Methodology for the characterization of the microstructure of nanocomposite polymeric foams using X-ray microtomography

E. Plougonven, P. Marchot, C. Detrembleur, T. M. Phuong, A. Léonard

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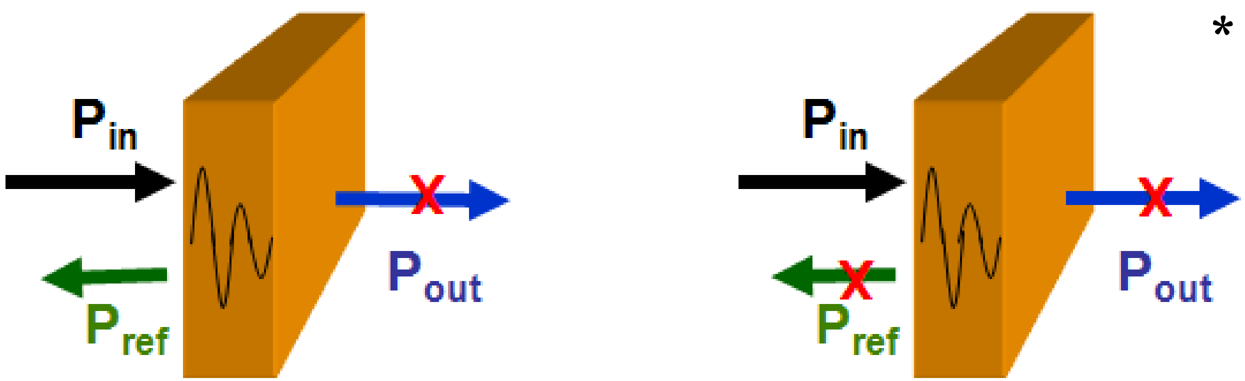
Summary

- Context
- Scanning Electron Microscopy and X-ray microtomography
- Autocorrelation and the rose diagram
- Method validation

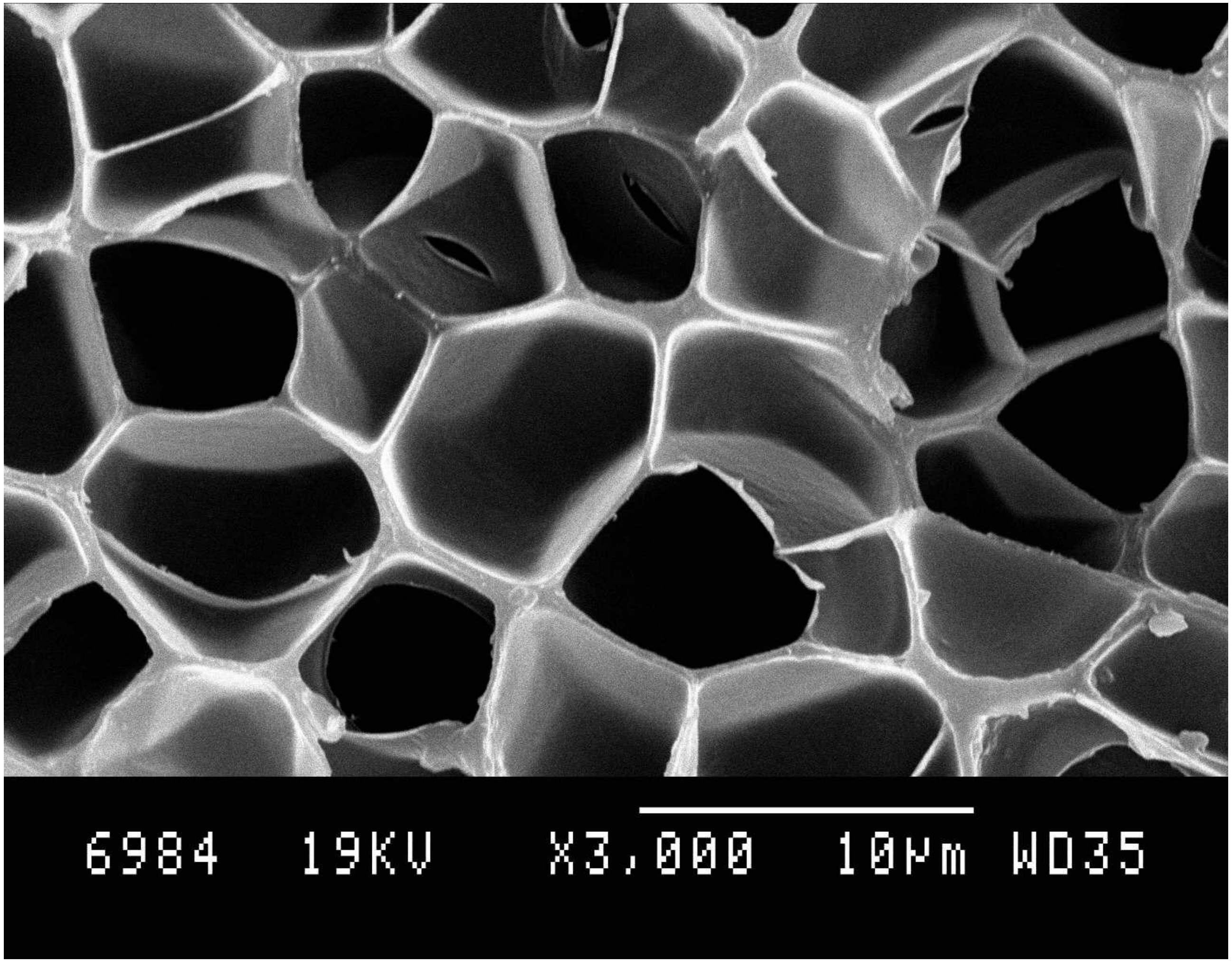
Context

- Objectives
  - Create a good absorber of Electromagnetic Interferences (better than steel)
  - Compare shielding effectiveness with microstructure
  - Acquire and characterize the microstructure



* Image taken from : I. Molenberg, I. Huynen, A.-C. Baudouin, C. Bailly, J.-M. Thomassin, and C. Detrembleur. *Advanced Microwave and Millimeter Wave Technologies: Semiconductor Devices Circuits and Systems*, chapter Foamed Nanocomposites for EMI Shielding Applications, pages 453–470. InTech, 2010.

Scanning Electron Microscopy



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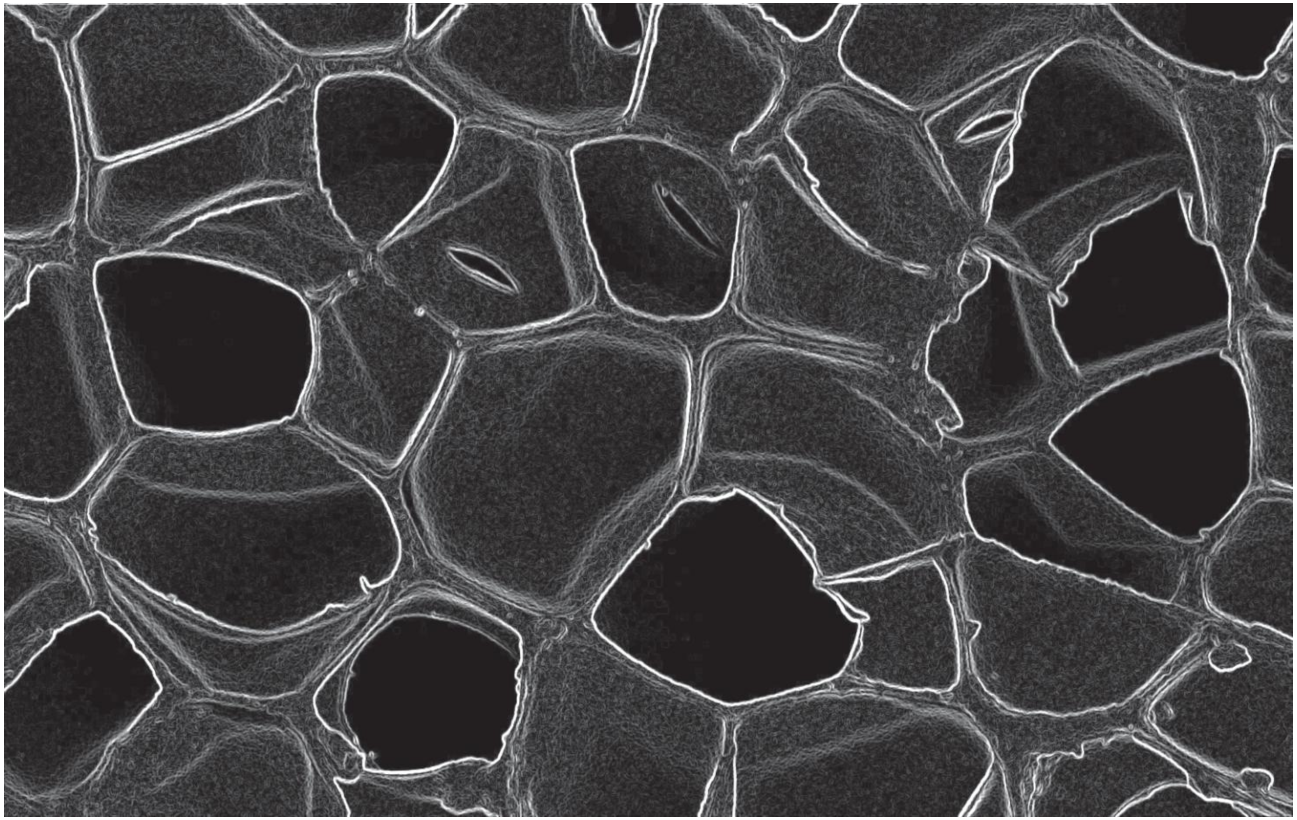
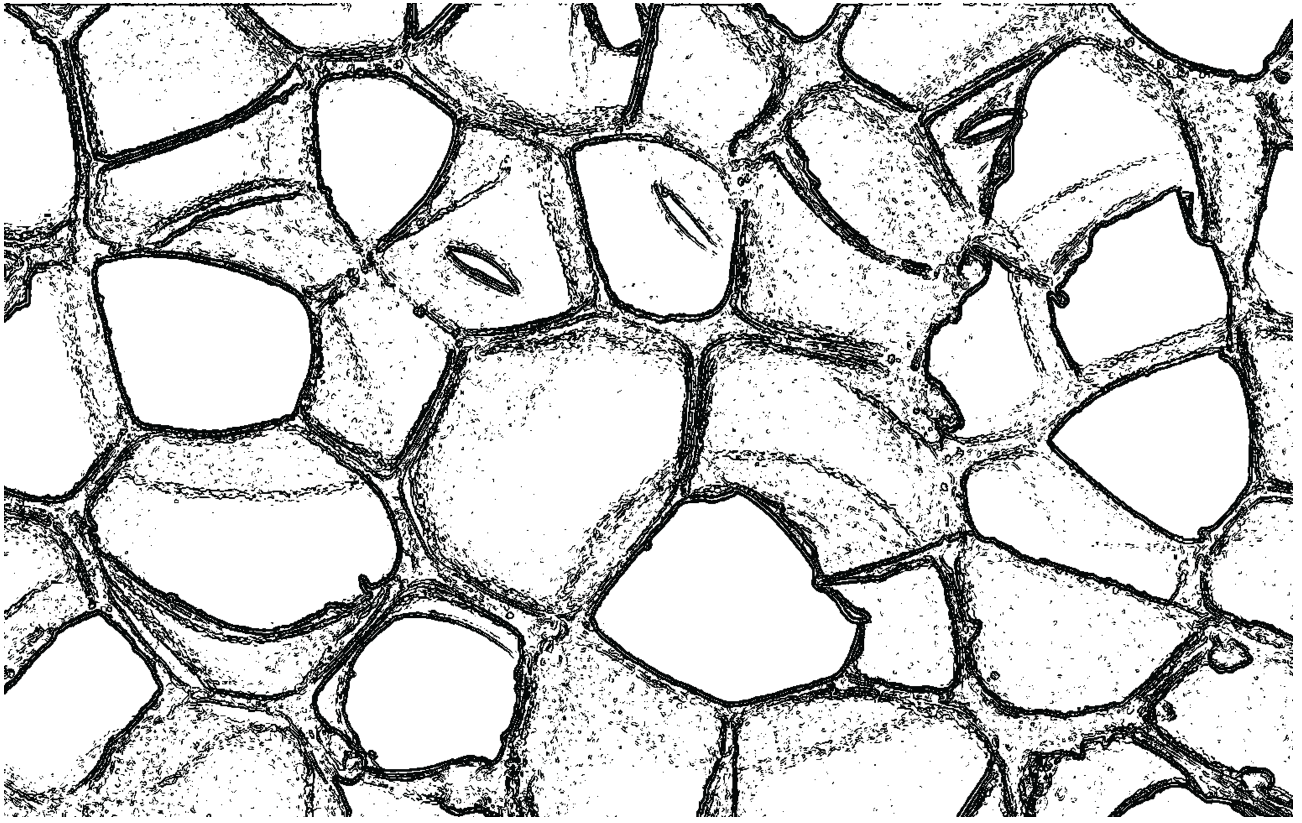
Scanning Electron Microscopy



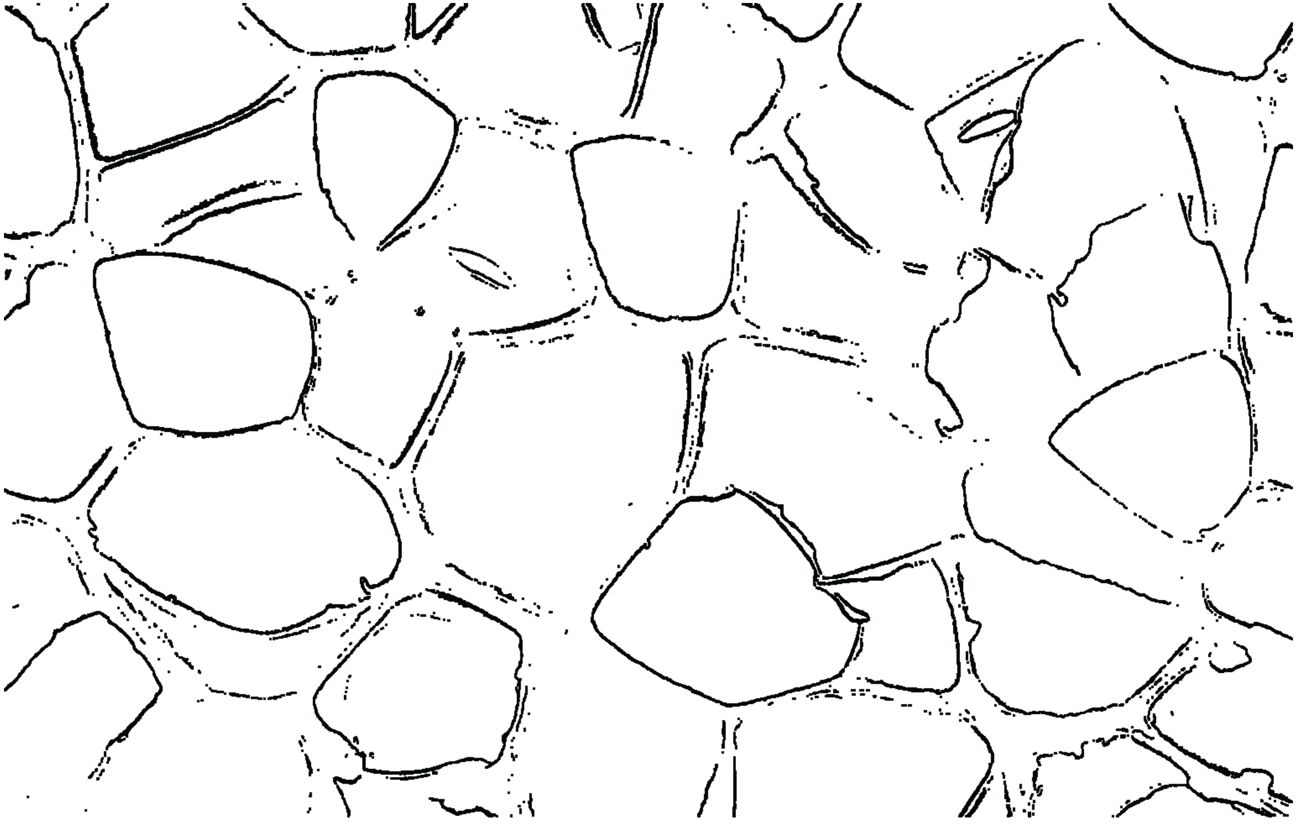
Gradient

Scanning Electron Microscopy



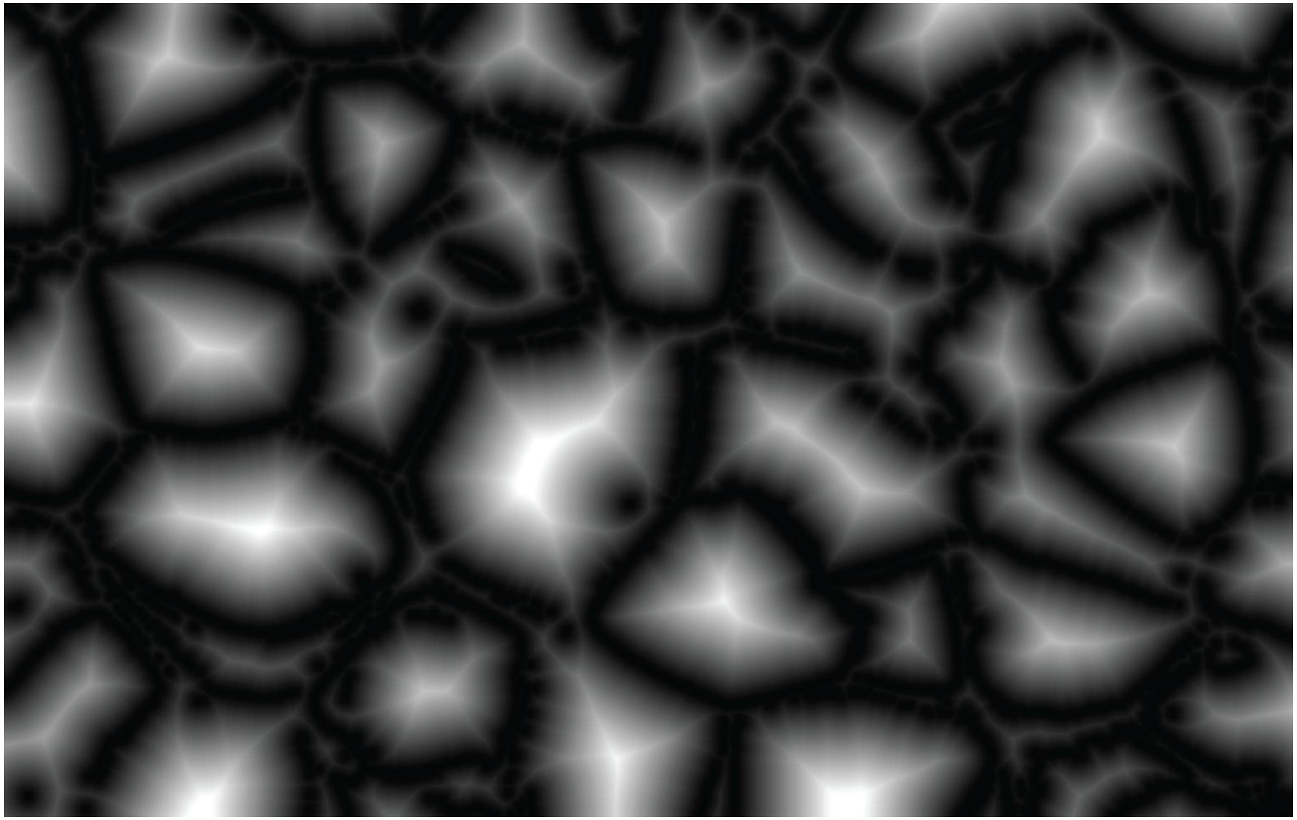
Thresholding

Scanning Electron Microscopy



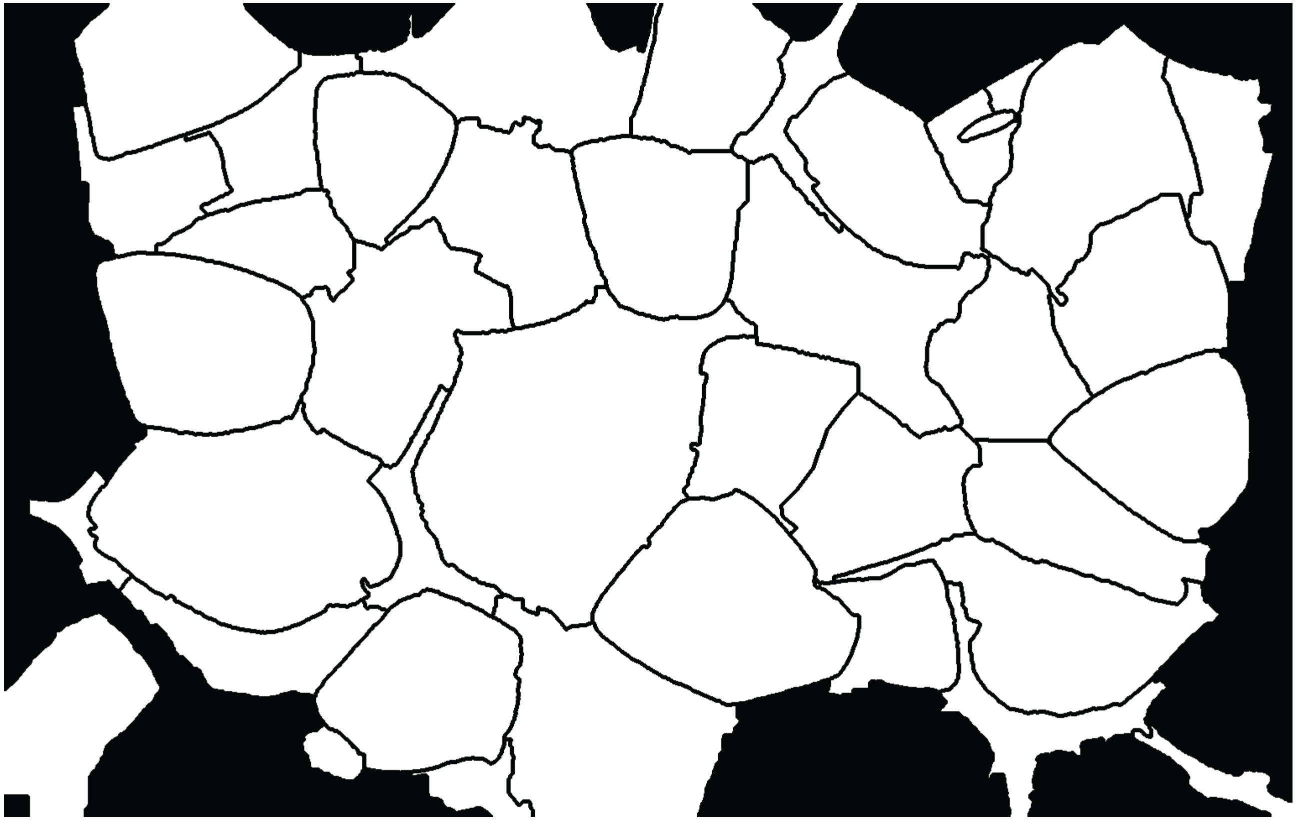
Opening

Scanning Electron Microscopy



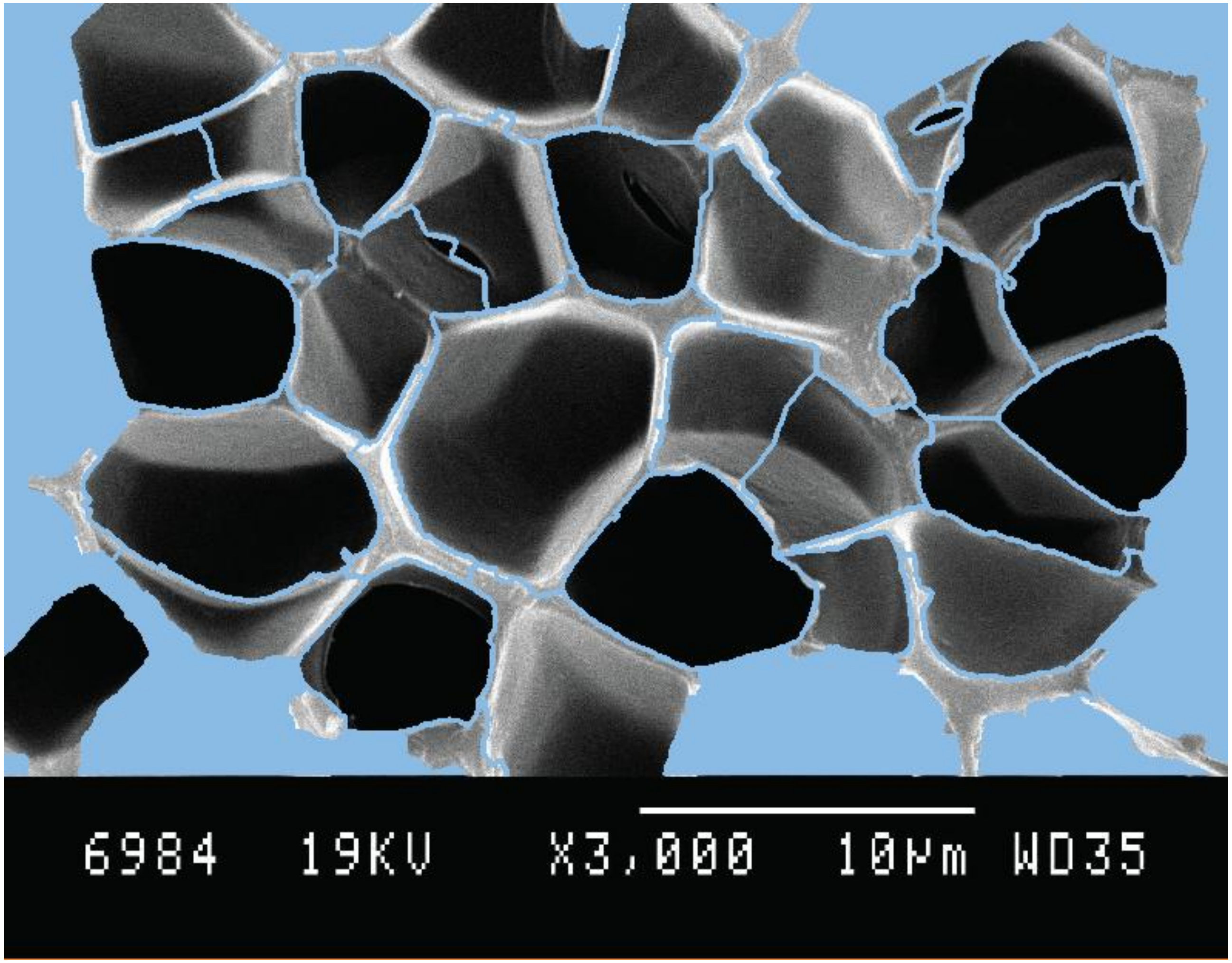
Distance map

Scanning Electron Microscopy

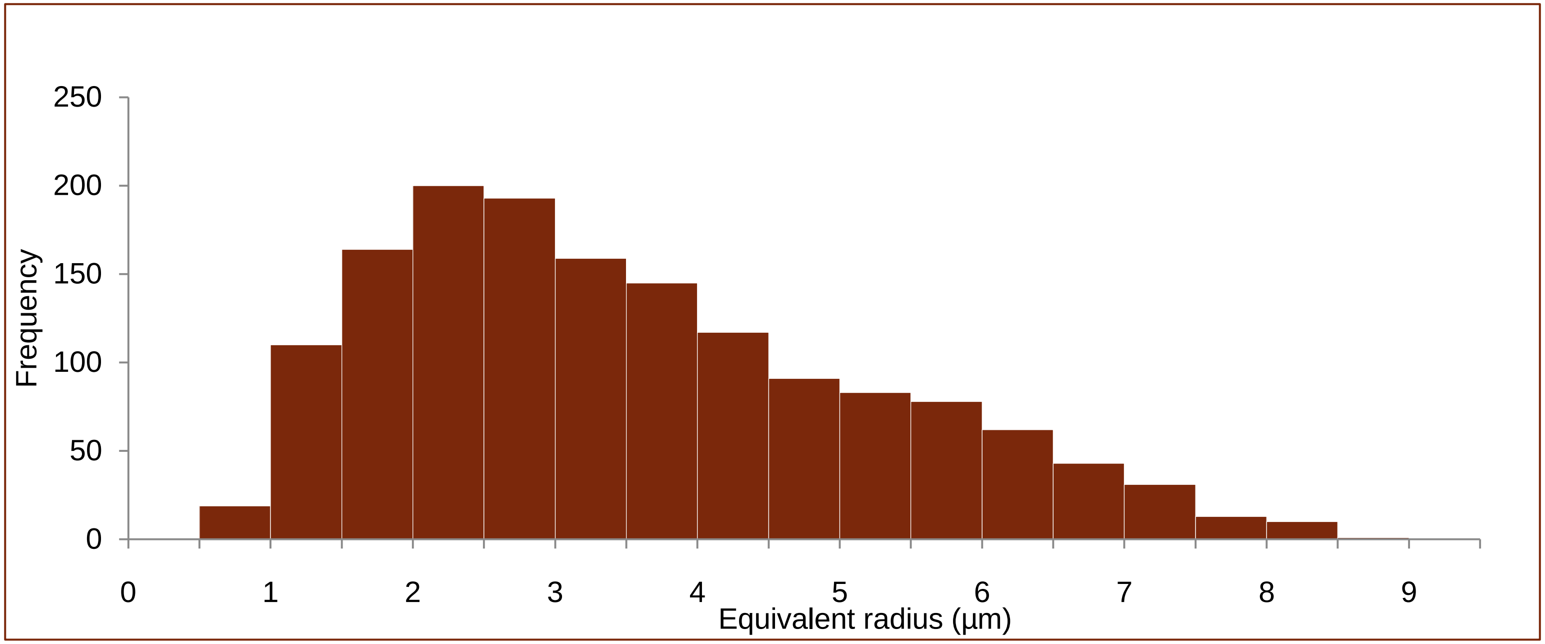


Watershed

Scanning Electron Microscopy

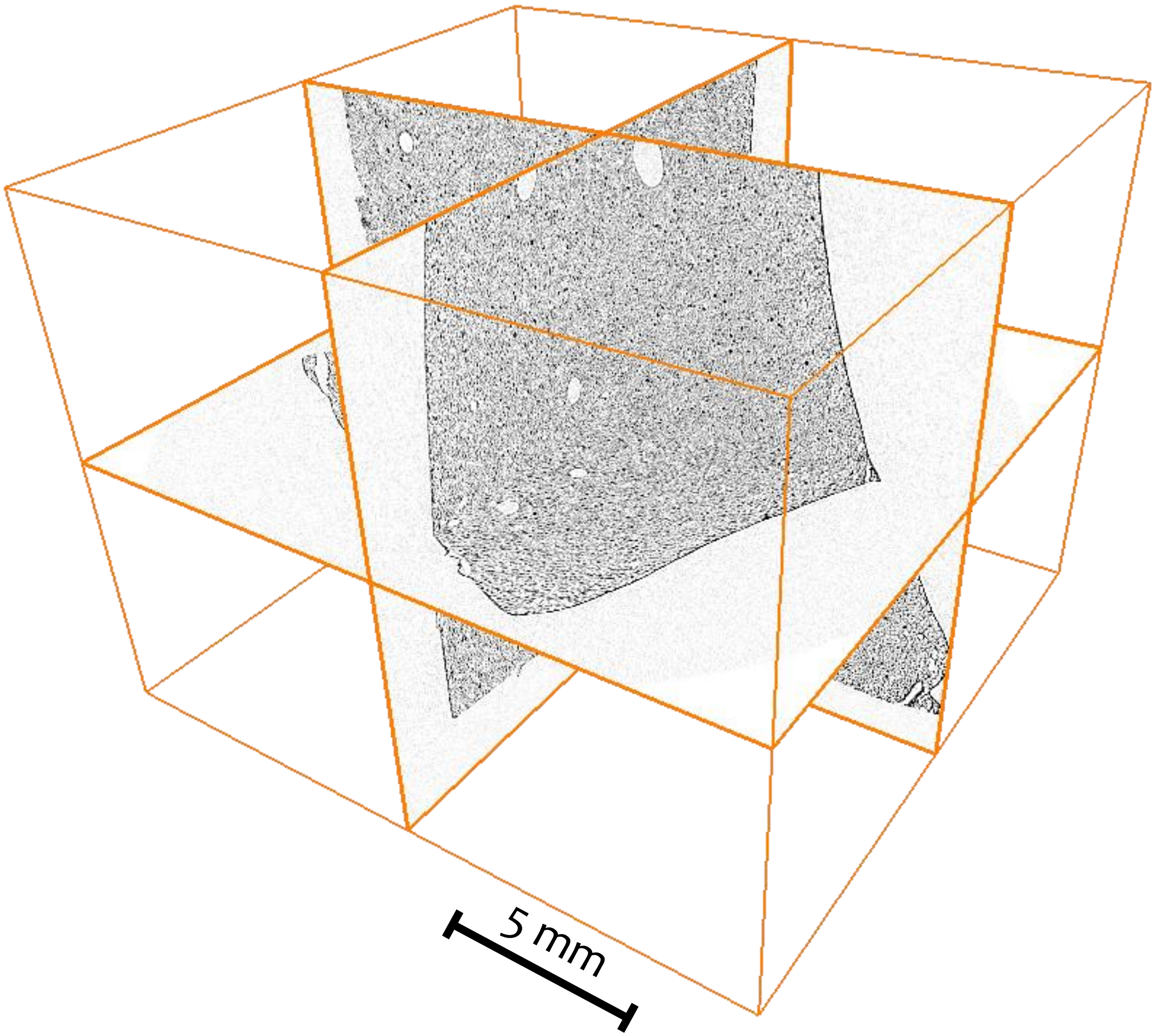


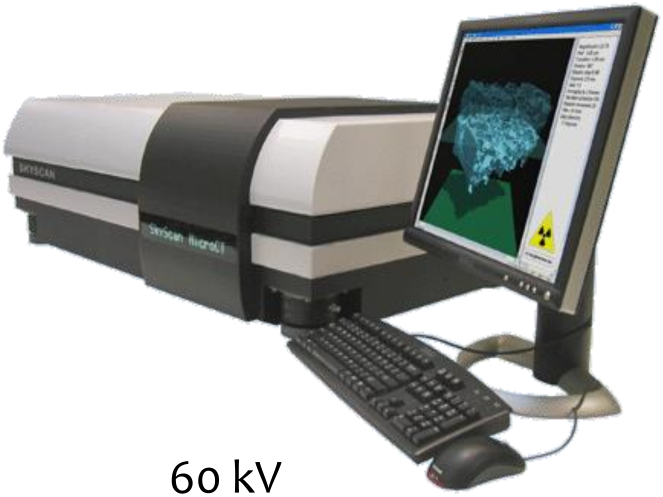
SEM



Frequency
250
200
150
100
50
0
0
1
2
3
4
5
6
7
8
9
Equivalent radius (µm)

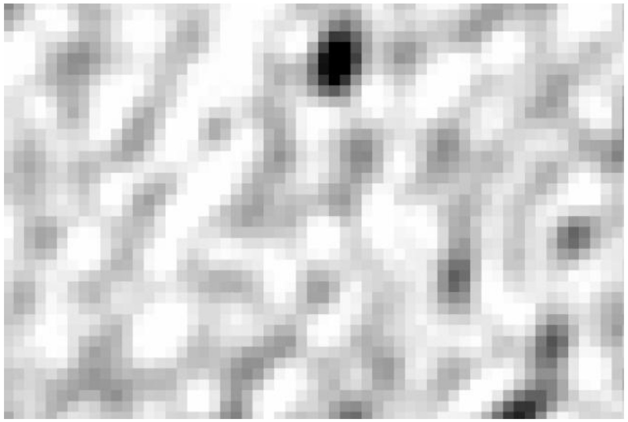
X-Ray microtomography

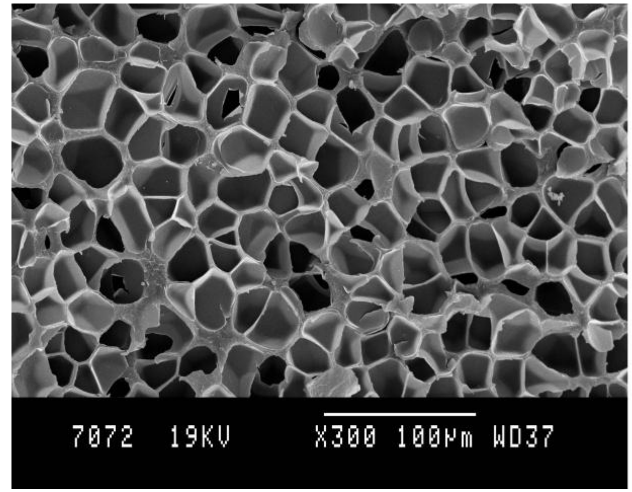


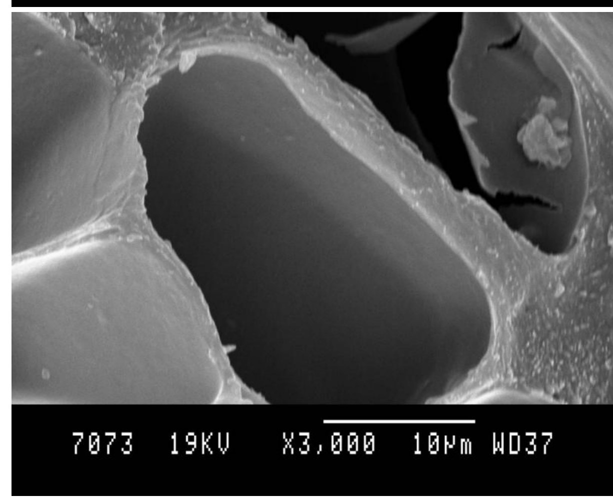


60 kV
6 µm / pixel
3600 projections

X-Ray microtomography





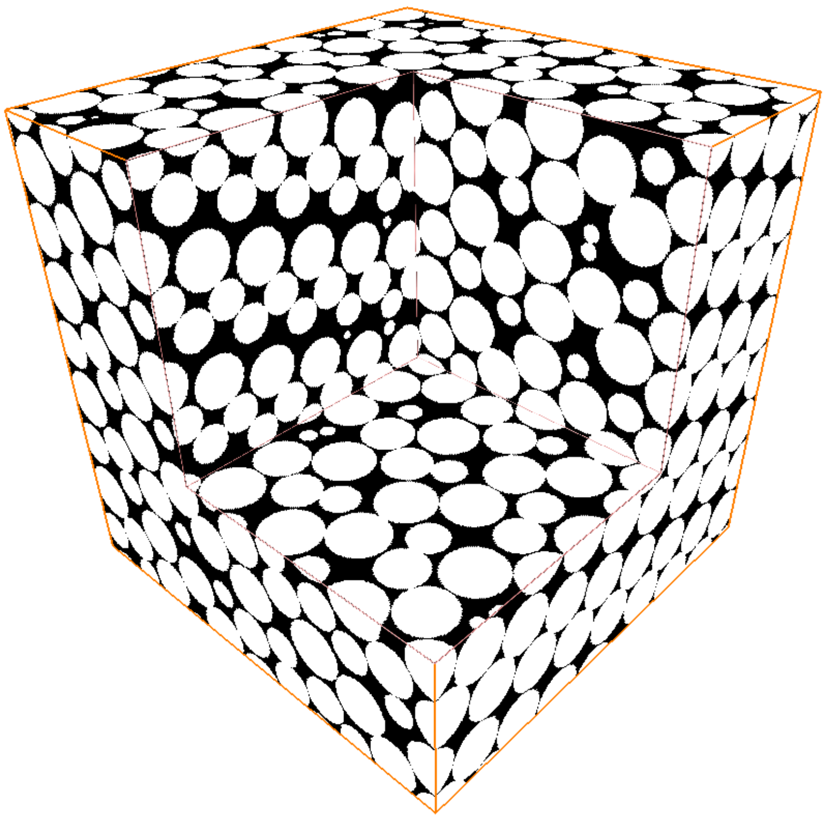


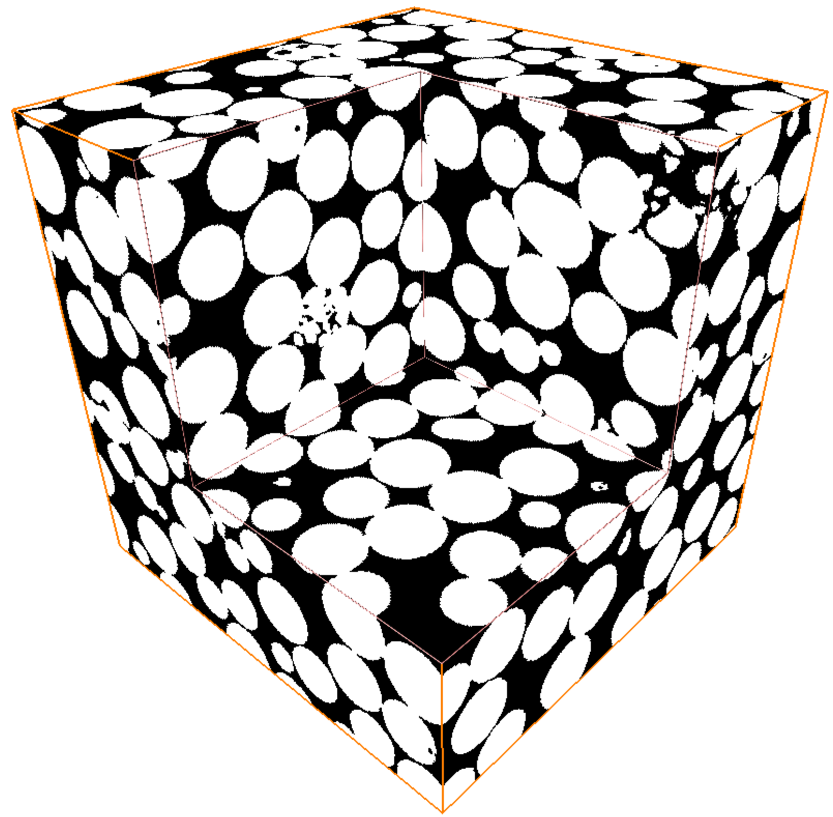
Autocorrelation

- Statistical method
  - 3D autocorrelation

$$R(\tau) = \frac{E[(X_t - \mu)(X_{t+\tau} - \mu)]}{\sigma^2}$$

$$R(\tau_x, \tau_y, \tau_z)$$





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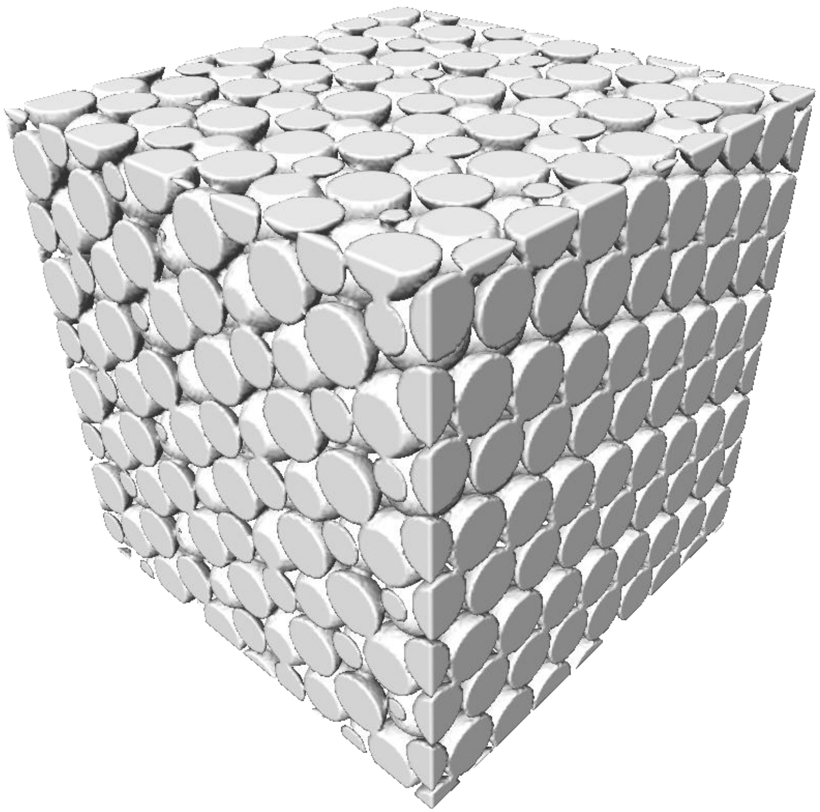

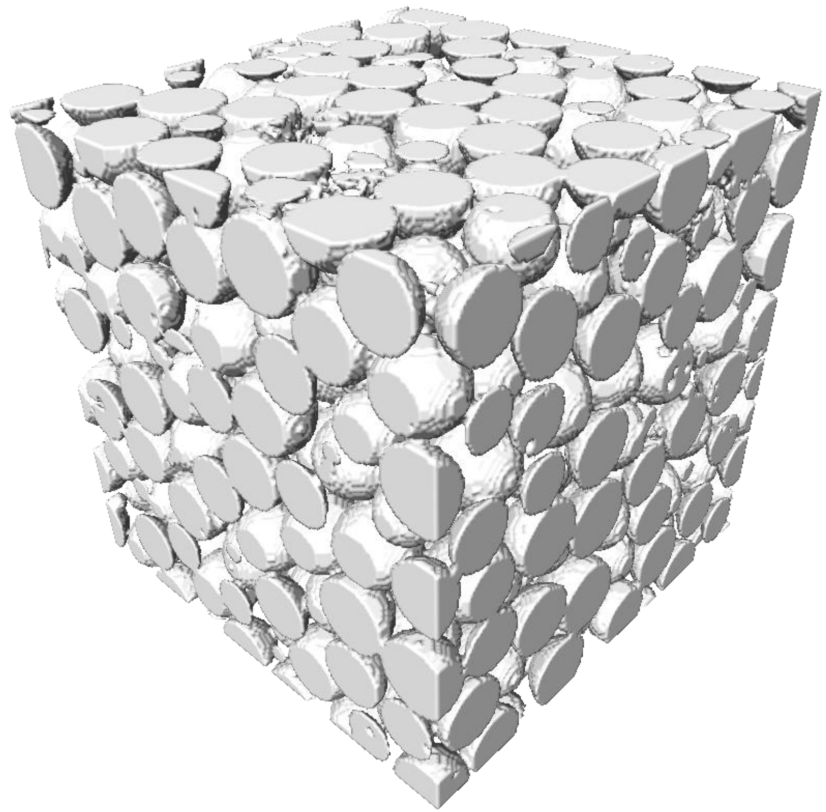

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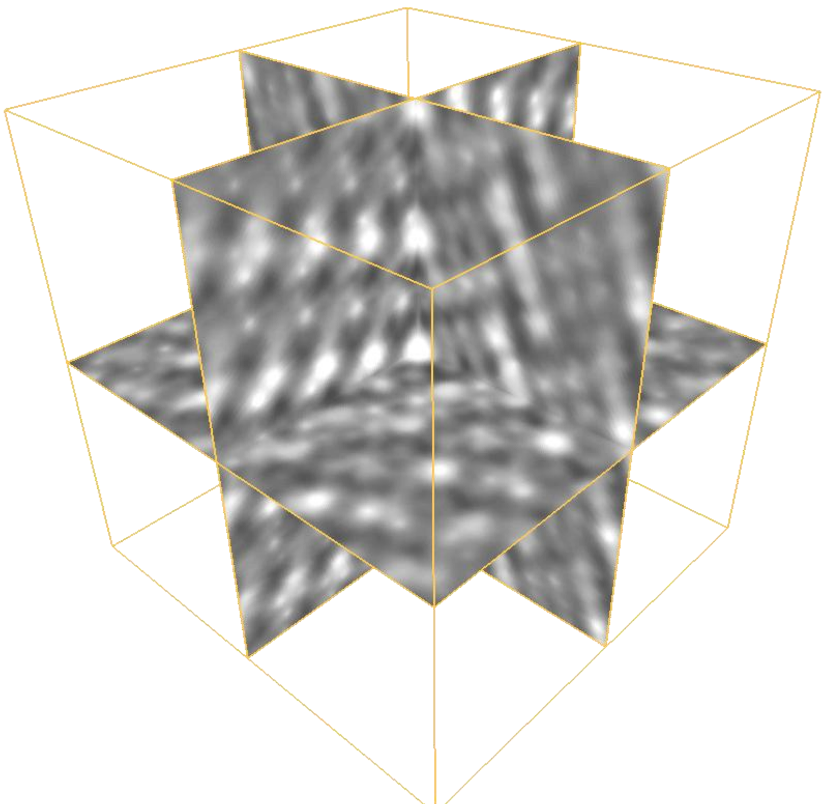

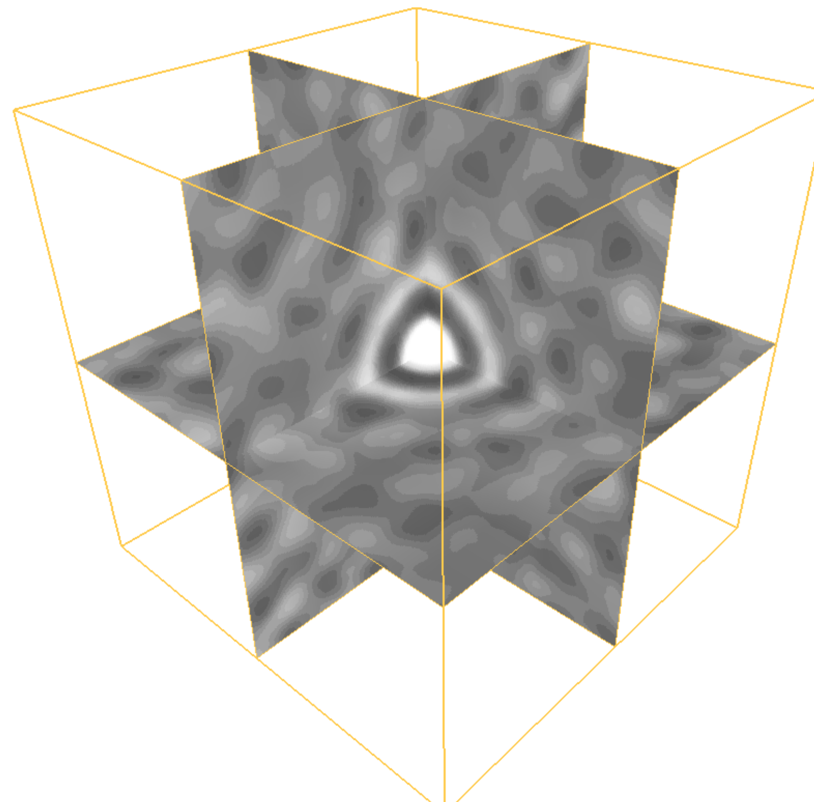

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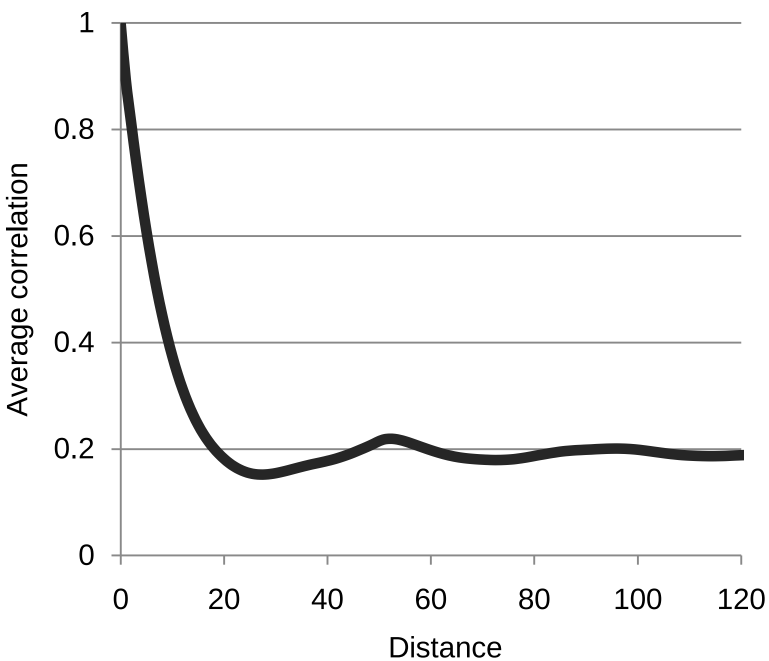

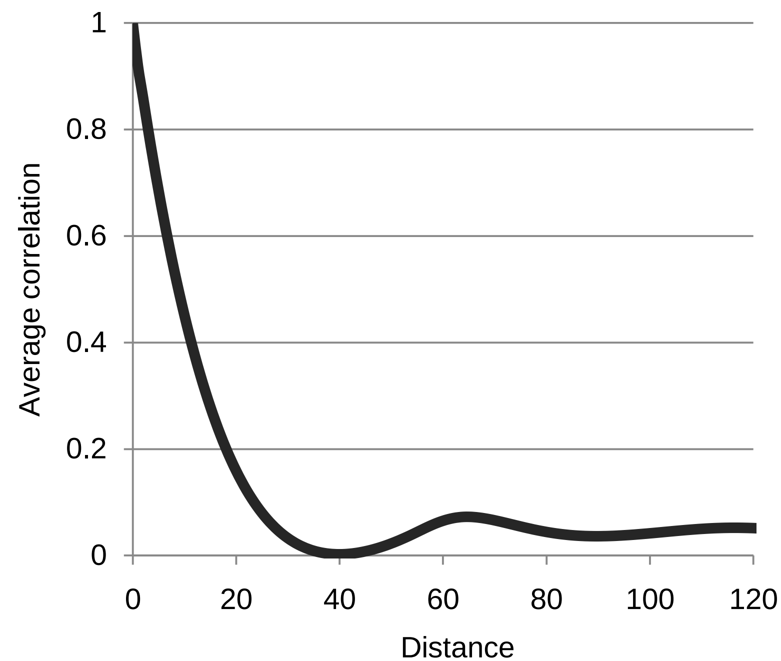

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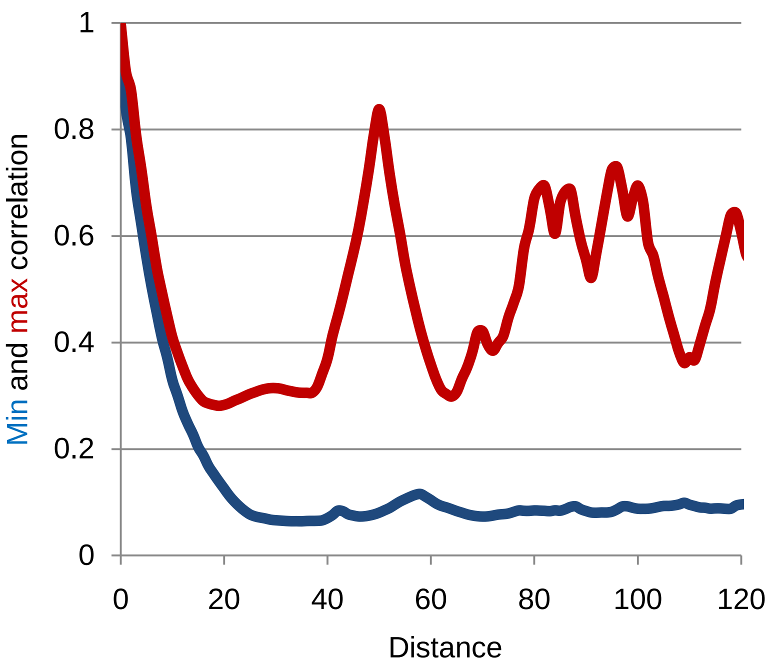

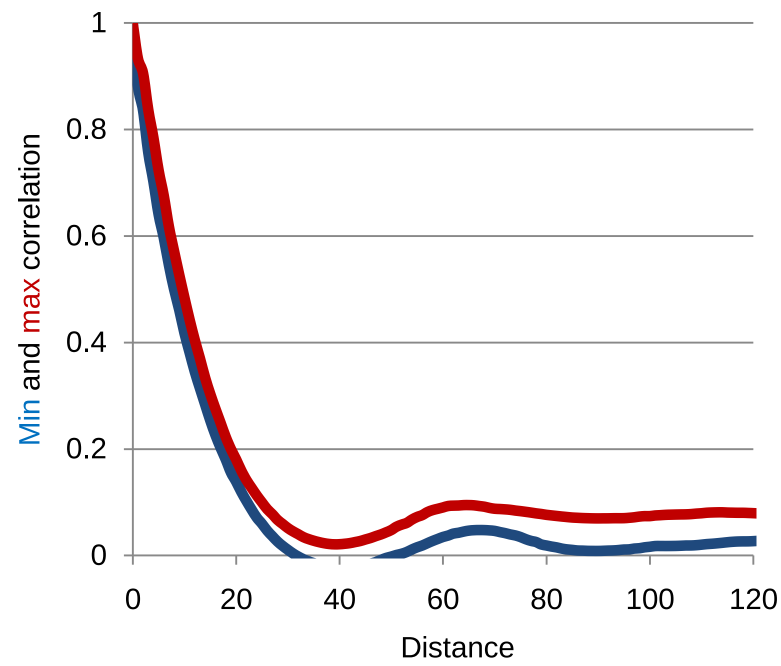

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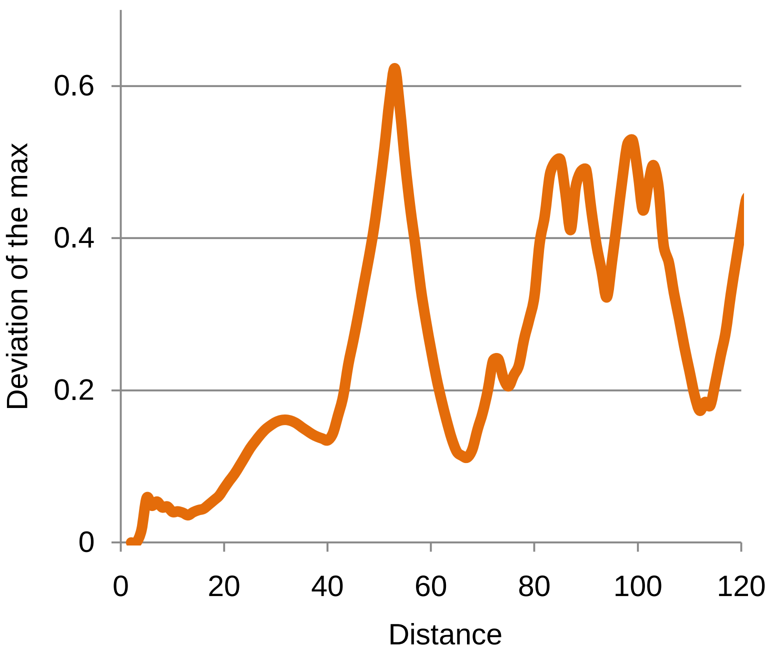
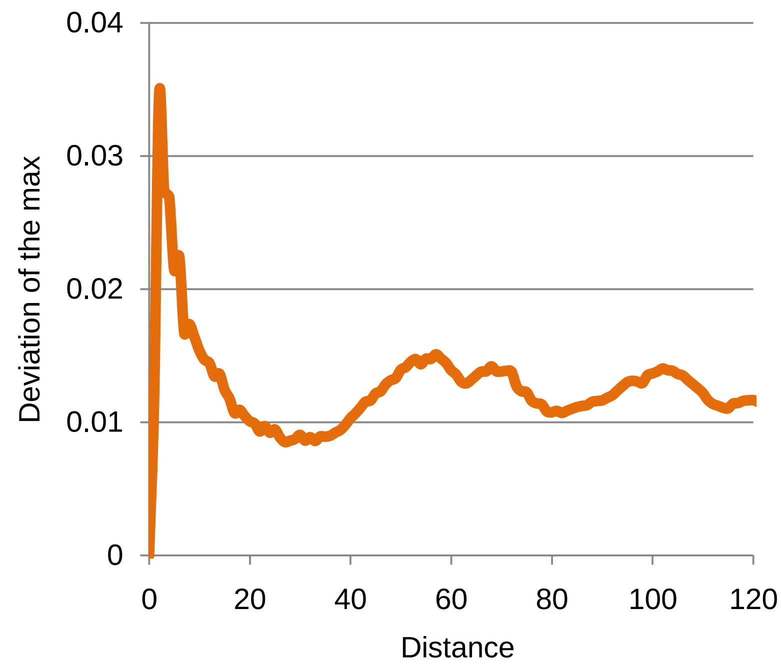
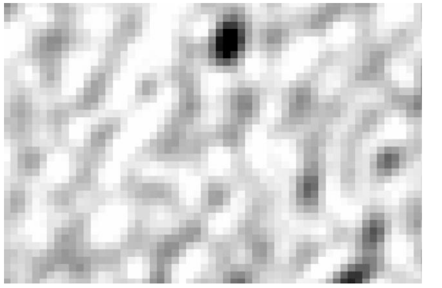
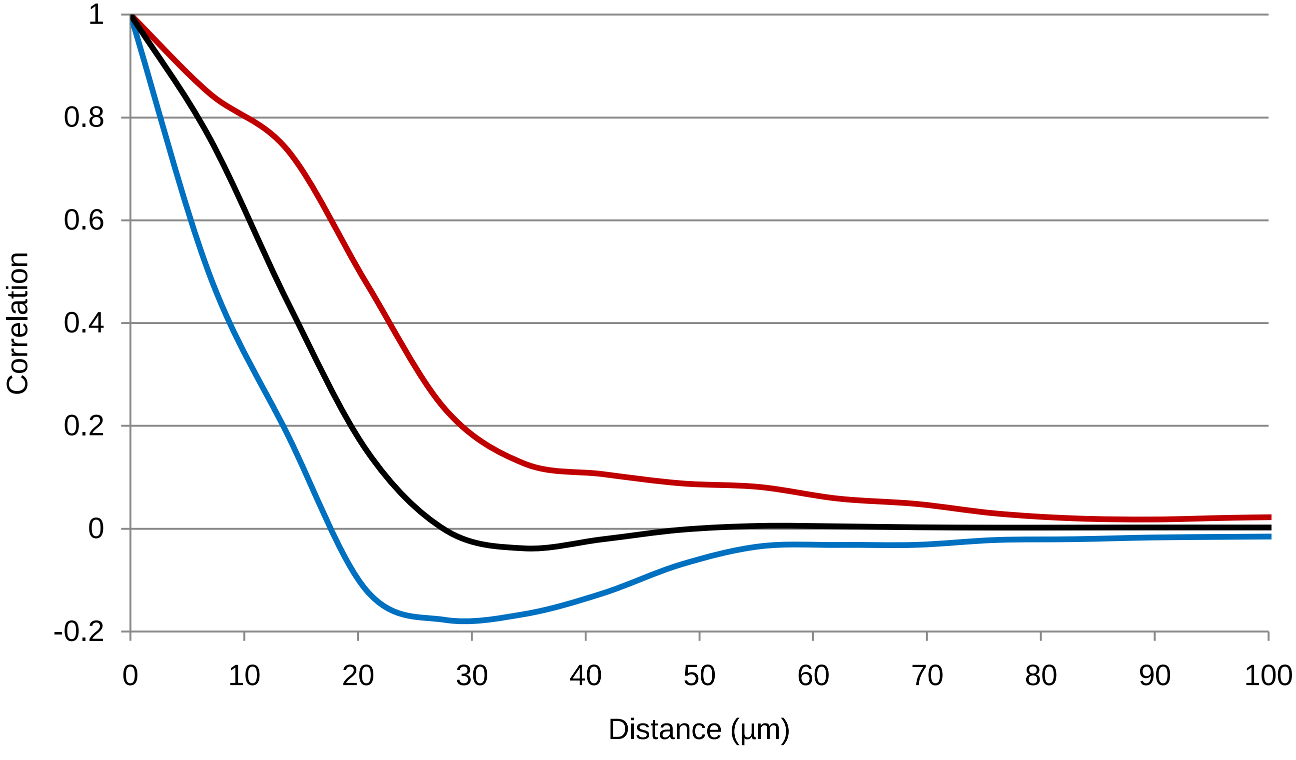




Autocorrelation

- Determination of a characteristic length





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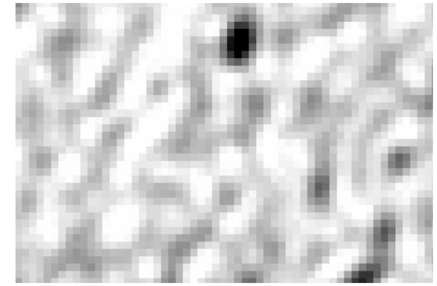

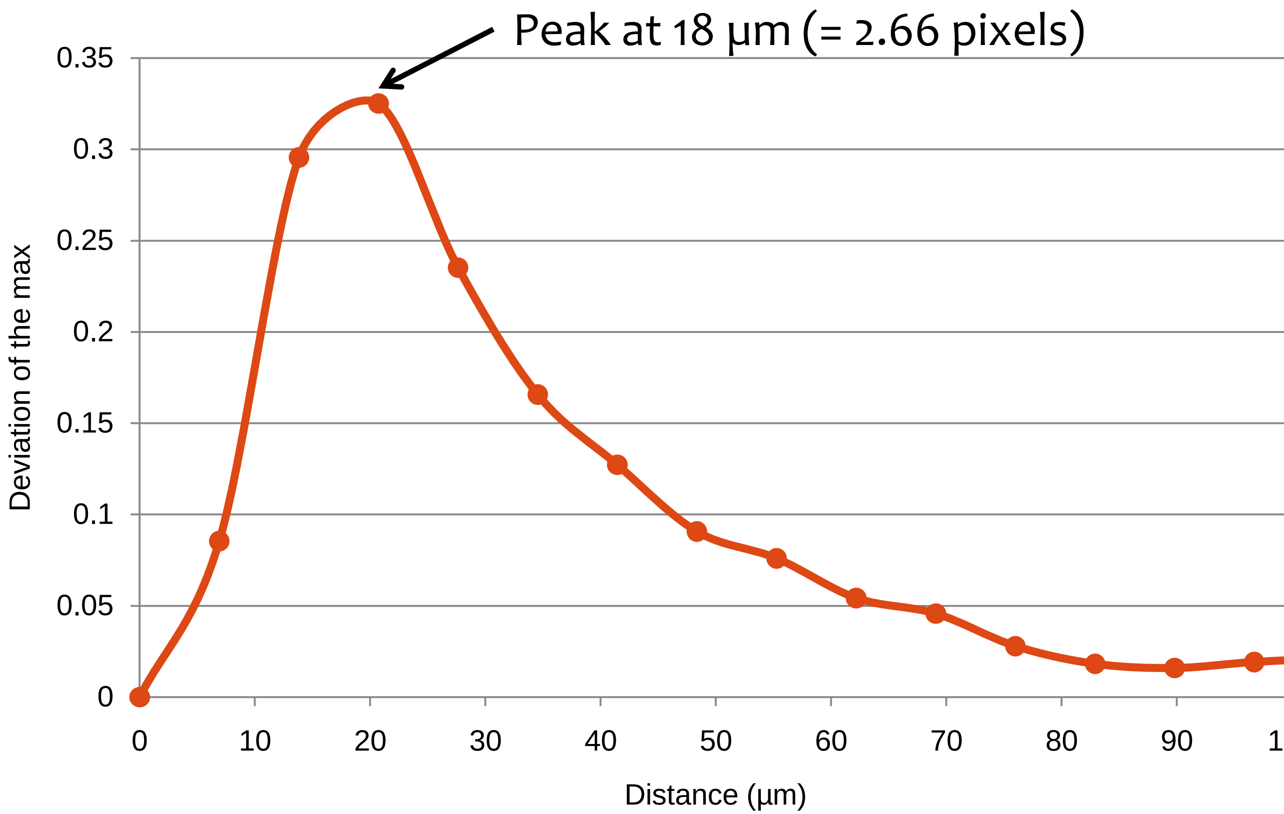

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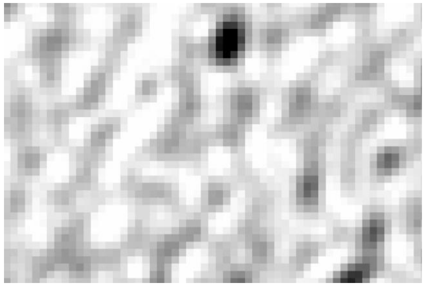
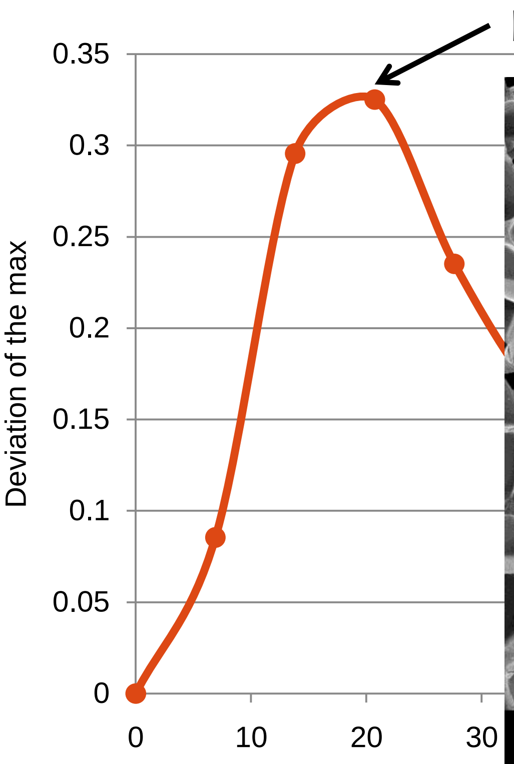
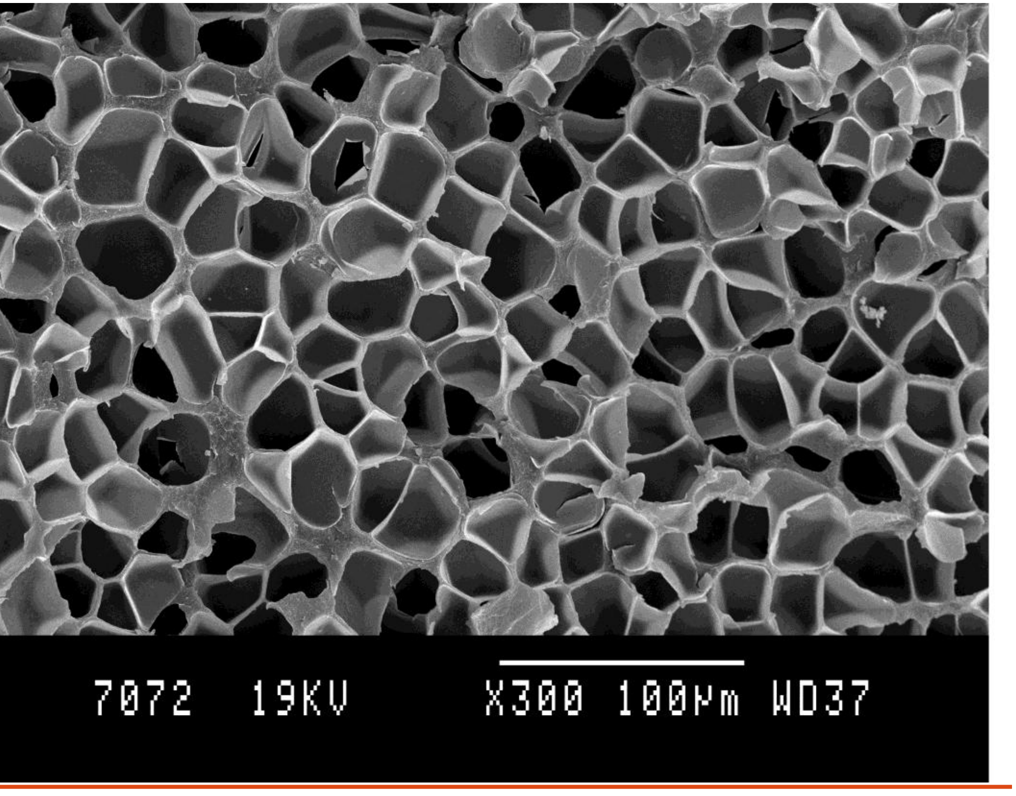
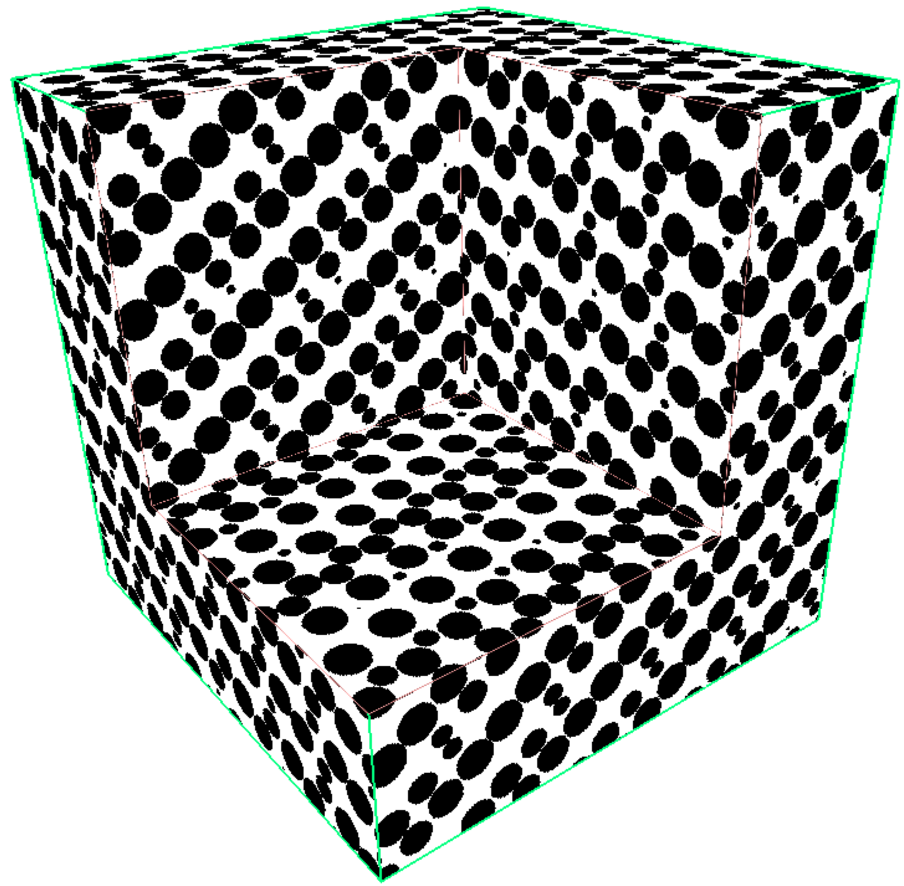






Rose diagram



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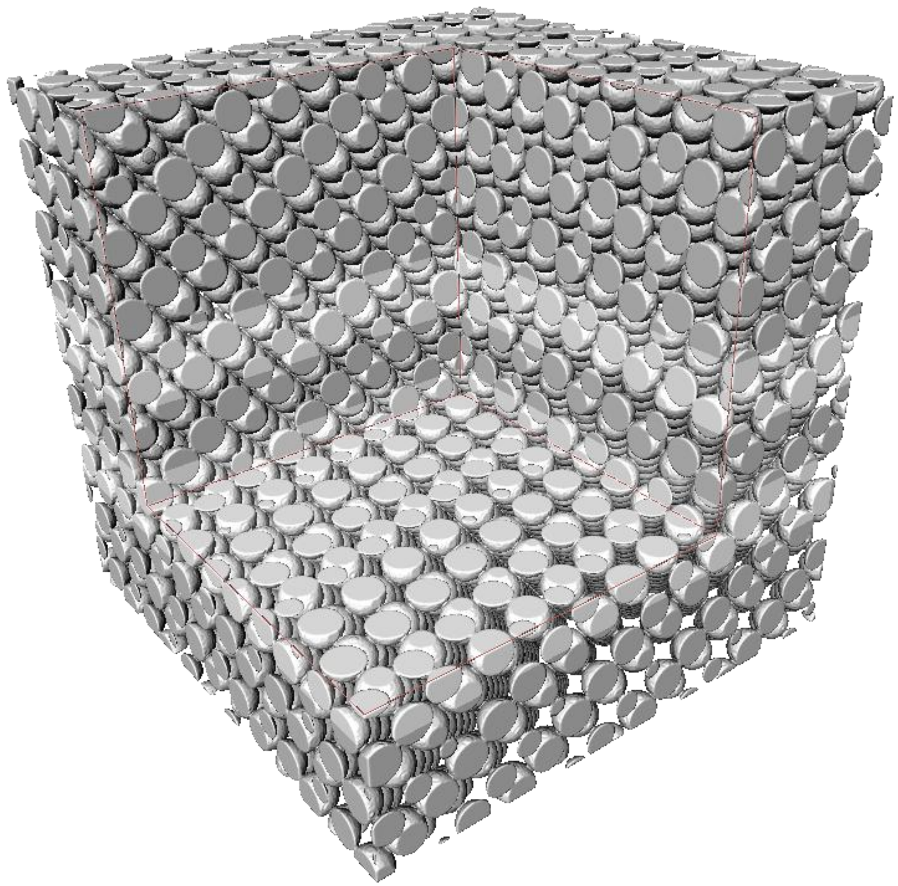

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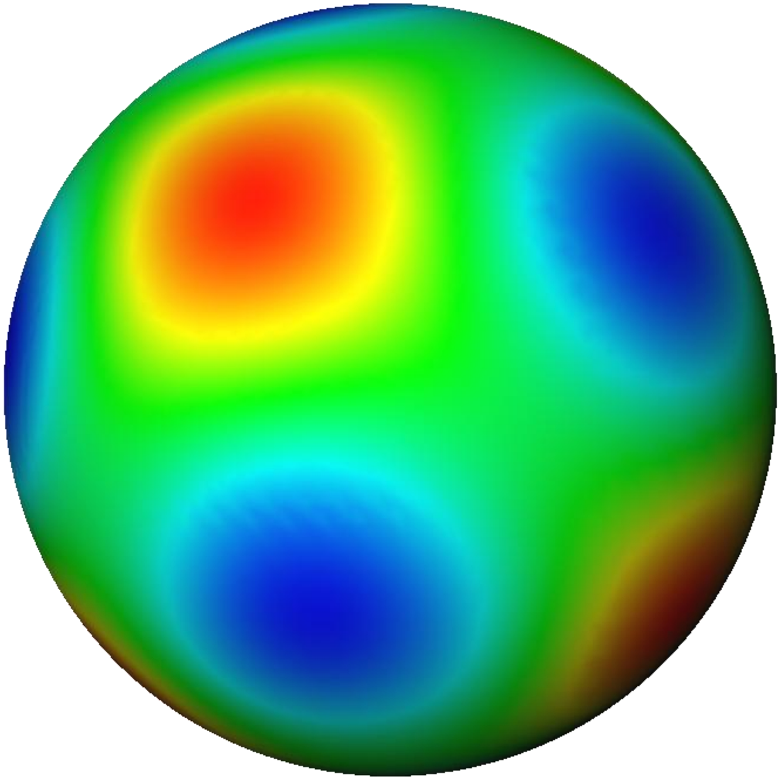
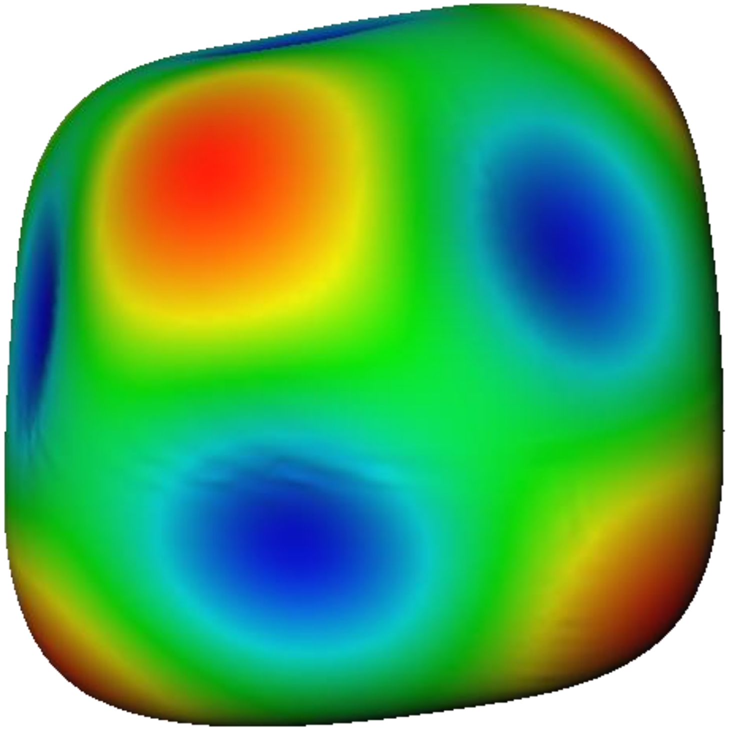
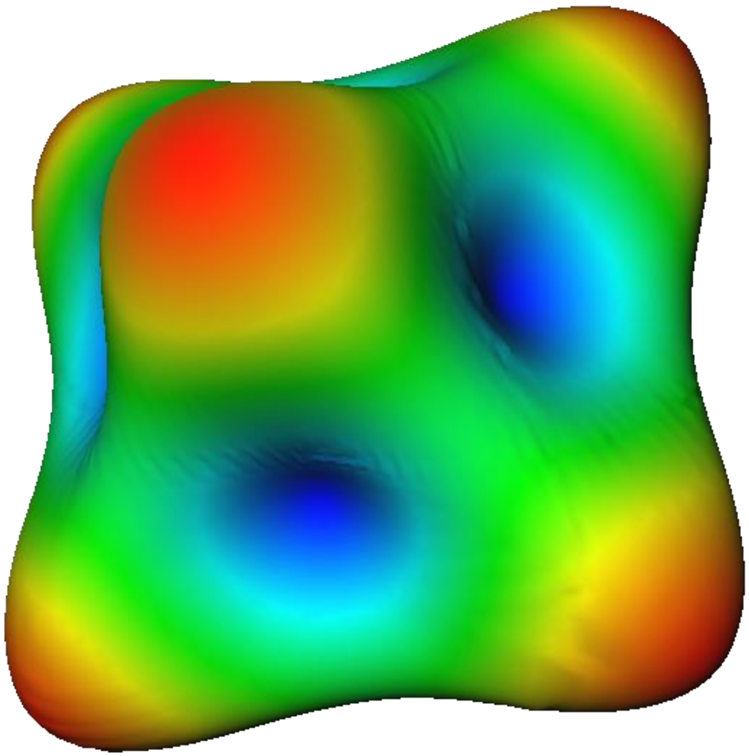


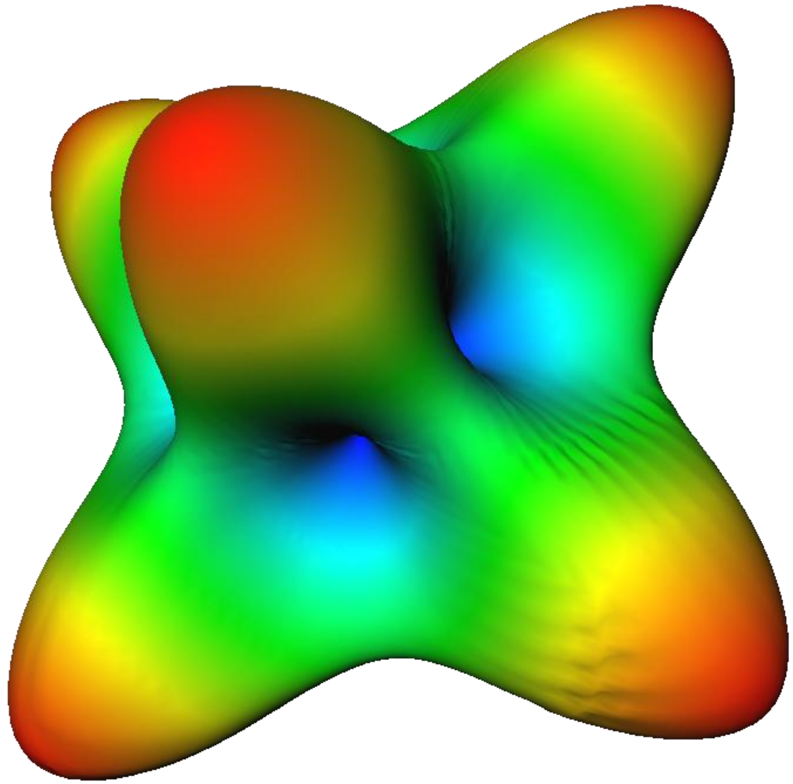
Rose diagram



Rose diagram



Rose diagram



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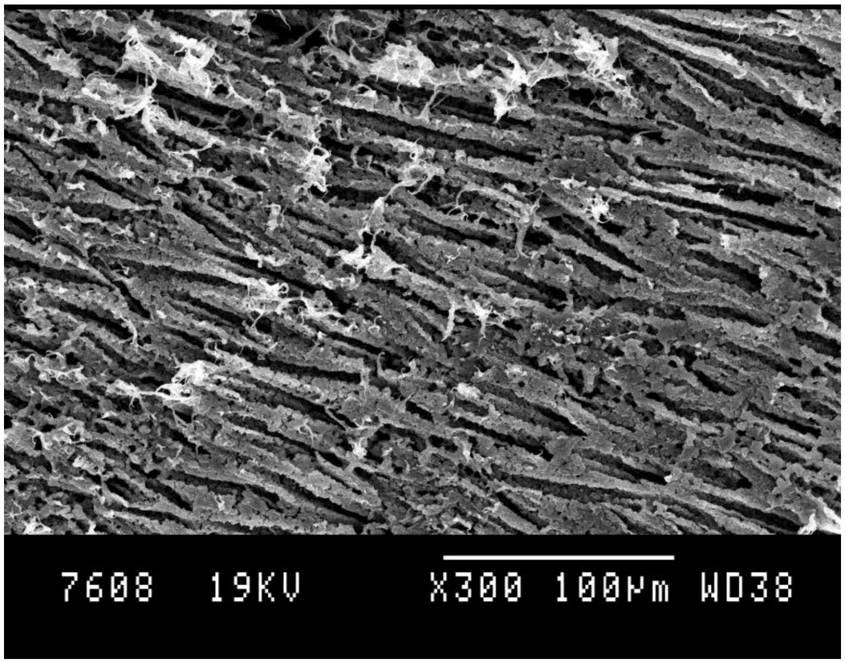

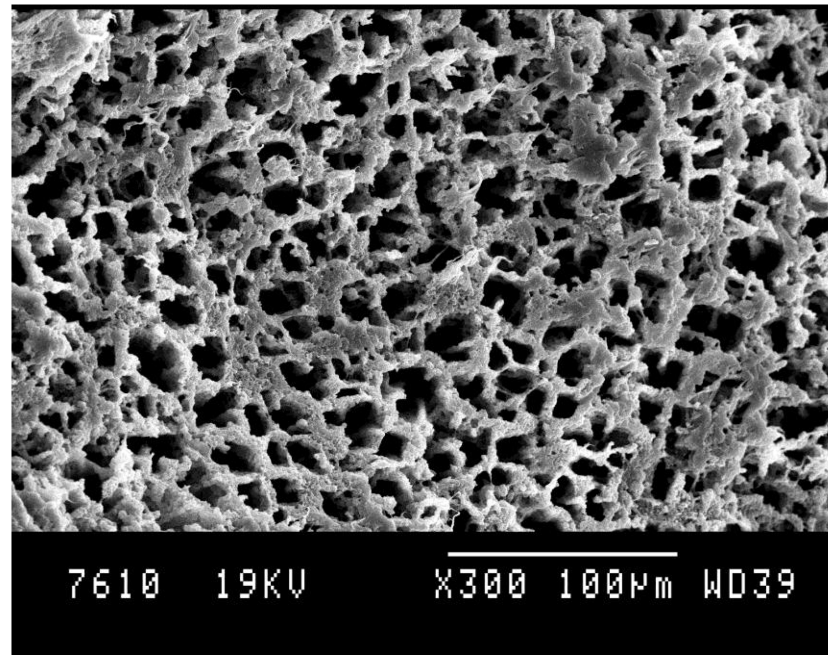

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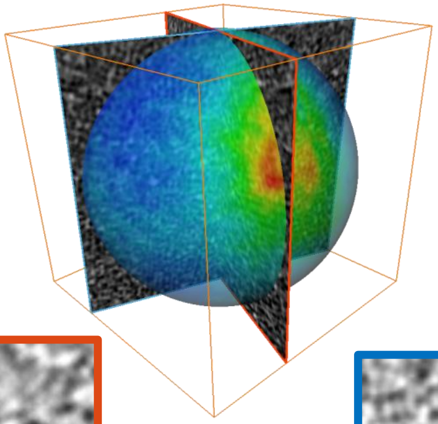
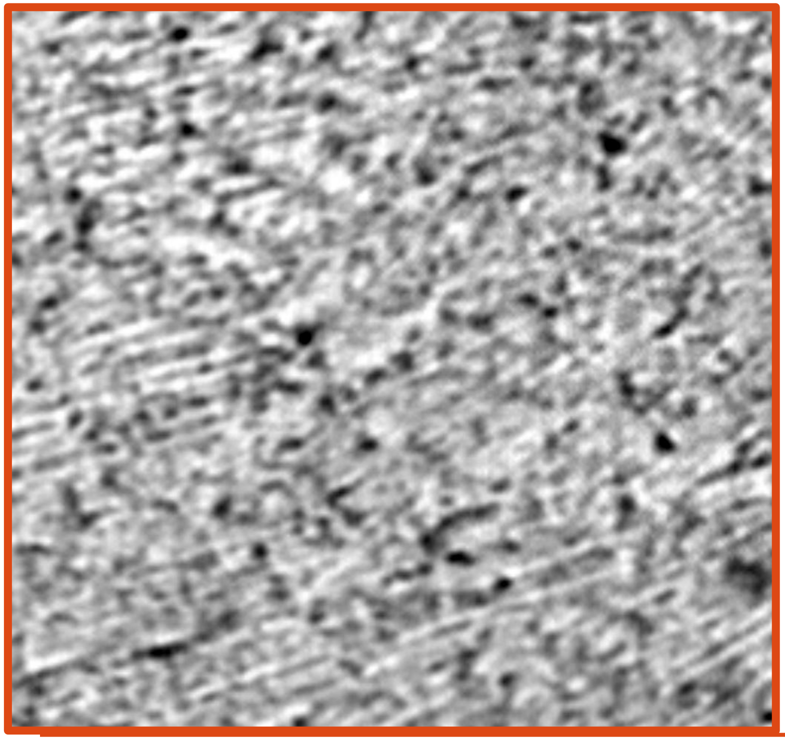
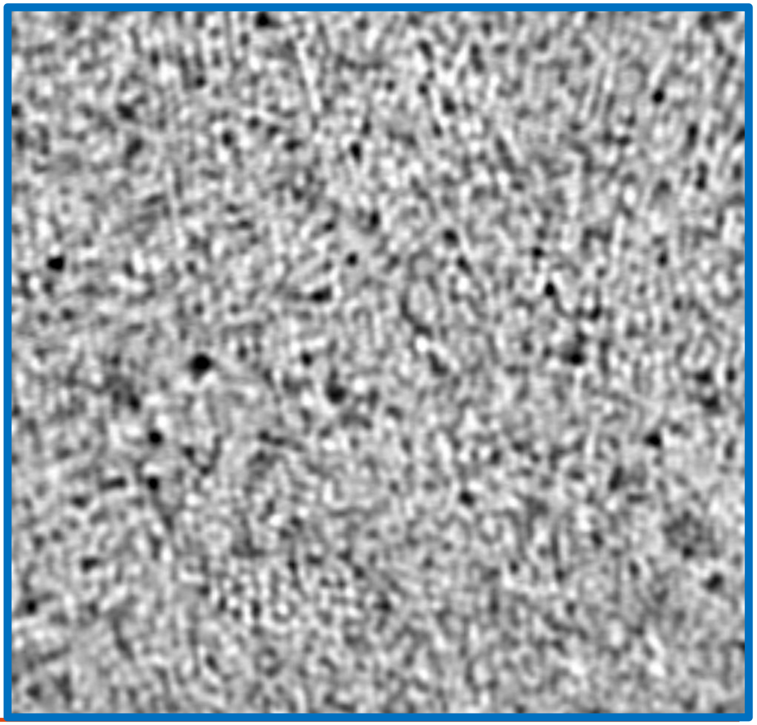




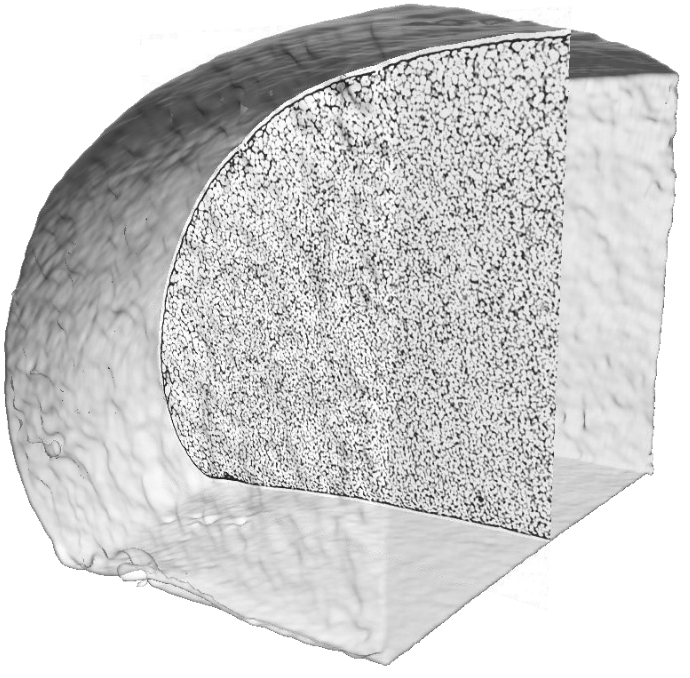


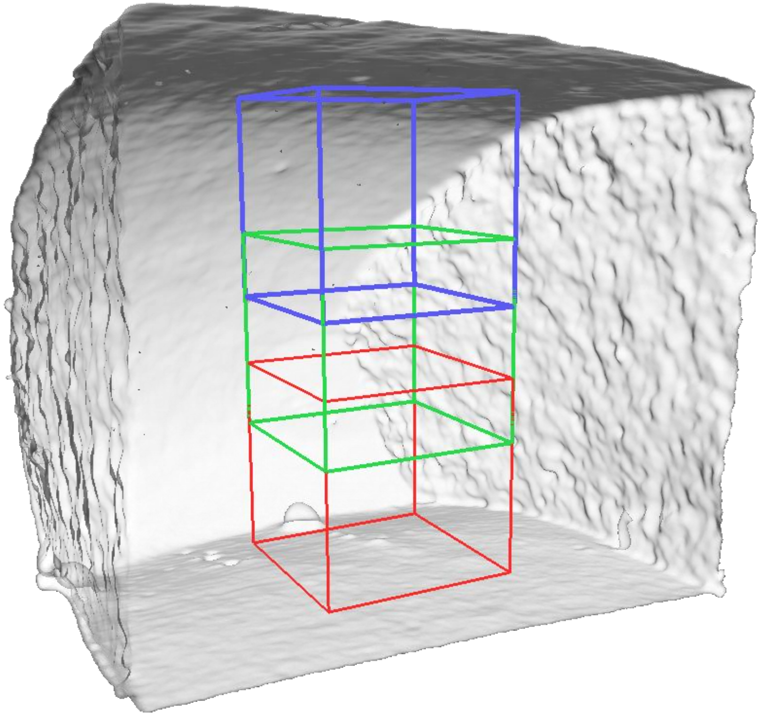
Method validation

- Representative Elementary Volume
- Effect of digitisation
- Effects of acquisition and reconstruction artefacts

Method validation

- Representative Elementary Volume





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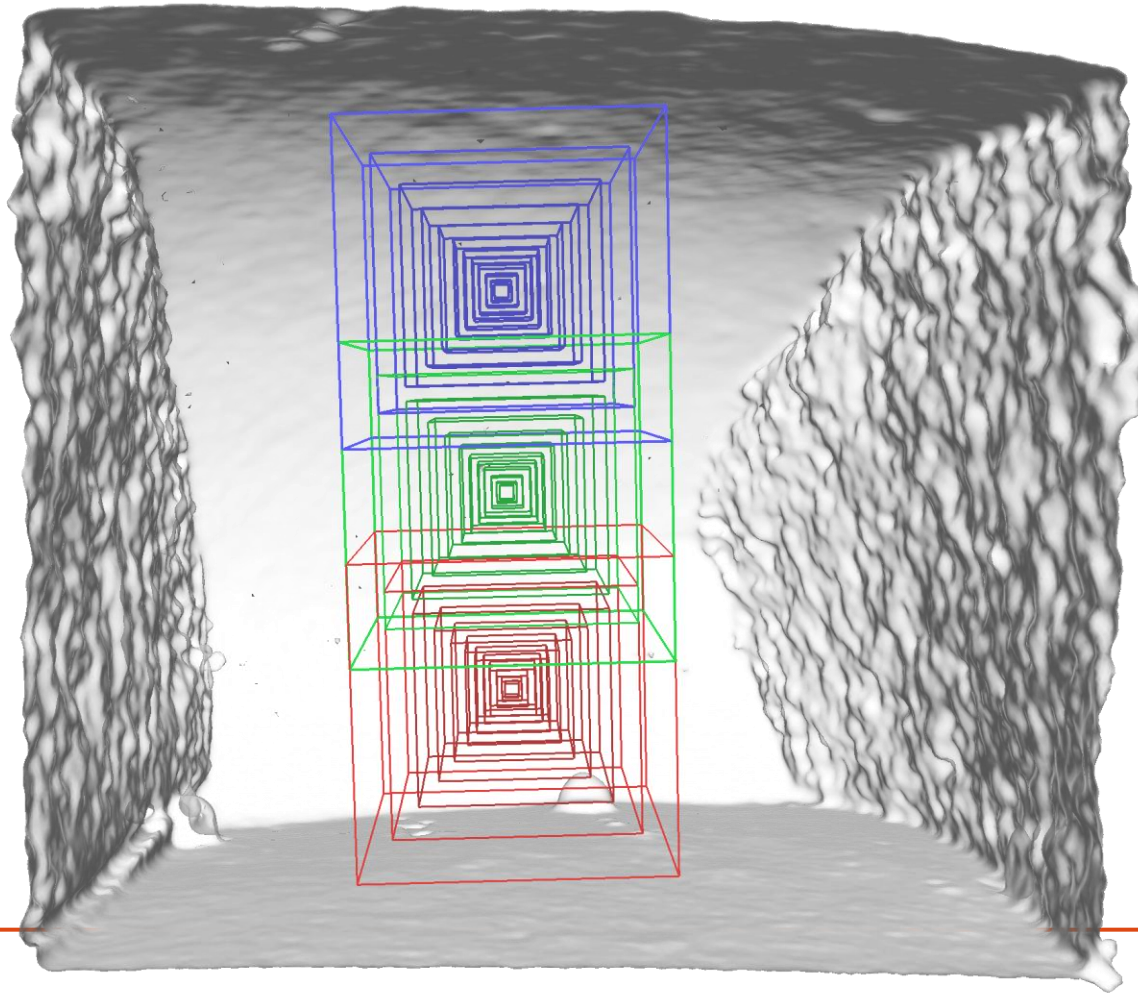

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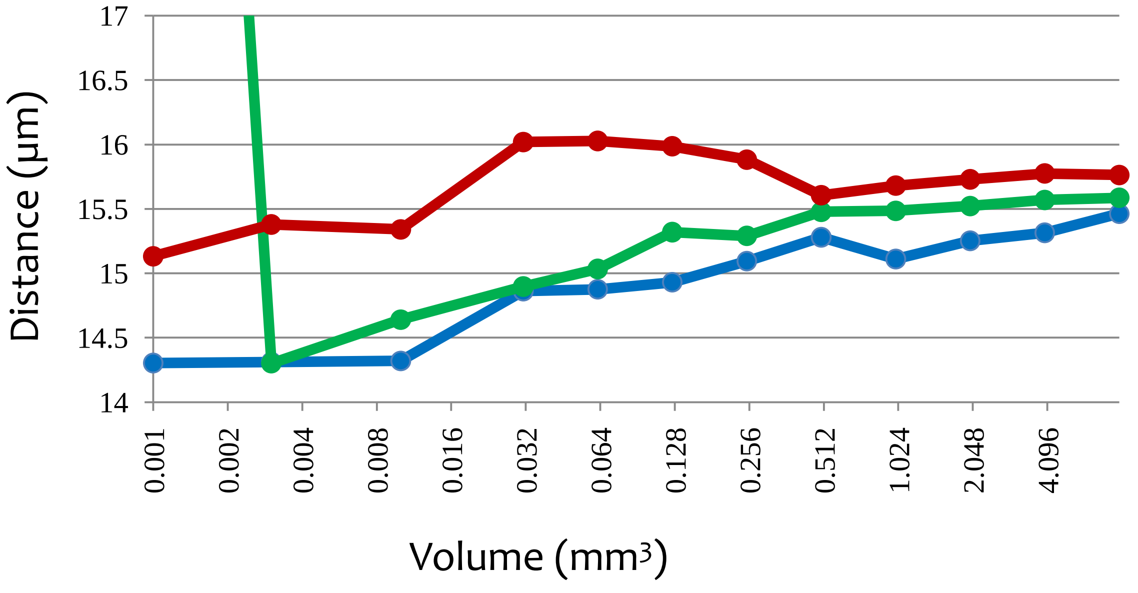

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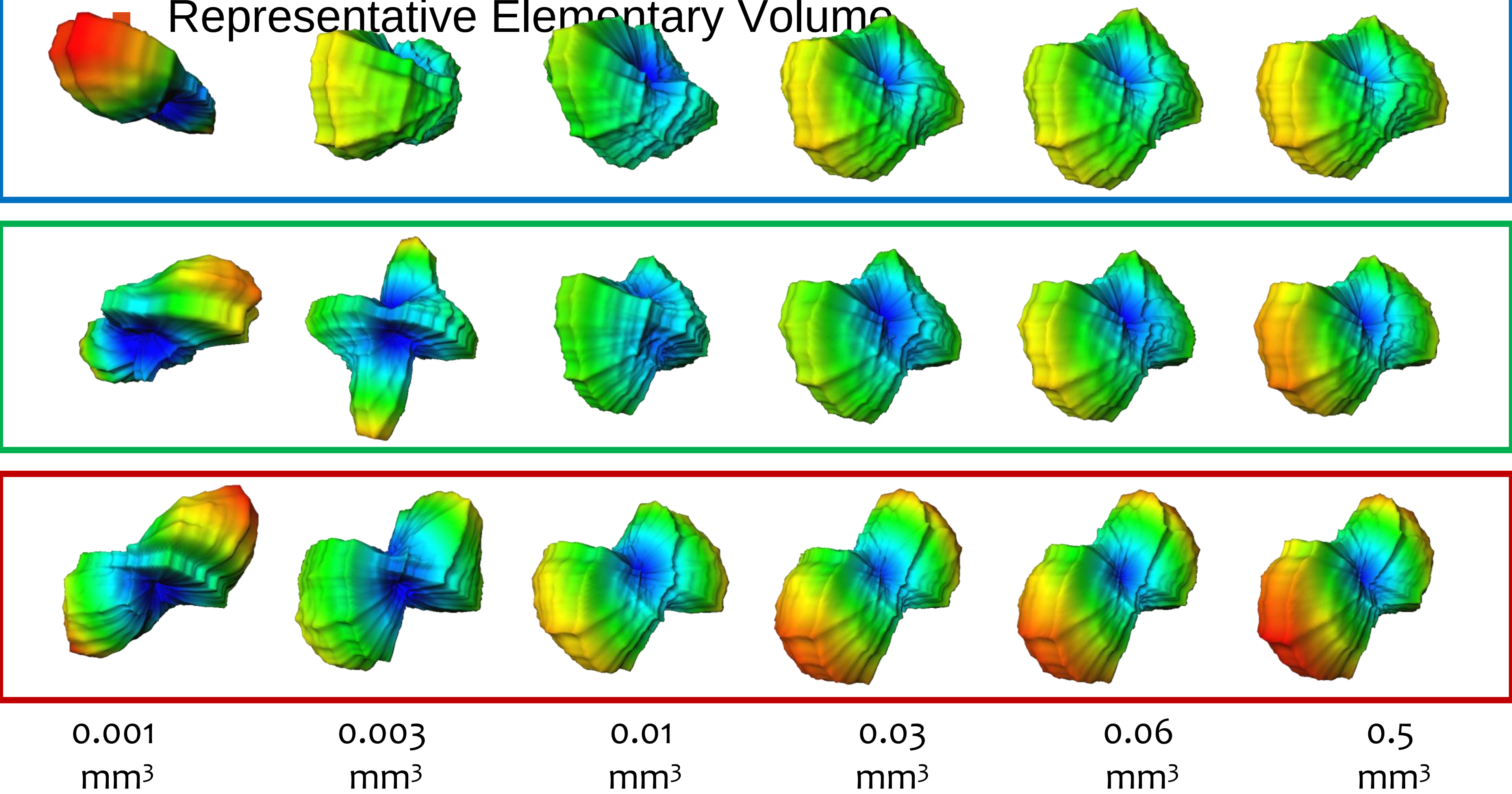

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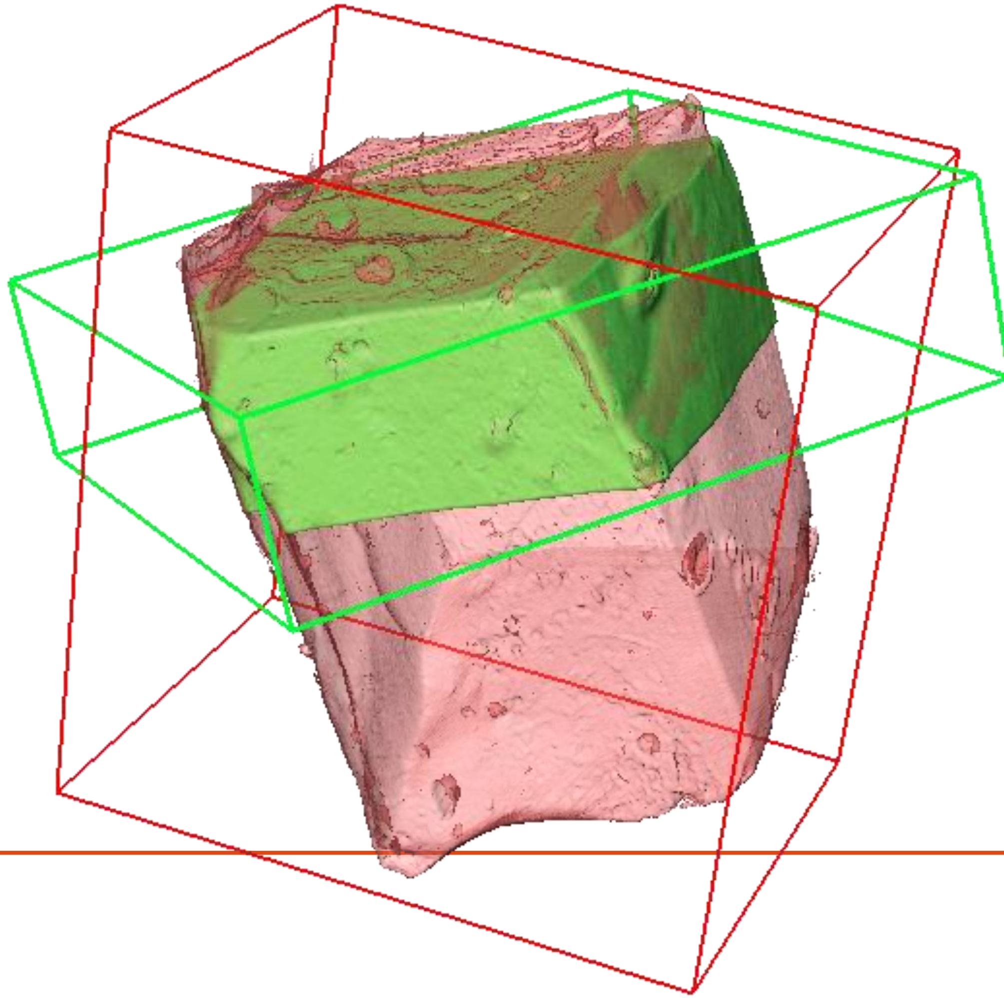
Method validation

- Effect of the imaging process (digitisation and artefacts)
  - Red volume → 4.49 µm/pixel, green → 6.74 µm/pixel



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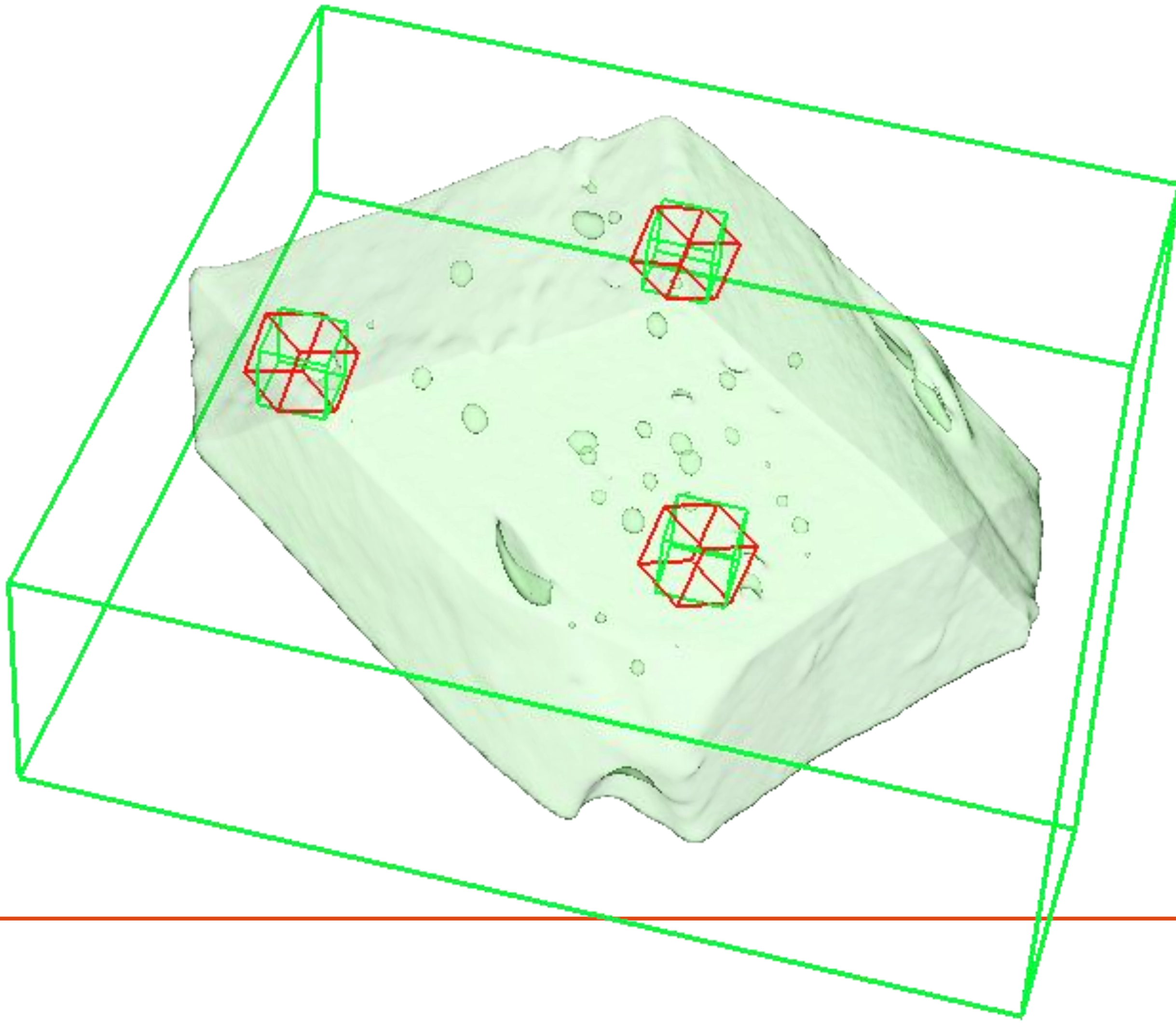

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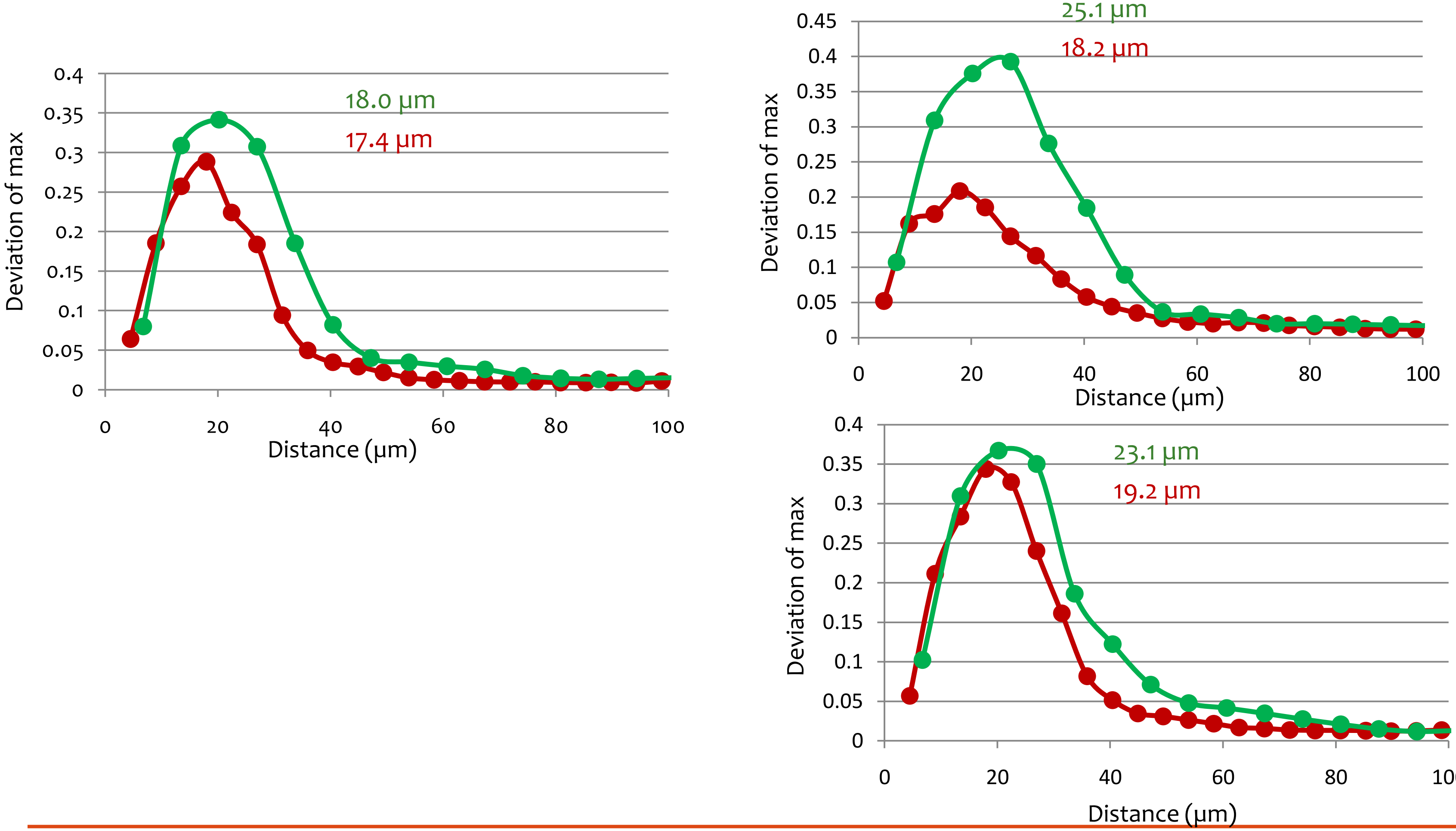

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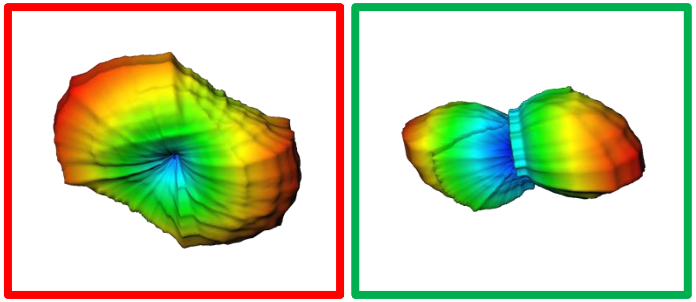

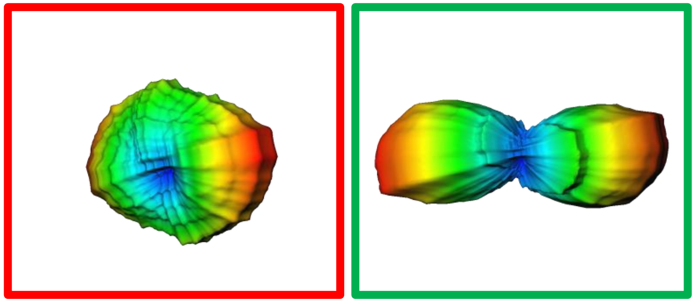

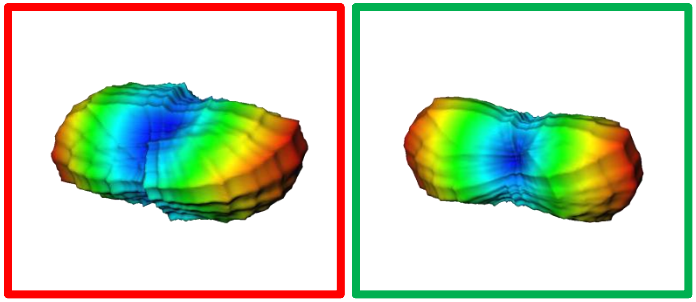

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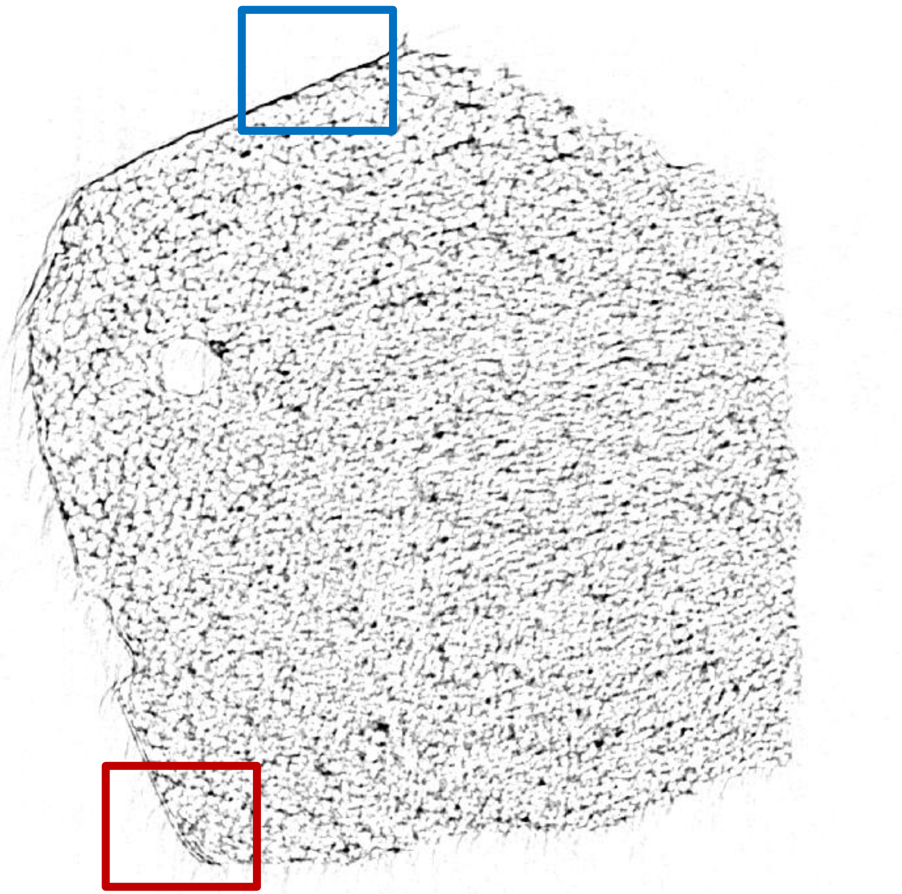

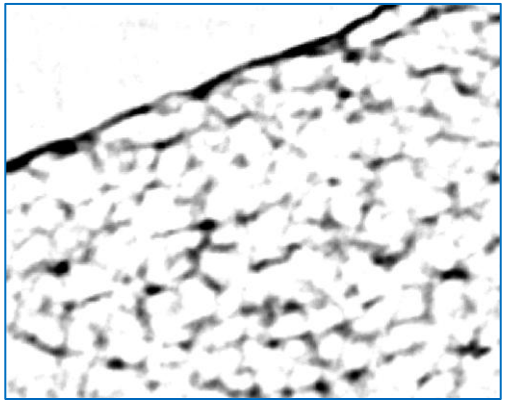

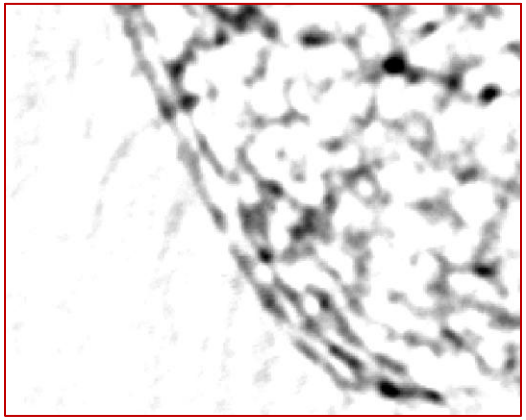

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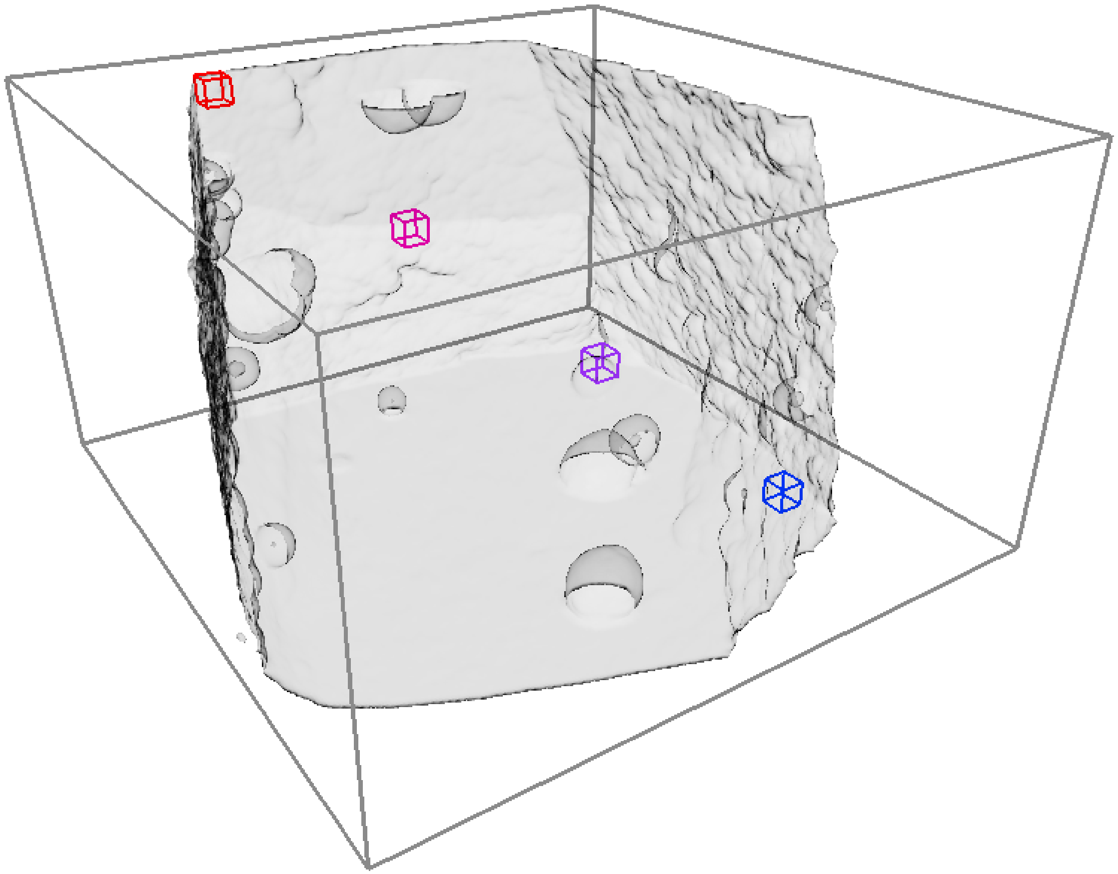
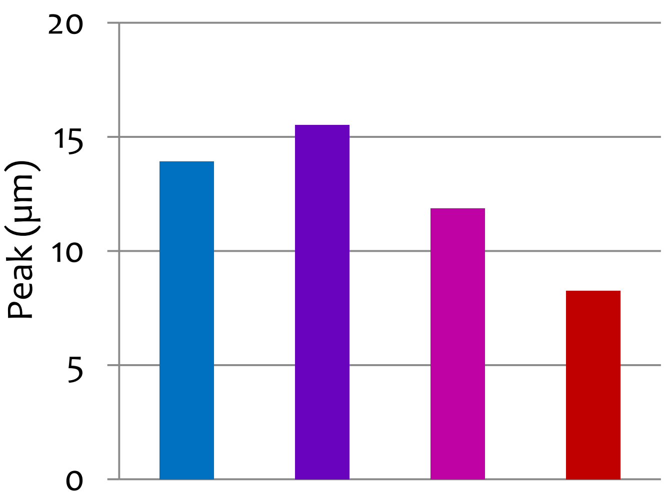
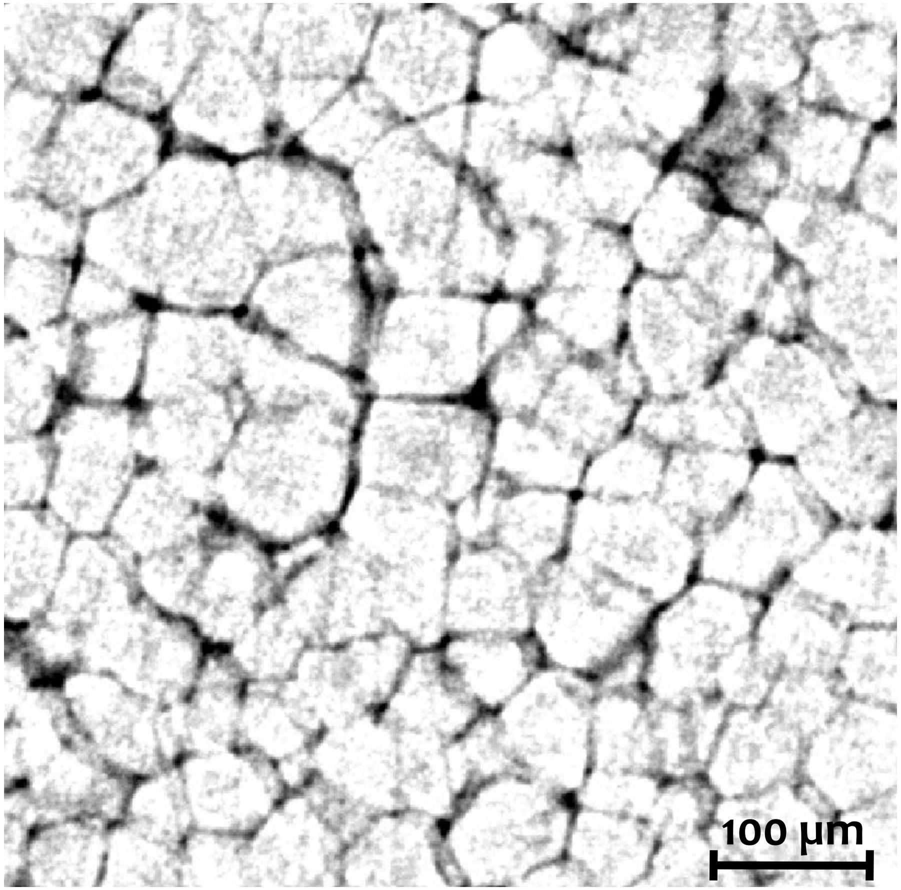




Conclusions

- Autocorrelation and the rose diagram can prove useful
- Difficulty in quantifying anisotropy
- Oversensitivity of tomographic artefacts
- Necessity for complementary methods

Perspectives



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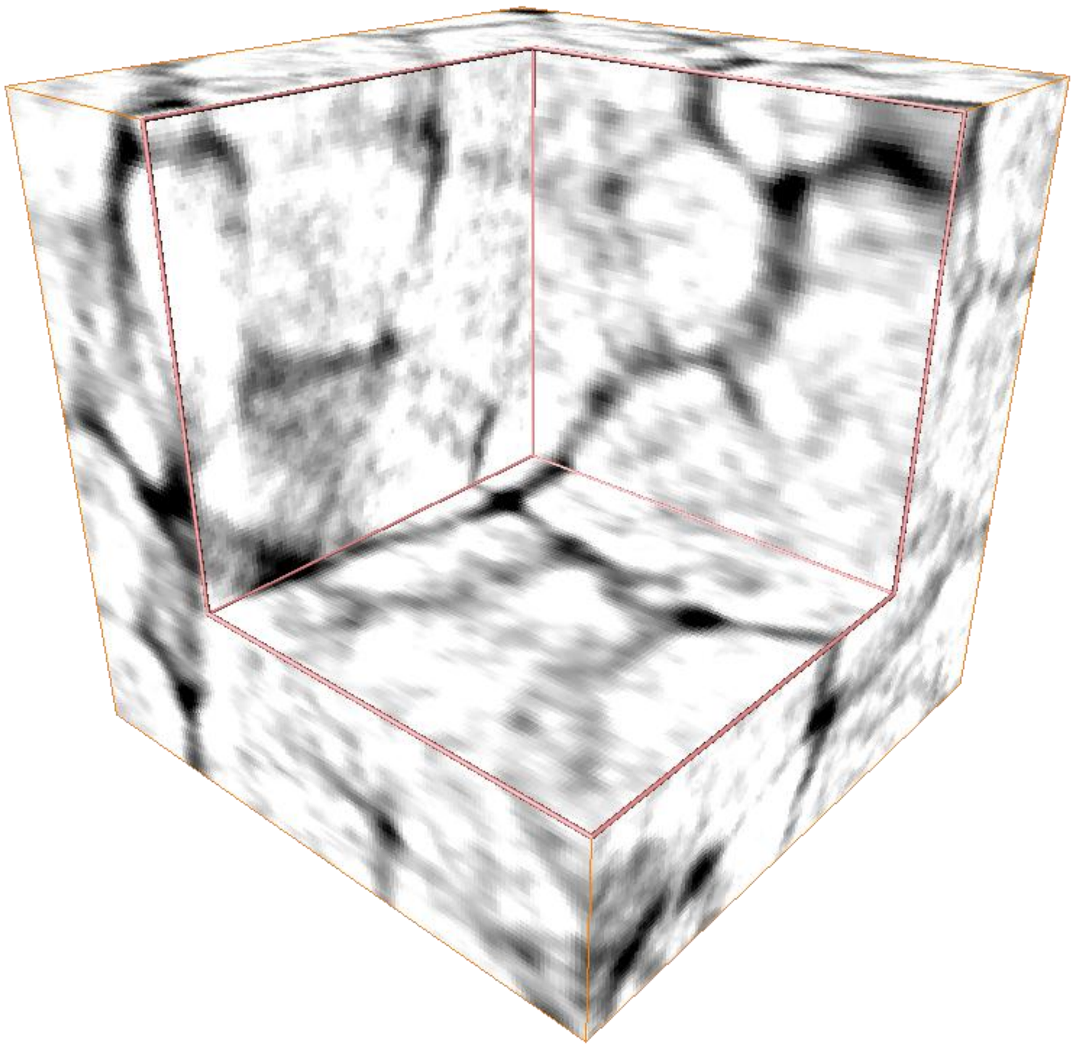

Perspectives