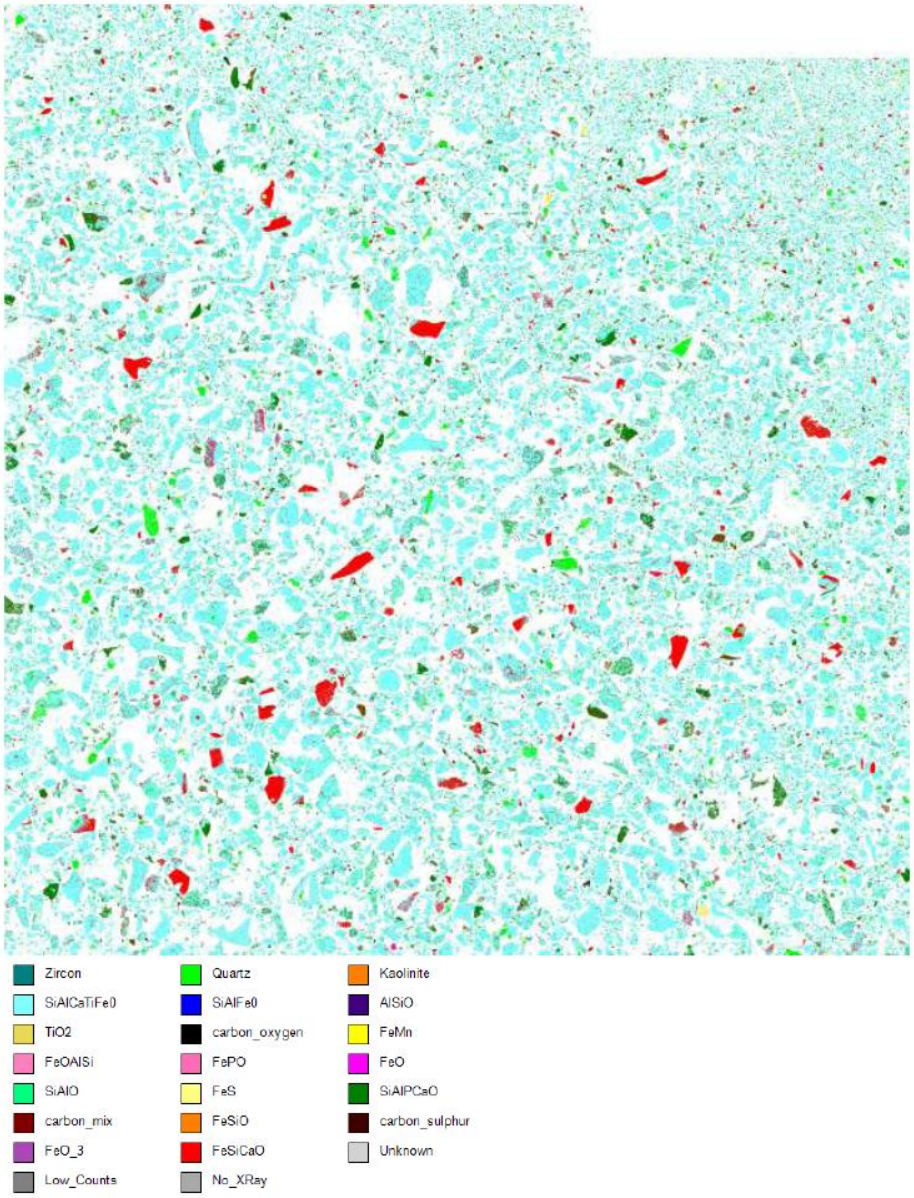


Coal
Kaolinite
Orthoclase
Dolomite_Fe
Unknown
Quartz
Pyrite
Albite
Calcite
Low_Counts
Muscovite_shale
Hematite
Dolomite
carbonate
No_XRay



Coal
Kaolinite
Orthoclase
Dolomite_Fe
Unknown
Quartz
Pyrite
Albite
Calcite
Low_Counts
Muscovite_shale
Hematite
Dolomite
carbonate
No_XRay

Char ‘Ash’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carbon_mix
FeO_3
Low_Counts
Quartz
SiAlFe0
carbon_oxygen
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FeS
FeSiO
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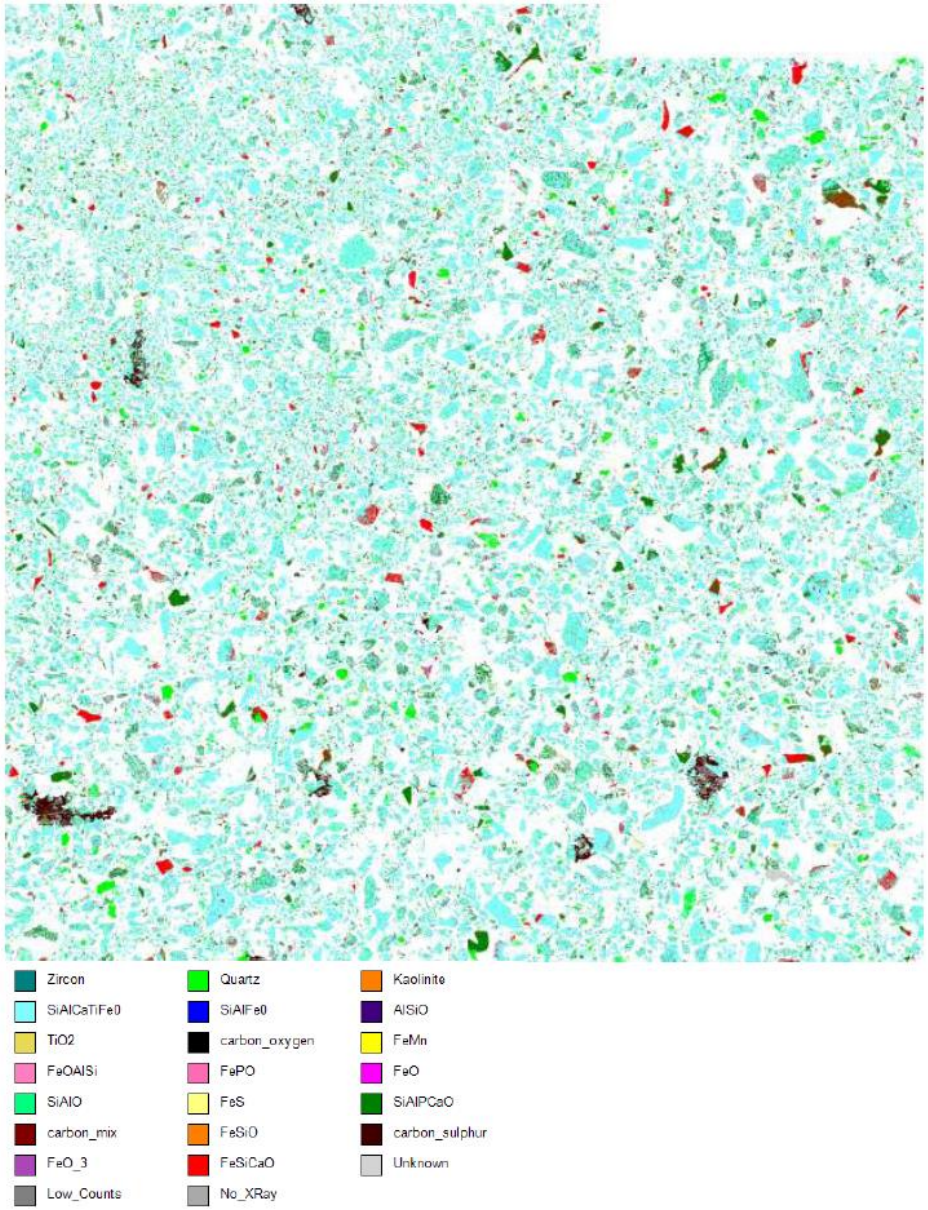
Kaolinite
AlSiO
FeMn
FeO
SiAlPCaO
carbon_sulphur
Unknown


Zircon
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TiO2
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FeO_3
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Quartz
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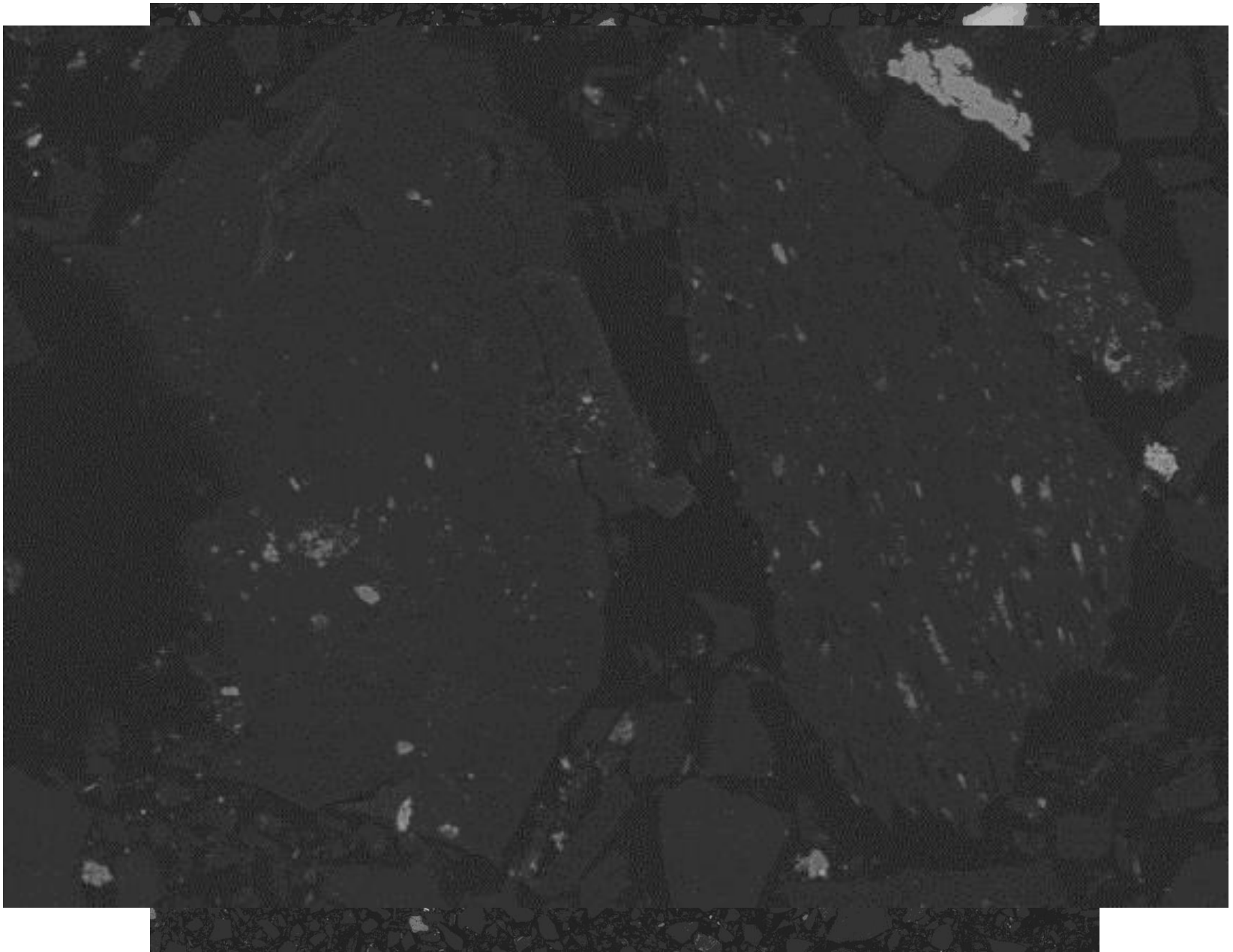
carbon_oxygen
FePO
FeS
FeSiO
FeSiCaO
No_XRay
Kaolinite
AlSiO
FeMn
FeO
SiAlPCaO
carbon_sulphur
Unknown

Minerals vs Macerals with SEM imaging



Blending Strategy

Criterion	Grade	Final Blend
Petrographic Composition		
Pyrite Characteristics		
Ash Composition		

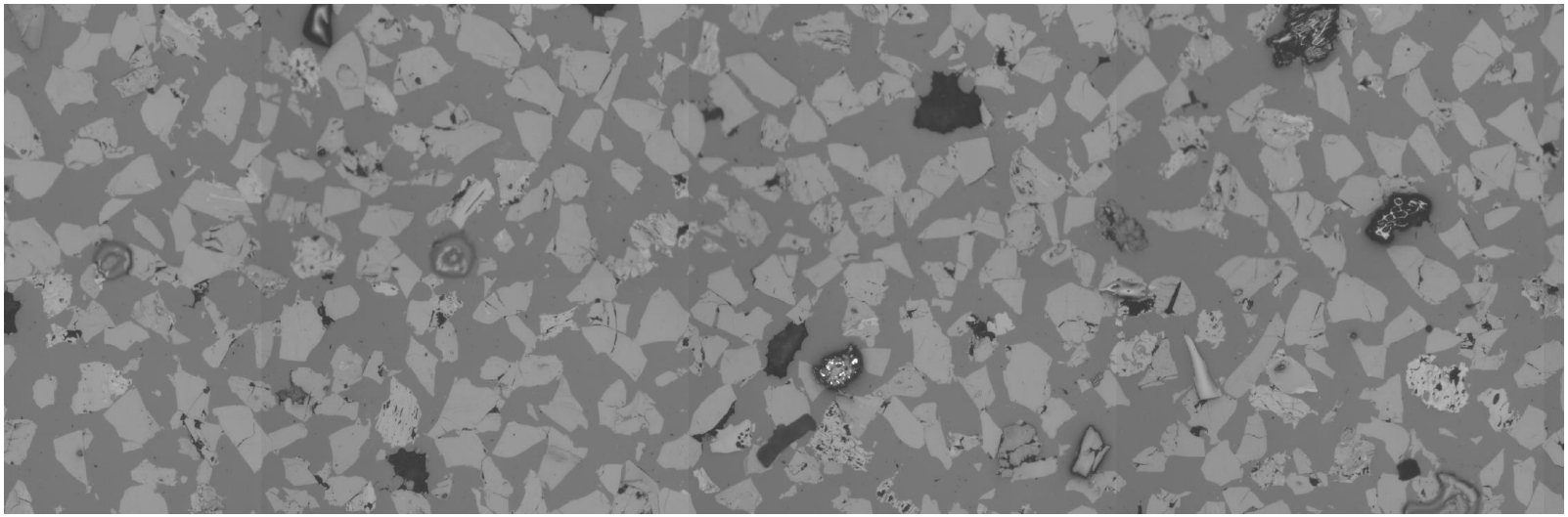
Oil Immersion Image Analysis

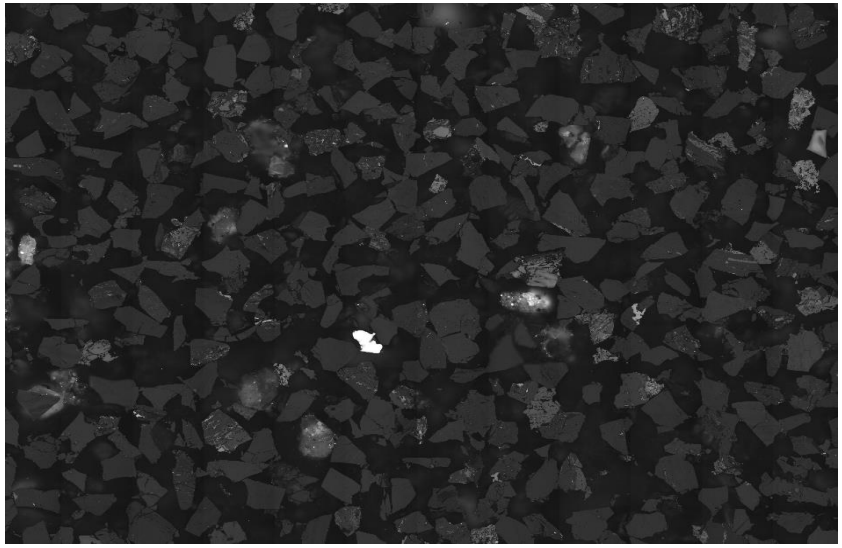
- Image Analysis has helped to improve coal and char assessment significantly over the last 20-30 years
- It remains a challenge to combine all the useful characteristics that are measured using EM/OI
- Predicting all major events (boiler performance, slagging and fouling and EP performance) would be a powerful tool for generators

This Research

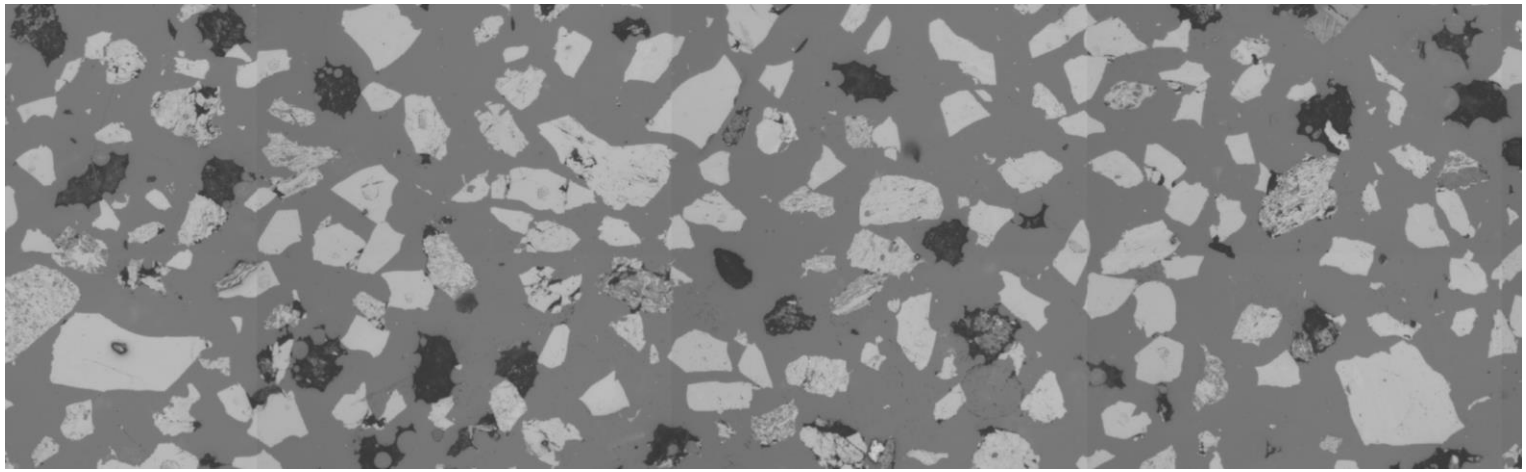
- Traditionally – Oil Immersion
- Recently - Air microscopy has been used to carry out coal and mineral analysis
  - 'Cleaner'
  - Analysing much larger areas
  - Identification of mineral matter
- Air immersion vs oil immersion microscopy with 12 different carbon materials coals, cokes and chars
- 12 different carbon materials were selected to represent a wide reflectance range from low rank coal, high volatile bituminous coals, low volatile bituminous, semi-anthracites through to coke material.
- Resin blocks were prepared and polished for each sample to allow air (at 100x magnification) and oil immersion microscopy (at 500x magnification) analysis.
- Automated petrographic analysis to evaluate macerals, microlithotypes and reflectance.

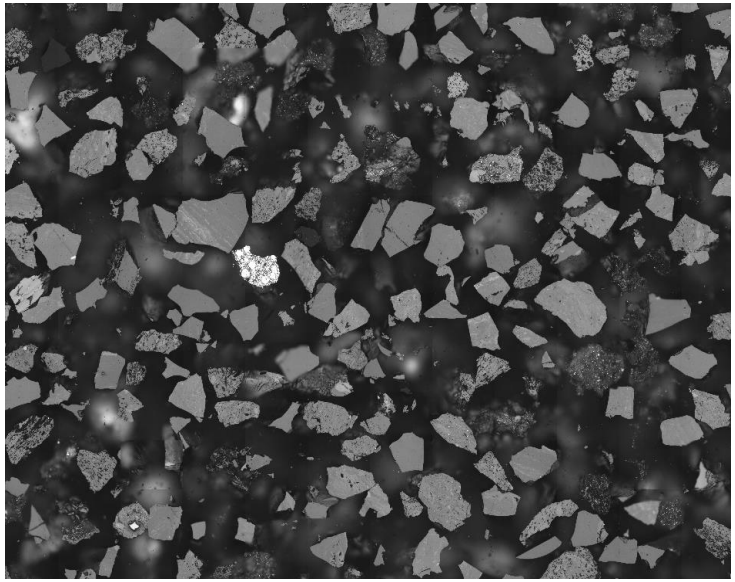
Air vs Oil Mosaic of Low Rank Russian



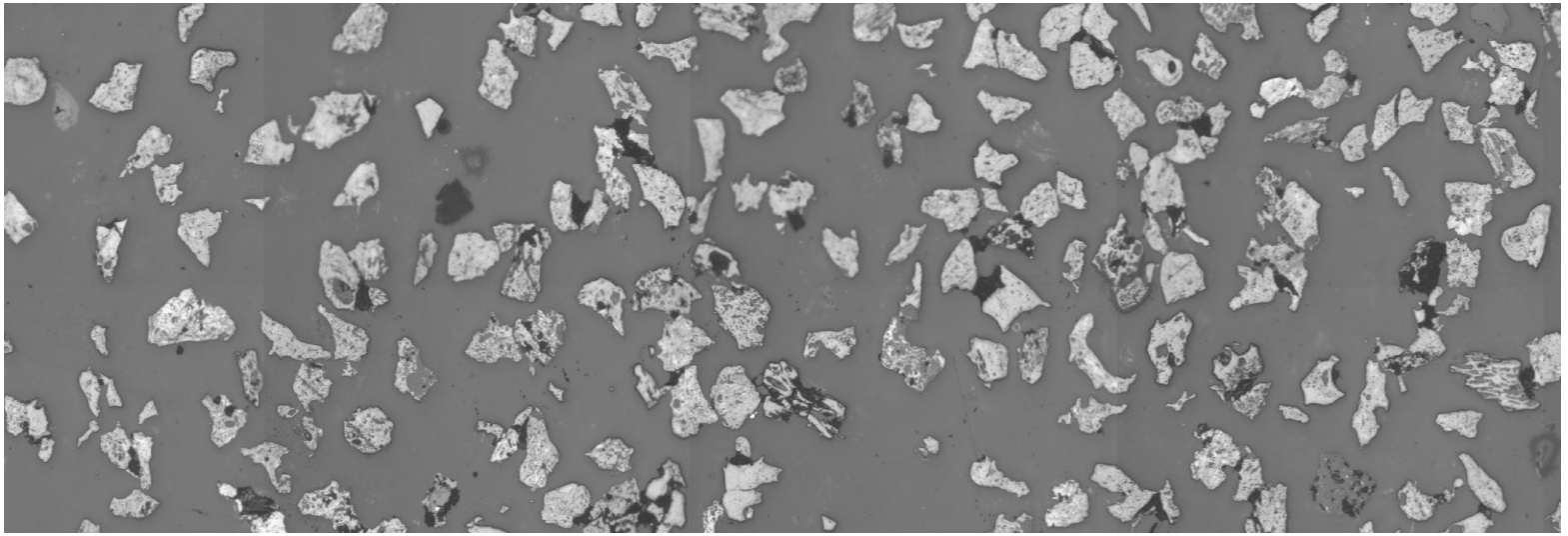


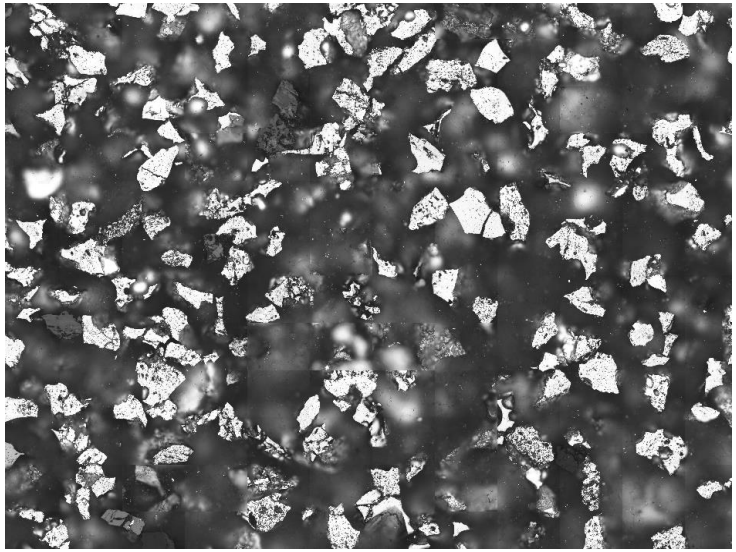
Air vs Oil Mosaic of Semi-Anthracite



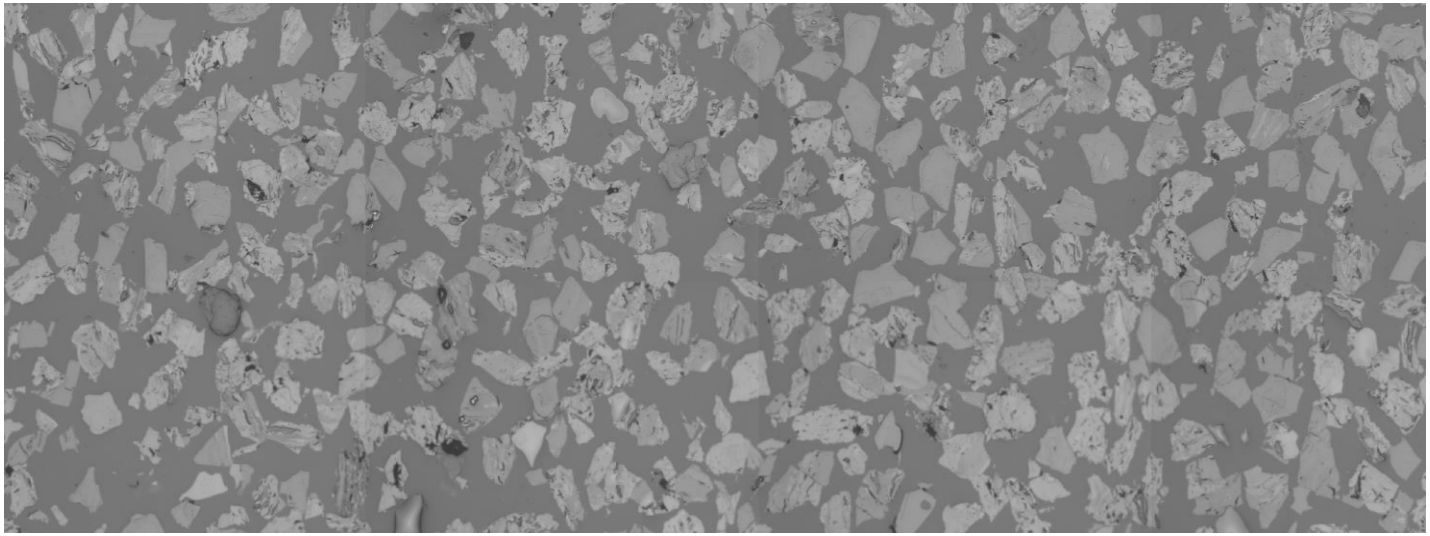


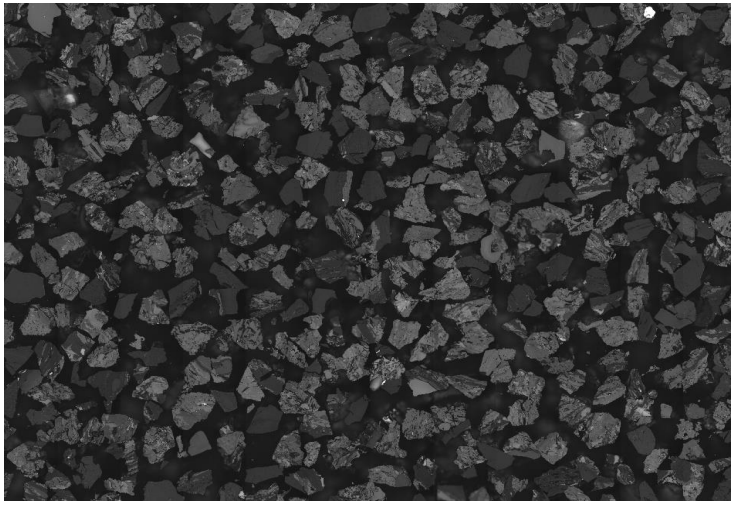
Air vs Oil Mosaic of Coke





Insert an air and oil mosaic of South African (12)

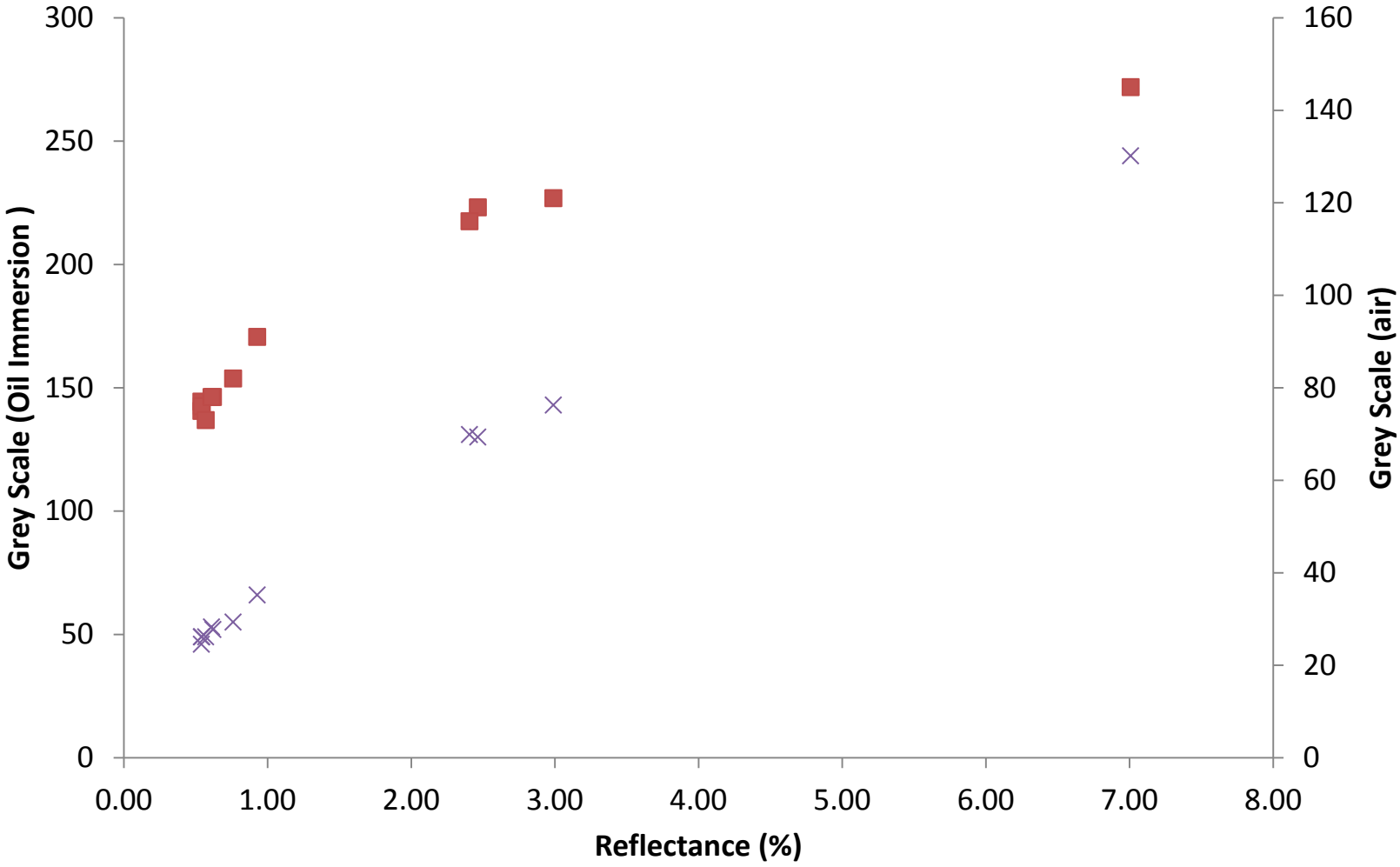


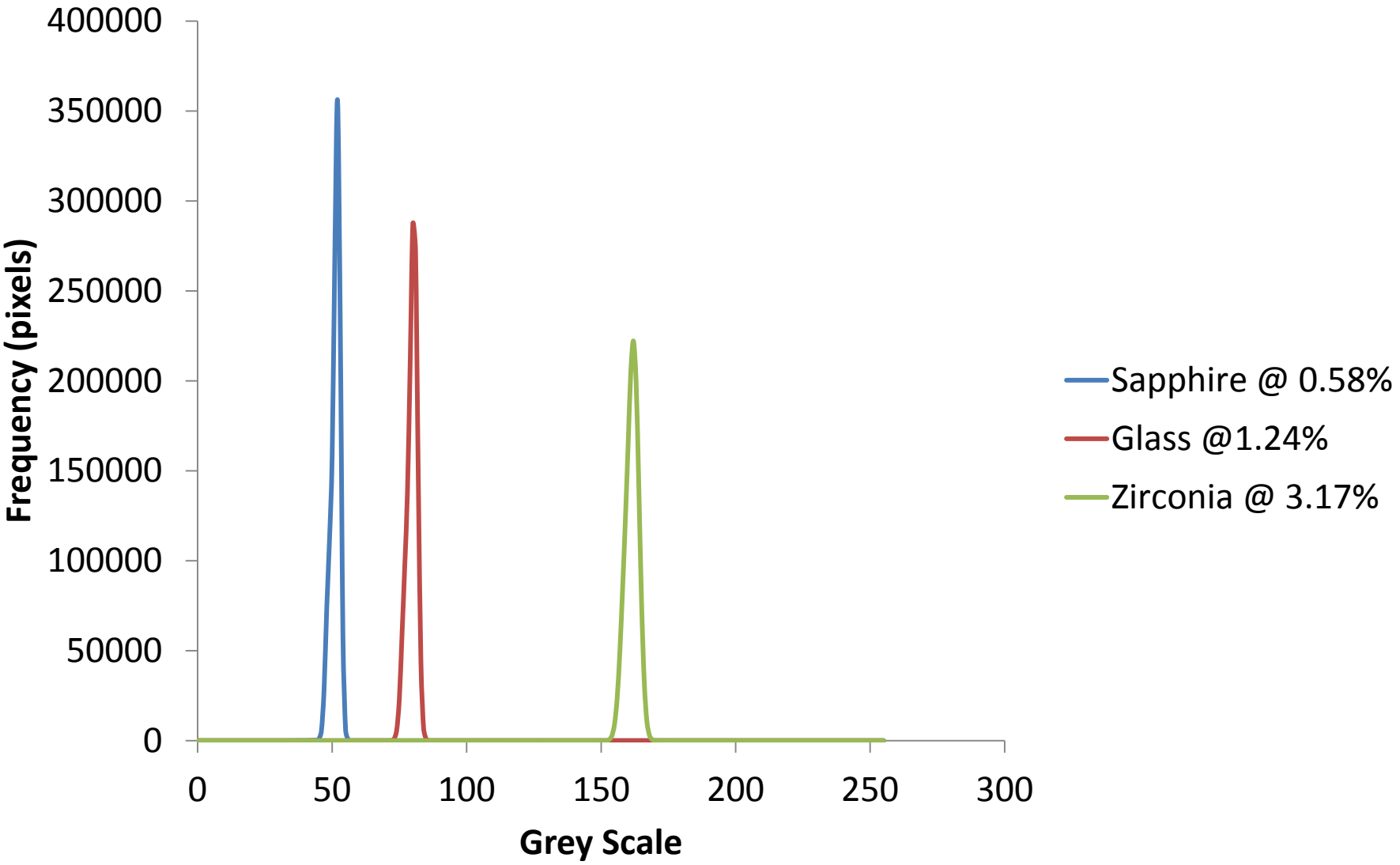


12 samples of coal/coke

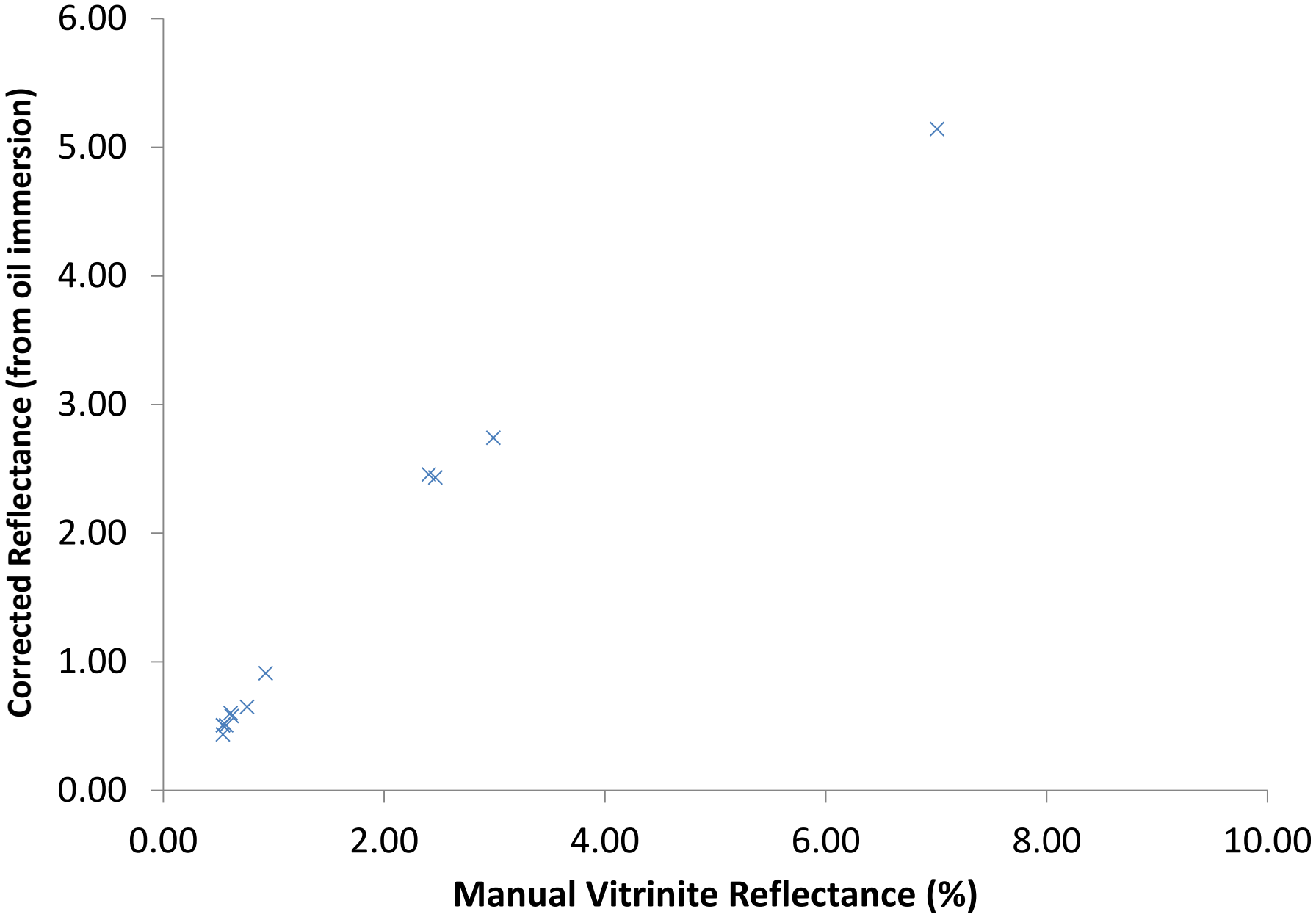
	[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]	[10]	[11]	[12]
Vitrinite	**85.2**	**88.4**	**72.0**	**57.0**	**49.4**	**49.6**	**N/A**	**97.6**	**91.2**	**84.0**	**84.2**	**48.0**
Liptinite	**1.2**	**0.0**	**3.2**	**1.0**	**6.2**	**0.0**	**N/A**	**0.0**	**0.4**	**3.0**	**0.0**	**1.0**
Semi-Fusinite	**5.4**	**3.6**	**9.2**	**18.0**	**27.8**	**39.6**	**N/A**	**1.7**	**4.4**	**6.0**	**7.4**	**23.0**
Fusinite	**8.2**	**8.0**	**15.6**	**24.0**	**16.6**	**10.8**	**N/A**	**0.7**	**4.0**	**7.0**	**8.4**	**28.0**
Vitrinite Reflectance (%)	**0.54**	**0.54**	**0.54**	**0.76**	**0.93**	**2.46**	**(7.01)**	**2.99**	**0.57**	**0.62**	**2.41**	**0.61**

Vitrinite Peak Position vs Vitrinite Reflectance (%)

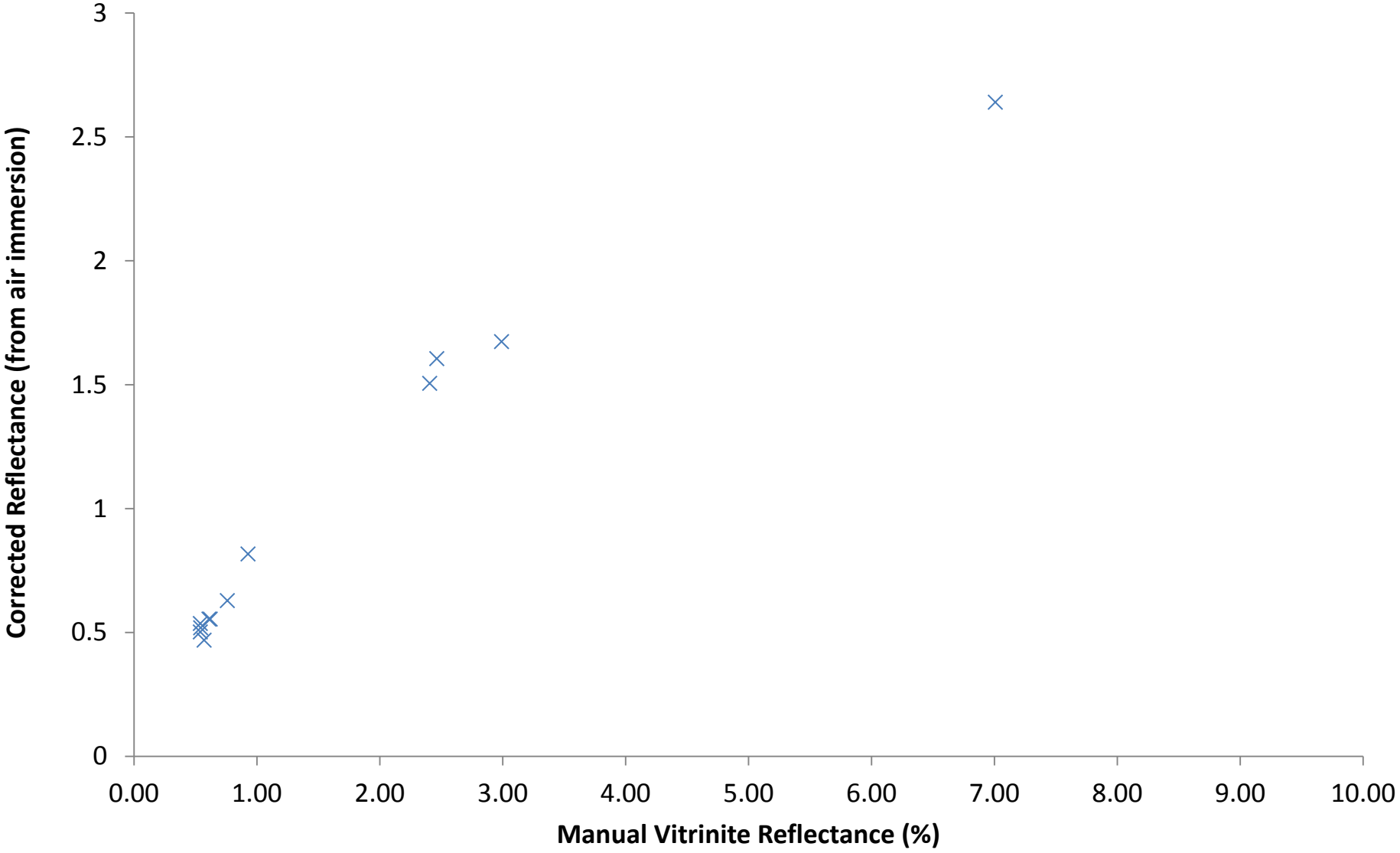


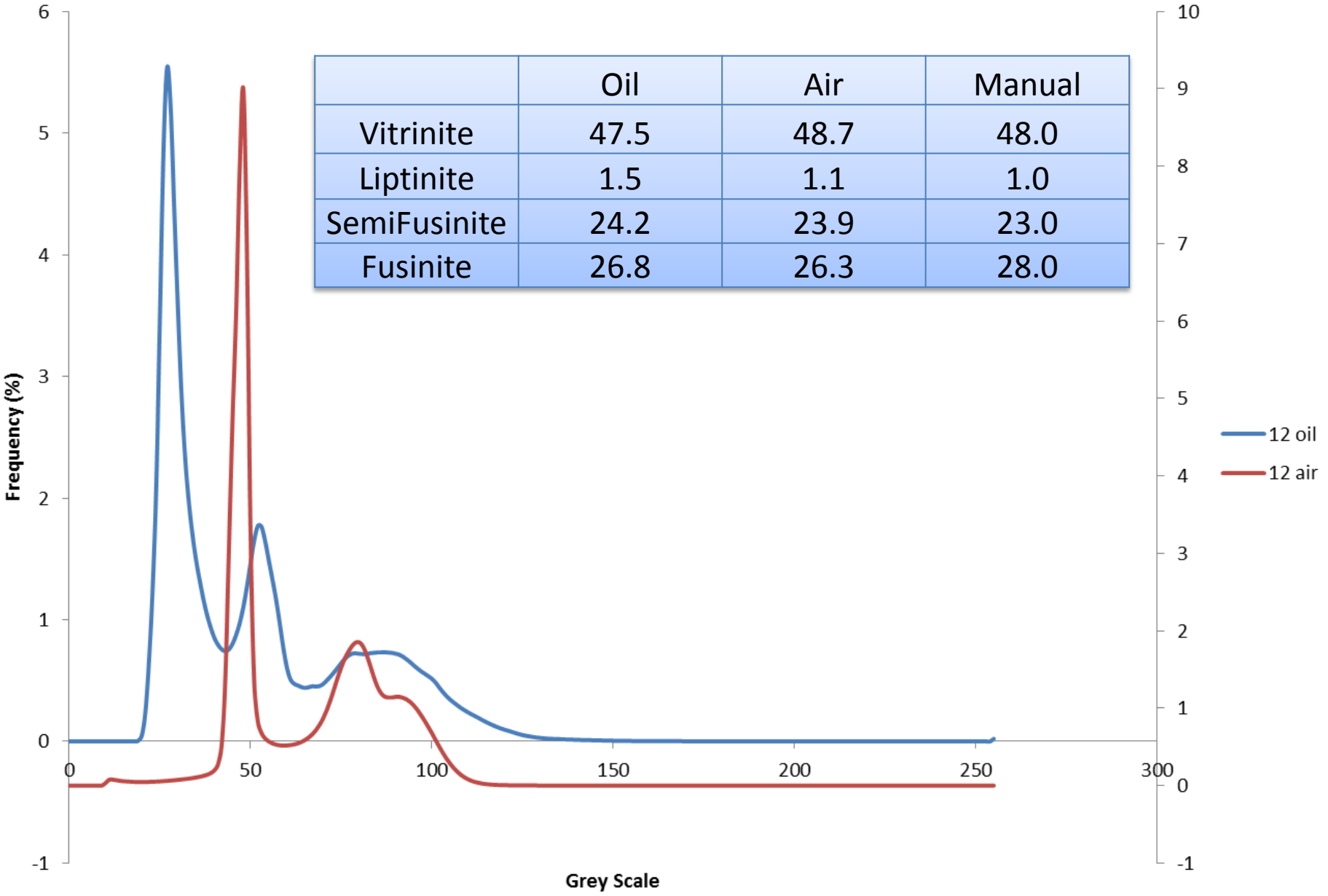

400000
350000
300000
250000
200000
150000
100000
50000
0
Frequency (pixels)
0
50
100
150
200
250
300
Grey Scale
Sapphire @ 0.58%
Glass @1.24%
Zirconia @ 3.17%

Automated vs Manual Vitrinite Reflectance (Oil)



Automated vs Manual Vitrinite Reflectance (Air)





	Oil	Air	Manual
Vitrinite	47.5	48.7	48.0
Liptinite	1.5	1.1	1.0
SemiFusinite	24.2	23.9	23.0
Fusinite	26.8	26.3	28.0

Conclusions

Advantages of Oil Immersion	Advantages of Air Immersion
Superior contrast	Larger captured area
Less sensitivity to sample blemishes	‘Cleaner’
	Much greater reflectance range (0.5 – 8.0)

To be Continued…

- The size and shape of each particle
- Characterised using mineral liberation analysis
- Thermal analysis for intrinsic reactivity
- Size & shape using a Malvern Camsizer

- Pyrolysis in a Drop tube furnace at 200ms, 1% oxygen and 1300^{o}C to produce a devolatilised char sample.
- Char samples analysed using air and oil immersion microscopy, thermal analysis, density and particle size and shape – for comparison with the initial samples.

Acknowledgements

- EPSRC EngD Centre
- BF2RA for funding the project