Technical University of Denmark

Towards automated 3D electrode microstructure characterisation from a data analysis perspective

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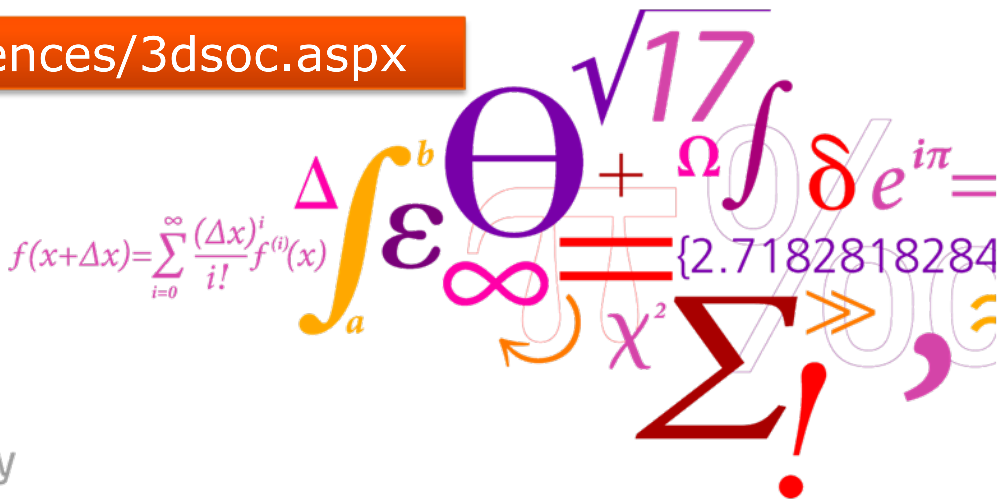
Towards automated 3D electrode microstructure characterisation from a data analysis perspective

and some examples of 2D electrode examinations at Risø-DTU

Jacob R. Bowen & **Peter Stanley Jørgensen**

http://www.risoe.dtu.dk/conferences/3dsoc.aspx



Strategic Electrochemistry
Research Center (SERC)

Risø DTU
National Laboratory for Sustainable Energy

Overview

Brief stereological motivation for 3D studies

From a series of images to 3D measurements:

- Image alignment
- Non-uniform illumination correction
- Segmentation
- Interface characterization
- Network characterization
- Conclusions

Brief 2D SEM based examples

2D microstructural measurements

TABLE 1.1

List of basic symbols and their definitions

Symbol	Dimensions*	Definition
P		Number of point elements, or test points
P_P		Point fraction. Number of points (in areal features) per test point
P_L	mm^{-1}	Number of point intersections per unit length of test line
P_A	mm^{-2}	Number of points per unit test area
P_V	mm^{-3}	Number of points per unit test volume
L	mm	Length of lineal elements, or test line length
L_L	mm/mm	Lineal fraction. Length of lineal intercepts per unit length of test line
L_A	mm/mm^2	Length of lineal elements per unit test area
L_V	mm/mm^3	Length of lineal elements per unit test volume
A	mm^2	Planar area of intercepted features, or test area
S	mm^2	Surface or interface area (not necessarily planar)
A_A	mm^2/mm^2	Area fraction. Area of intercepted features per unit test area
S_V	mm^2/mm^3	Surface area per unit test volume
V	mm^3	Volume of three-dimensional features, or test volume
V_V	mm^3/mm^3	Volume fraction. Volume of features per unit test volume
N		Number of features (as opposed to points)
N_L	mm^{-1}	Number of interceptions of features per unit length of test line
N_A	mm^{-2}	Number of interceptions of features per unit test area
N_V	mm^{-3}	Number of features per unit test volume
$\bar{L}$	mm	Average lineal intercept, L_L/N_L
$\bar{A}$	mm^2	Average areal intercept, A_A/N_A
$\bar{S}$	mm^2	Average surface area, S_V/N_V
$\bar{V}$	mm^3	Average volume, V_V/N_V

*Arbitrarily shown in millimeters.

TABLE 2.1

Relationship of measured (○) *to calculated* (□) *quantities*

Microstructural feature	Dimensions of symbols (arbitrarily expressed in terms of millimeters) mm^0	mm^{-1}	mm^{-2}	mm^{-3}
Points	P_P	P_L	P_A	P_V
Lines	L_L	L_A	L_V	—
Surfaces	A_A	S_V	—	—
Volumes	V_V	—	—	—

$$V_V = A_A = L_L = P_P \qquad mm^0$$

$$S_V = (4/\pi)L_A = 2P_L \qquad mm^{-1}$$

$$L_V = 2P_A \qquad mm^{-2}$$

$$P_V = \tfrac{1}{2}L_V S_V = 2P_A P_L \qquad mm^{-3}$$

TPB density

BUT

No information on percolation

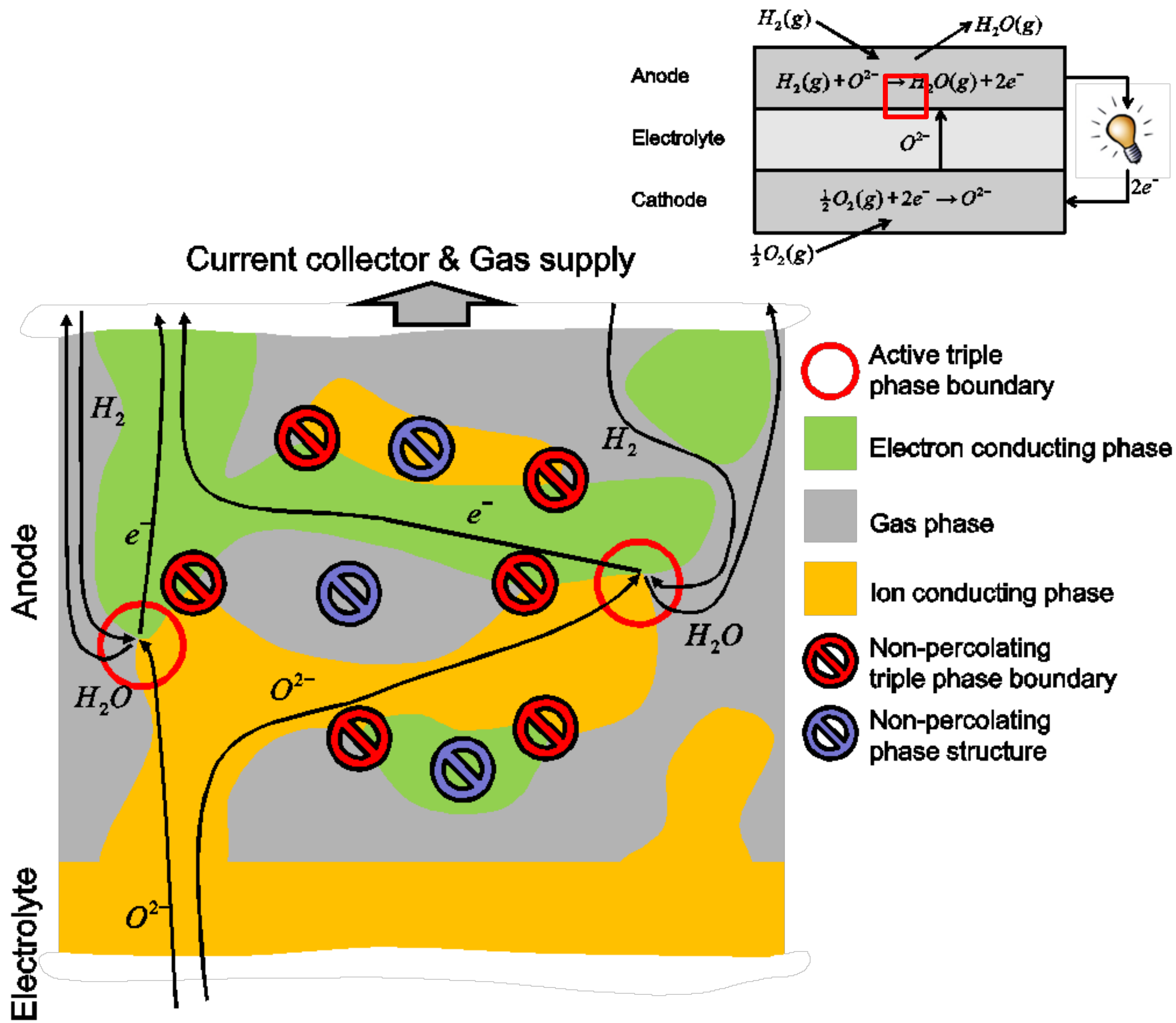
Applicable to any type of linear system in space, provided that all angles of intersection between lines and test planes are equally possible.

Ervin E. Underwood, Quantitative Stereology, Addison-Wesley, 1970

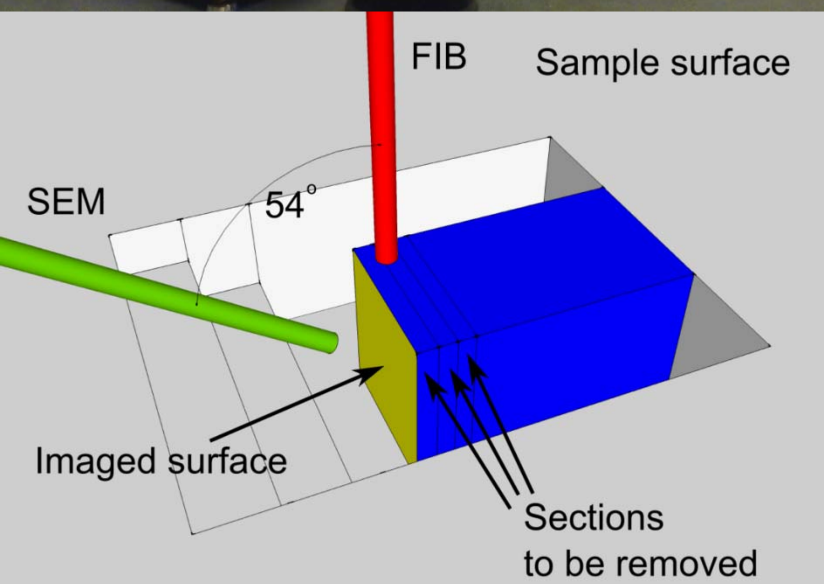
The importance of microstructure

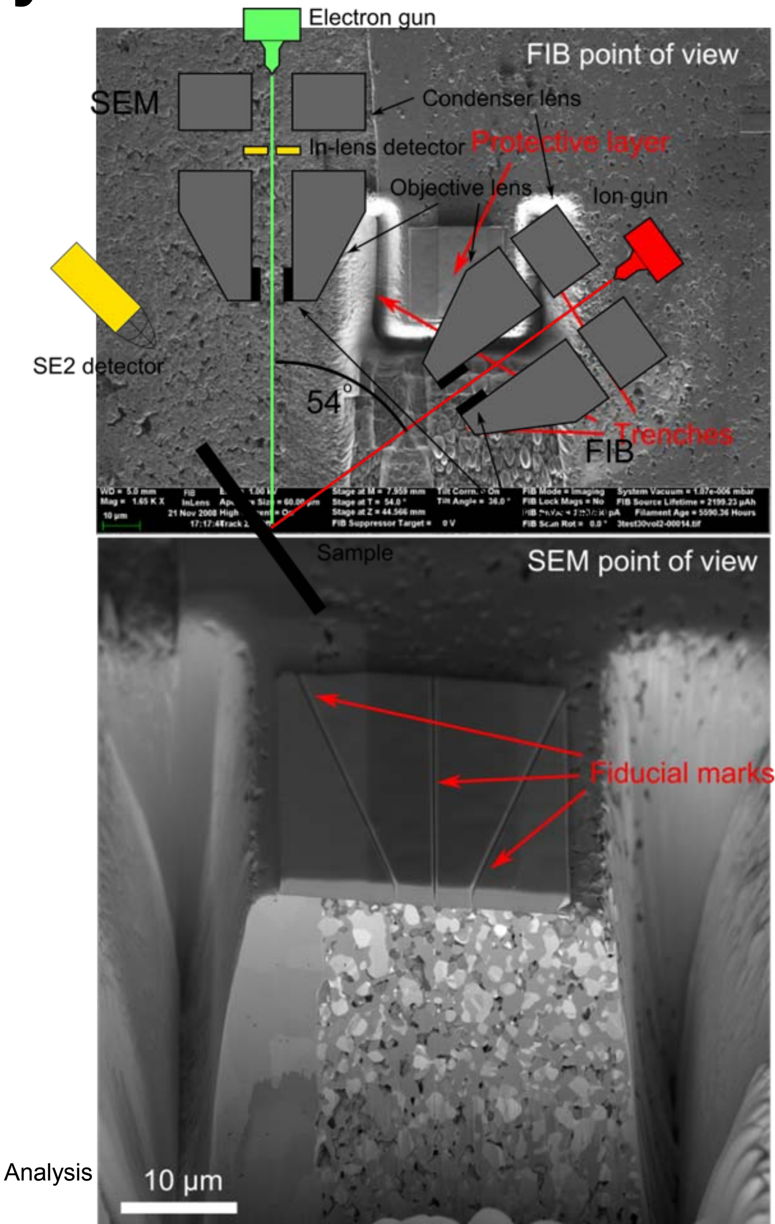
We would like to quantify:

- The linear density of triple phase boundaries.
- How difficult it is to get to them.
- Are the TPBs active?
- How well is the material used?
- Anything else that can help us characterize the microstructure.



Focused ion beam tomography





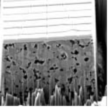
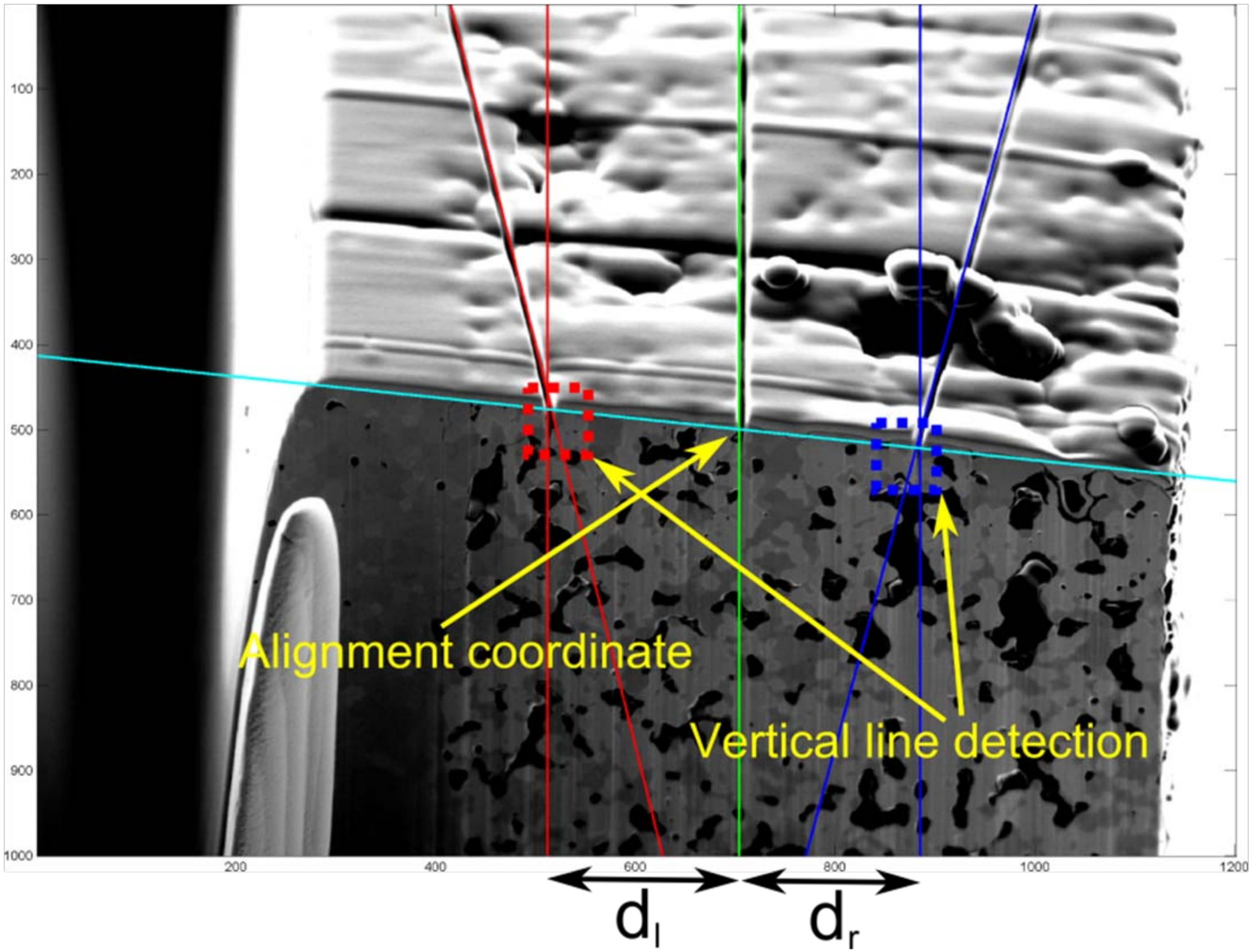


Image alignment



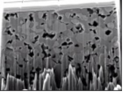
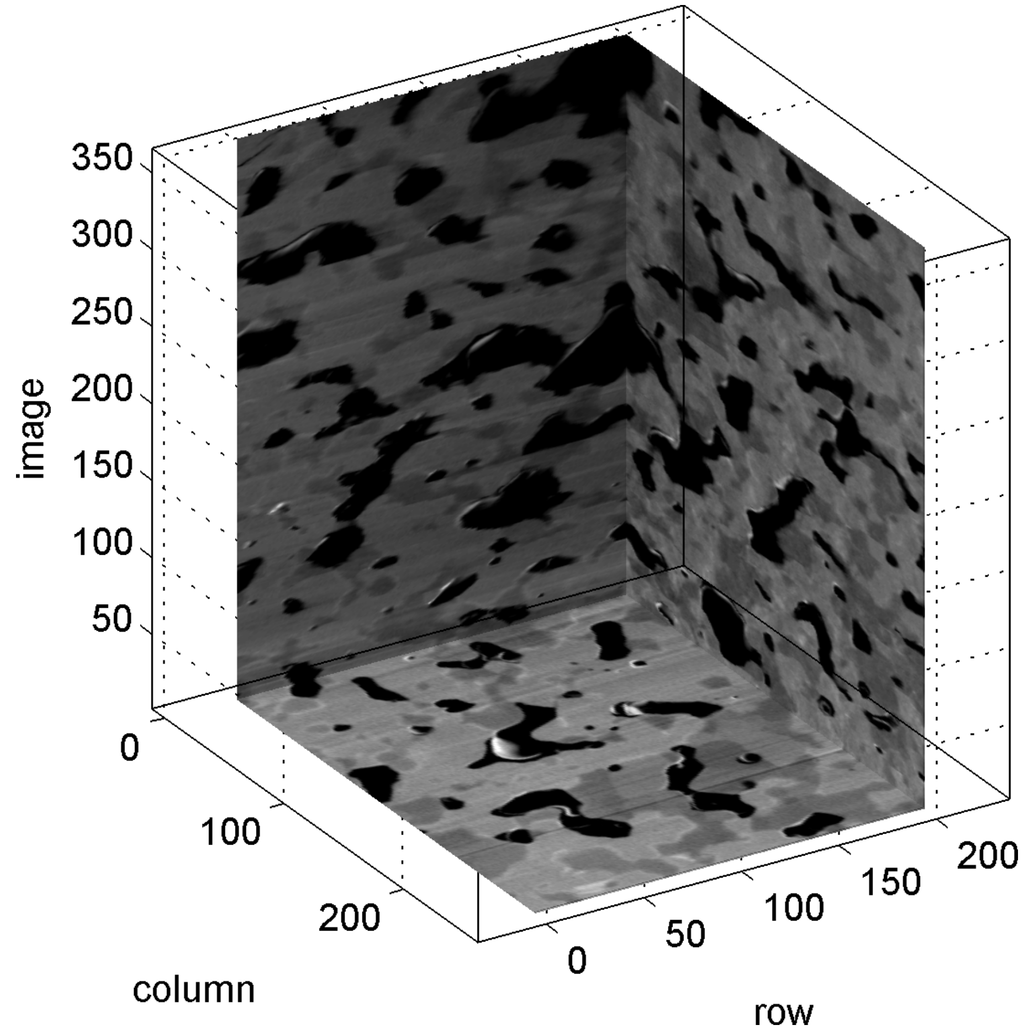
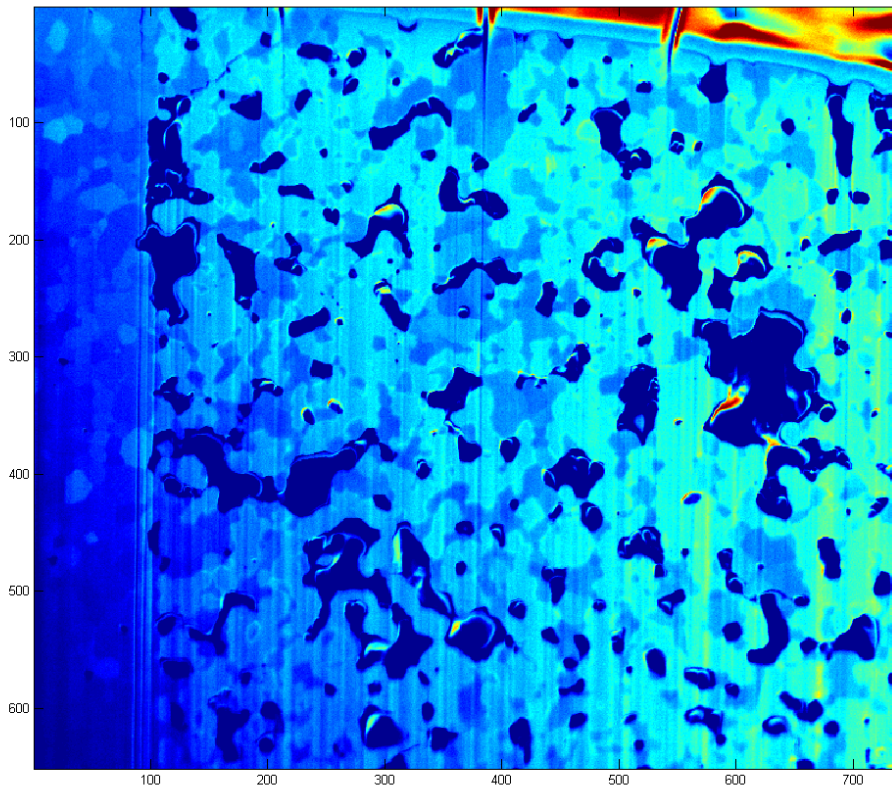


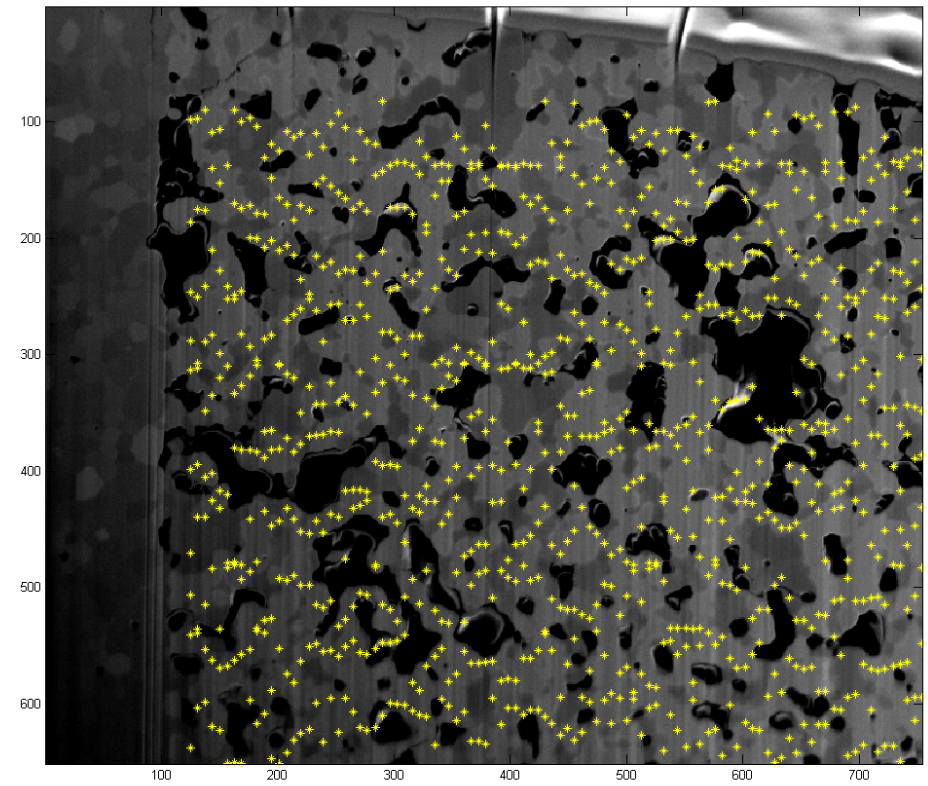
Image alignment after

After alignment and cropping a cuboid 3D image is extracted for further processing.

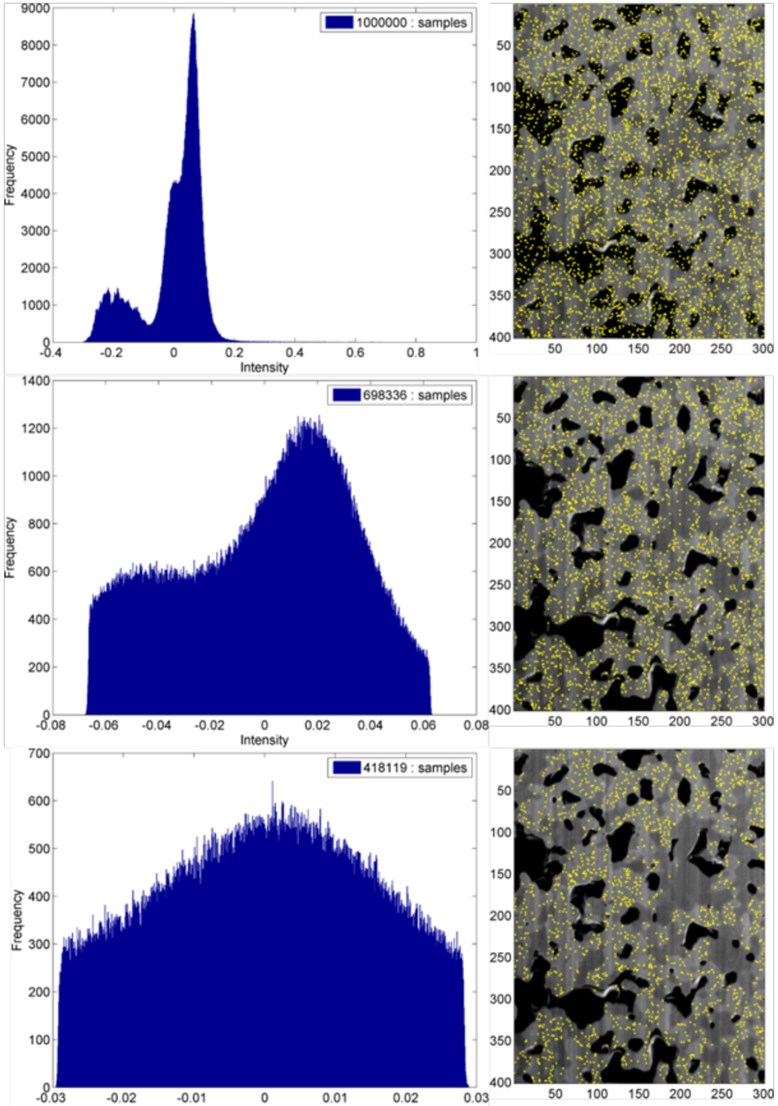


The non-uniform illumination problem





Iterative illumination correction

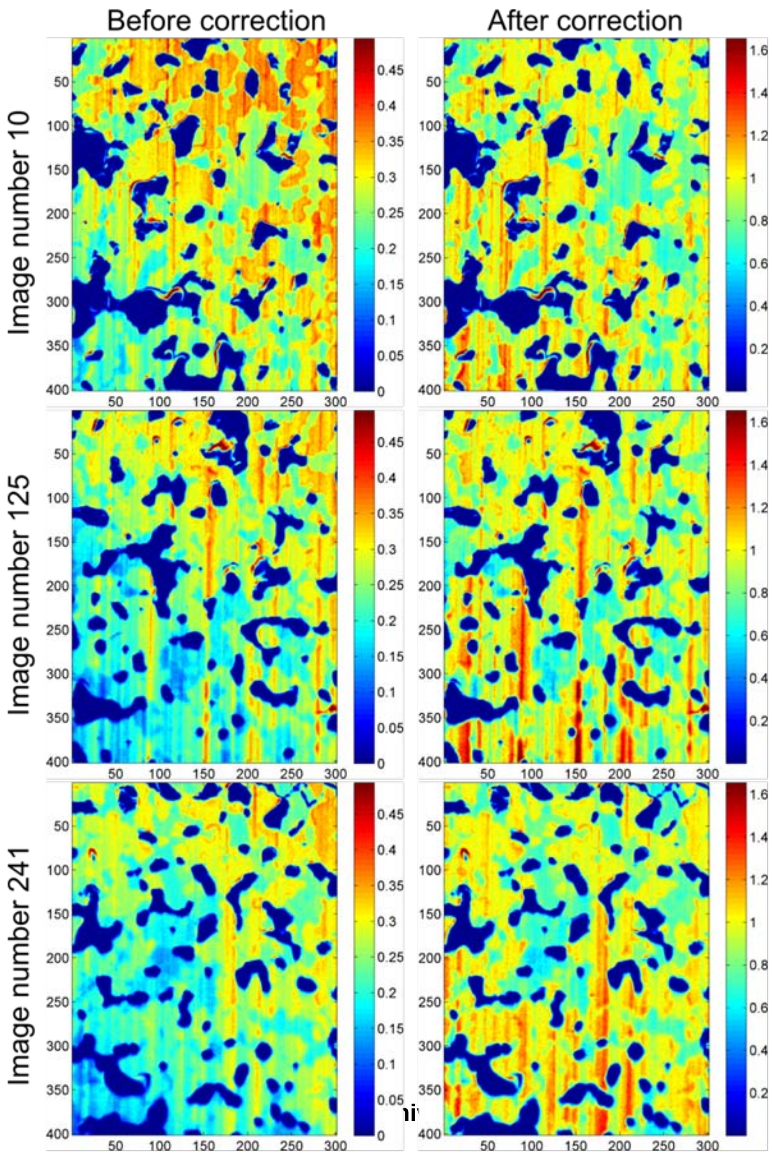


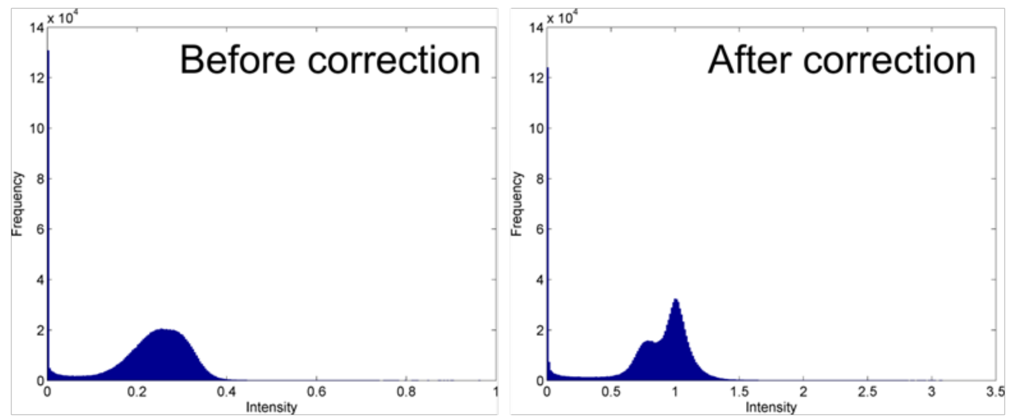
Pick a large number of voxels randomly in 3D.

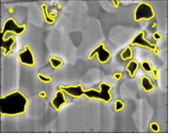
1. Fit a rigid hypersurface to the x,y,z + intensity data.
2. Remove the samples with the largest fit error.
3. Proceed from 2 with the reduced voxel set.

Proceed until a significant portion of the original samples have been removed.

Correction example







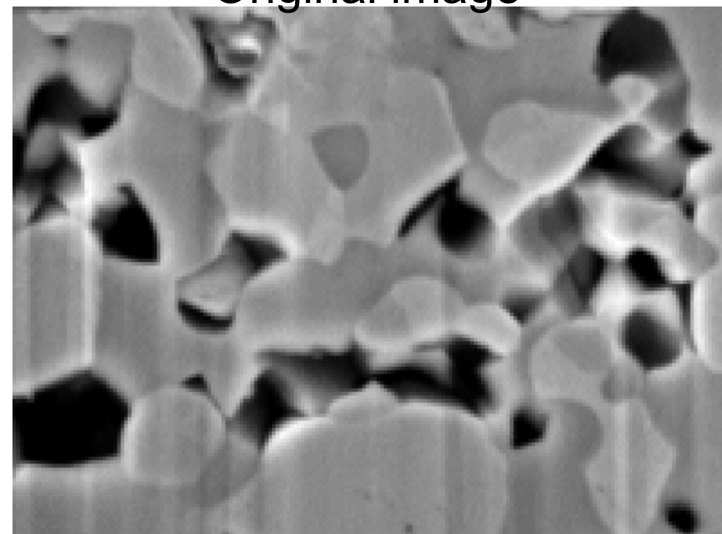
Segmentation strategy

- Segment two phases with level set segmentation
- Third remaining phase is then the remainder
- Parameterise the third phase as a level set

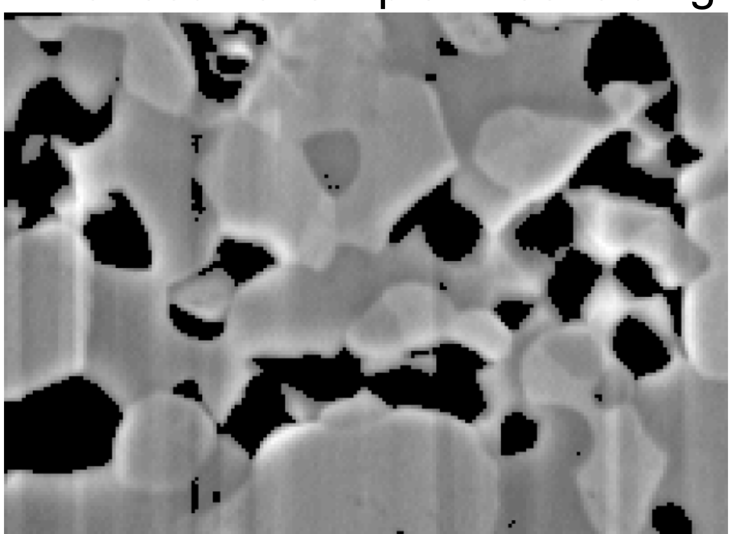
The segmentation problem

- Determine which part of the image corresponds to which phase.
- Image: SE2 detector (10 kV), Ni/YSZ anode. Size is 8.4 x 6.3 µm.

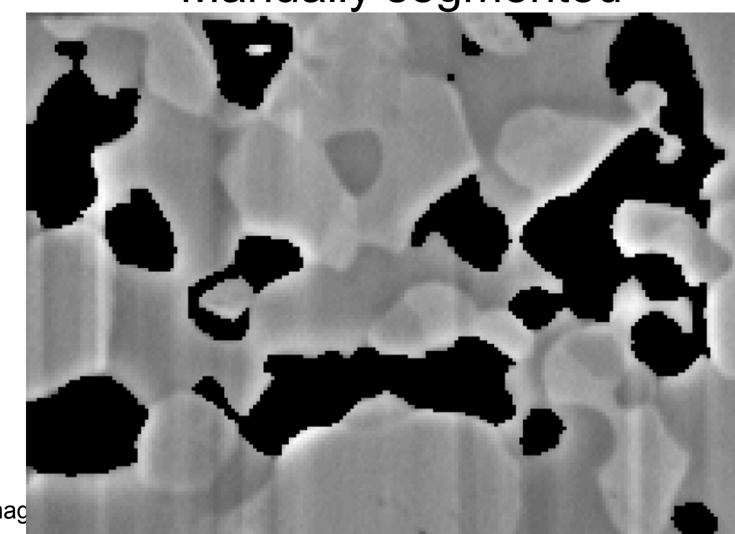
Original image



The result of simple thresholding



Manually segmented

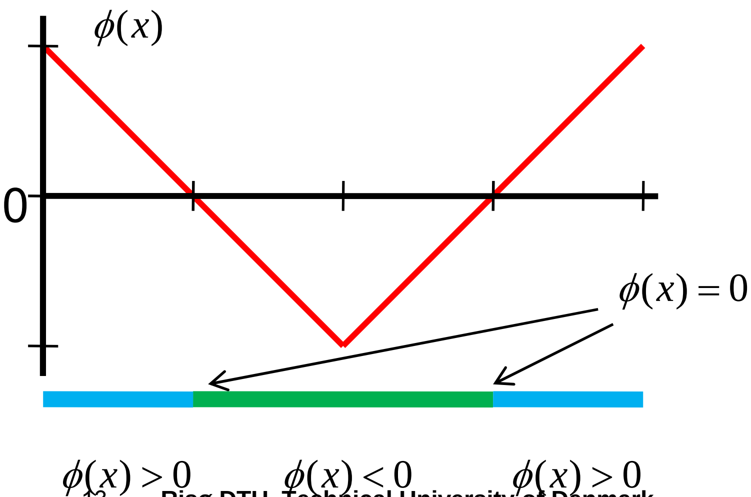


Implicit interfaces

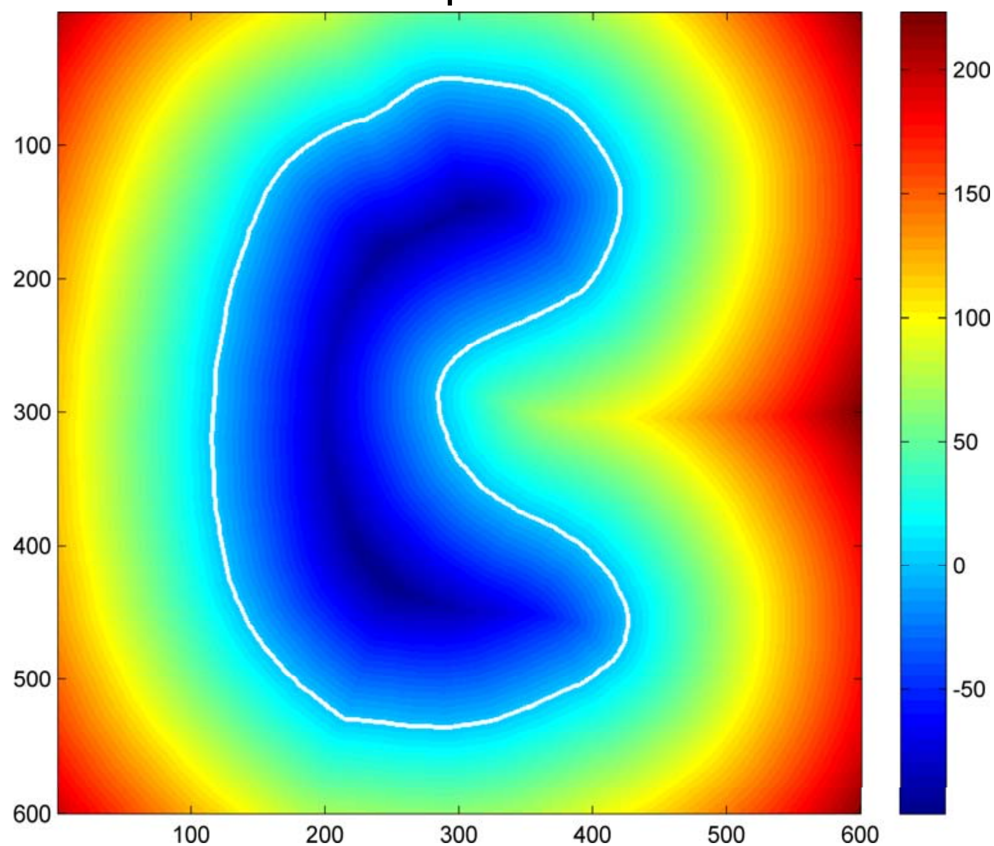
Define the interface as the locations where a signed distance function crosses zero.

We can then manipulate the interface by altering the function (the level set method)

1D line segment representation



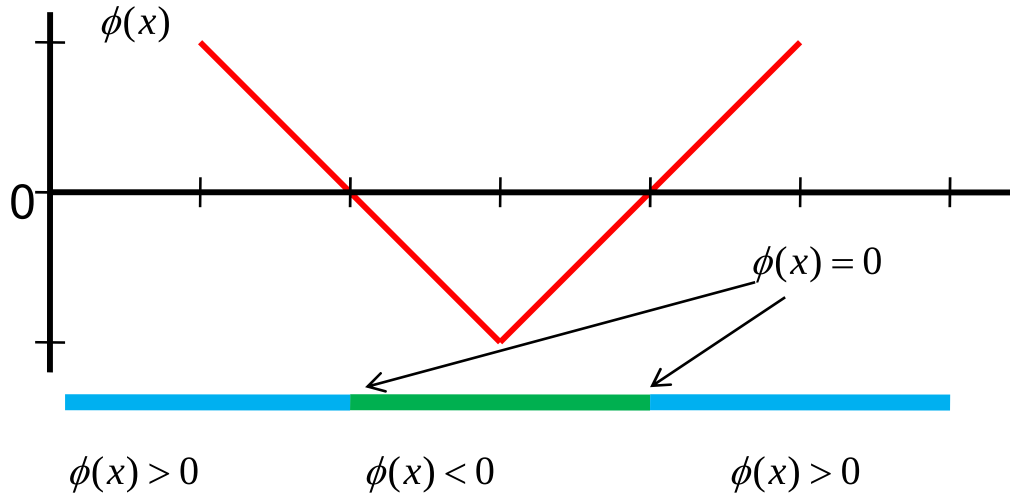
2D curve representation



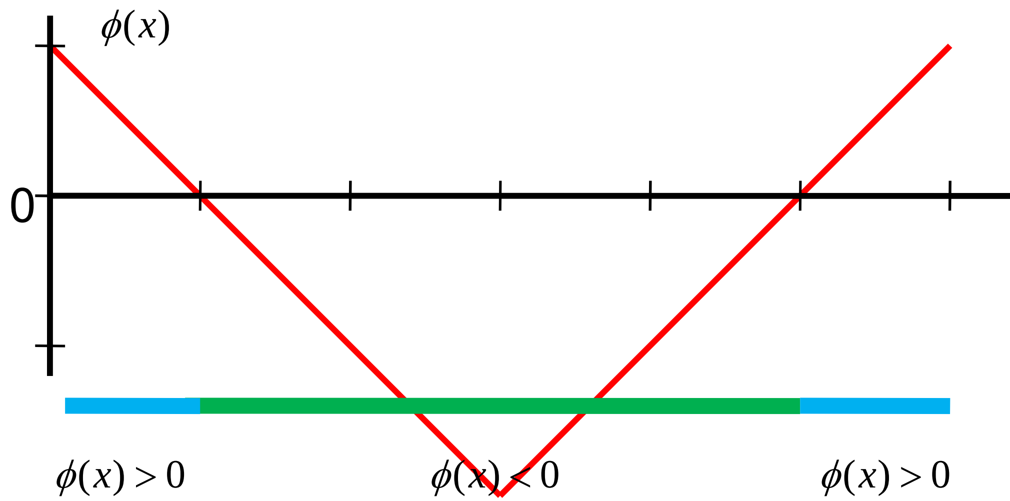
Interface evolution

Expansion:

$$\phi^{n+1} = \phi^{n} - a$$



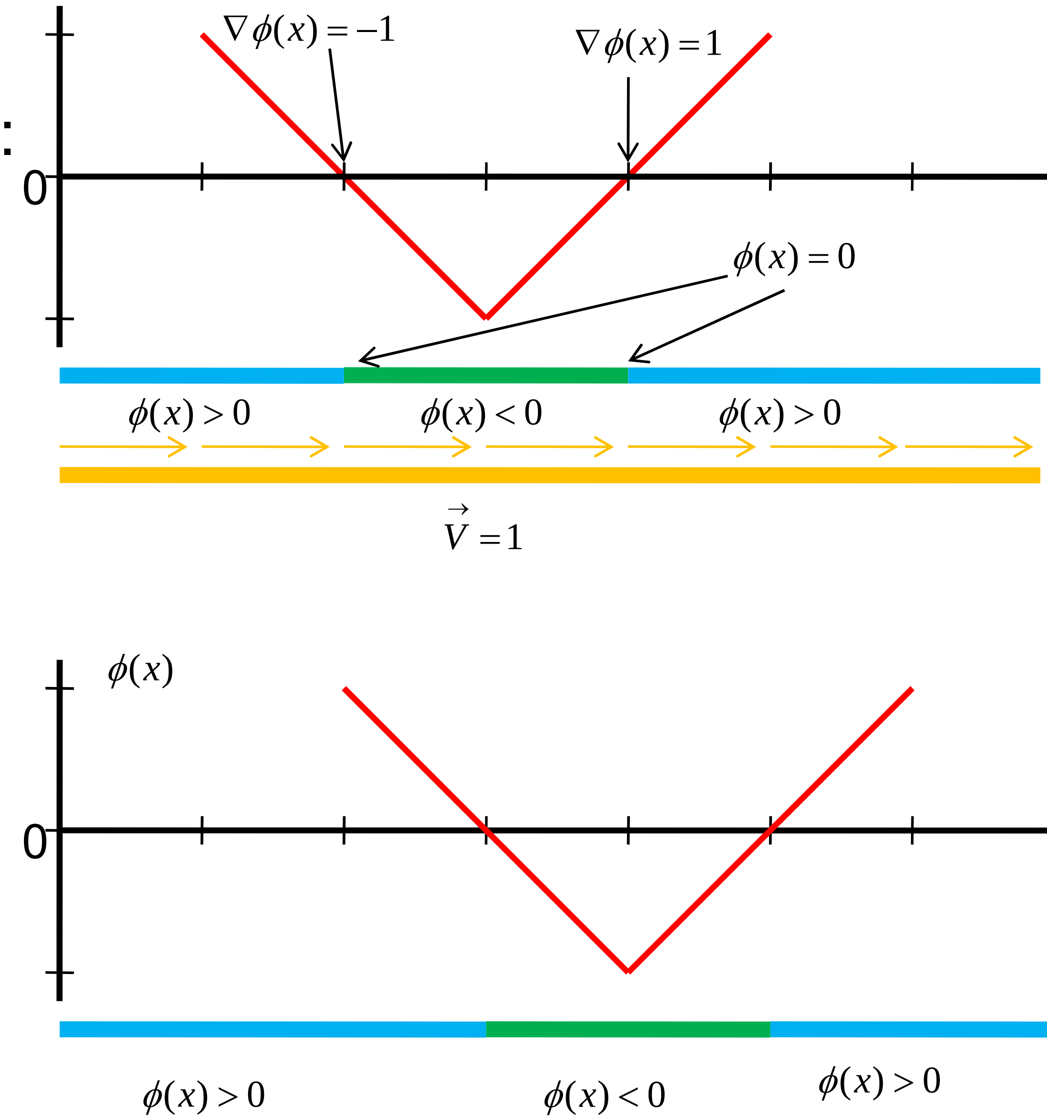
$$a = 1$$



Interface evolution

Manipulation by vector field:

$$\phi^{n+1} = \phi^n - \vec{V} \cdot \nabla \phi^n$$

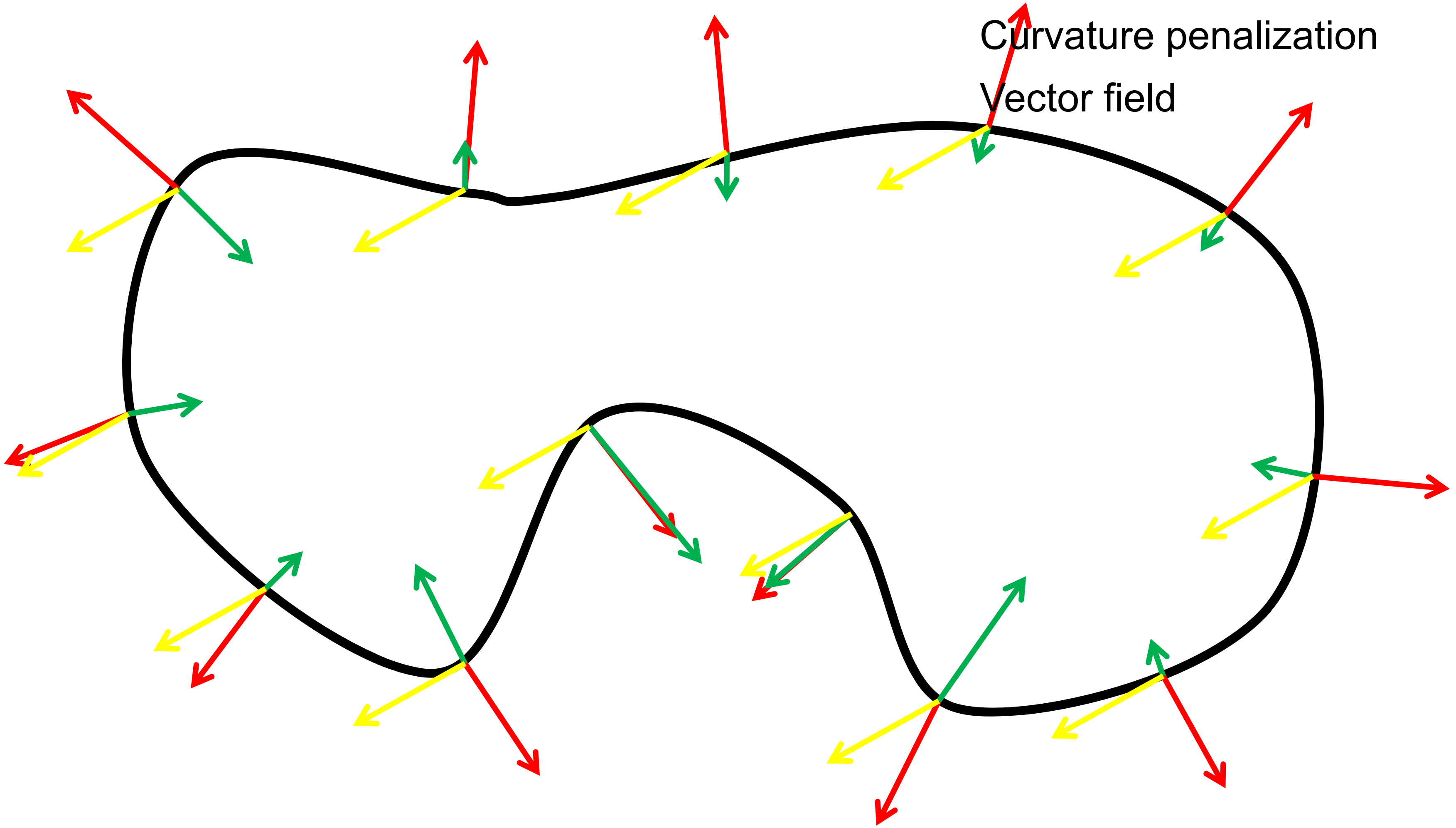


Vector velocity analogy

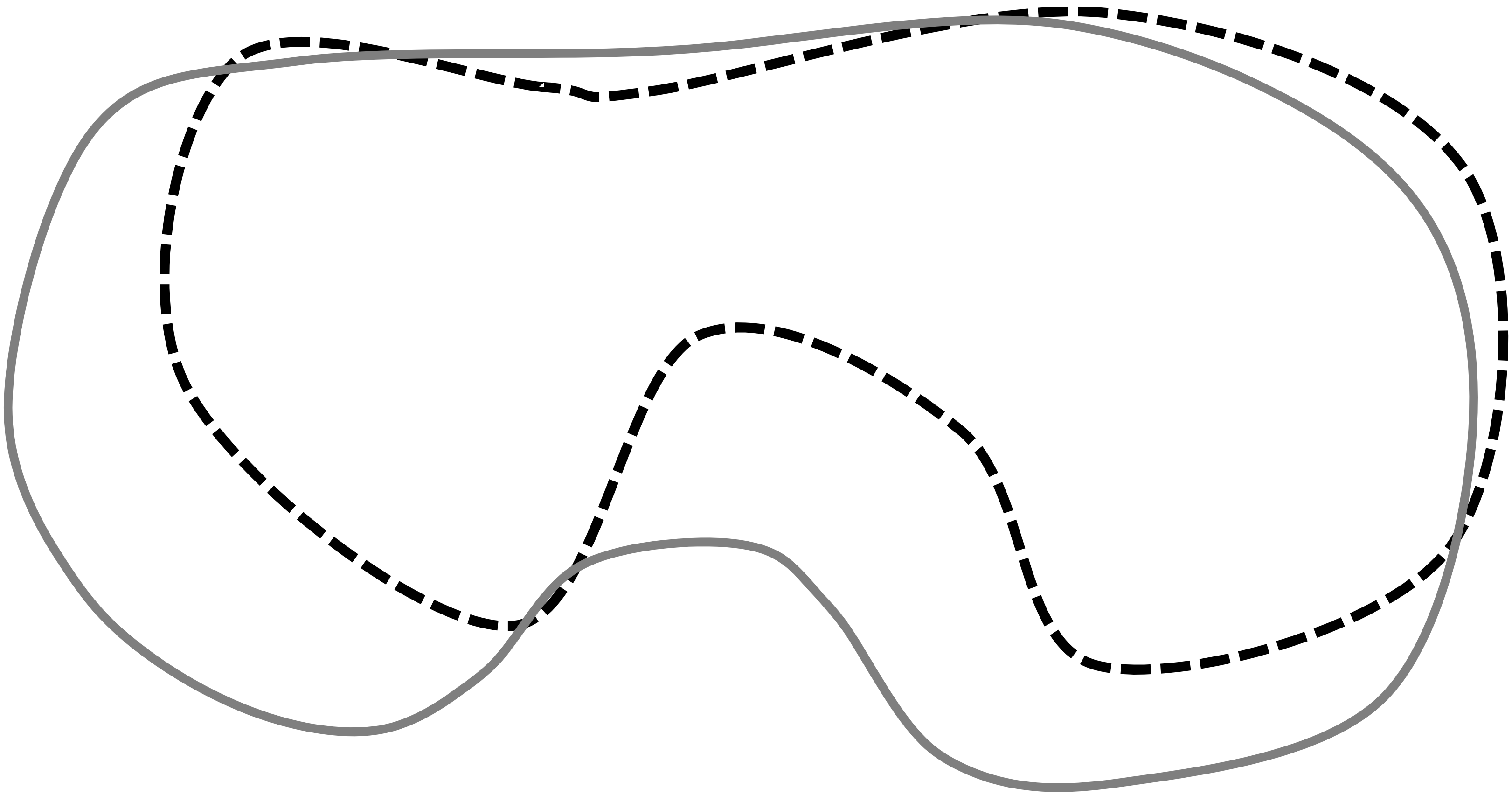
Expansion in normal direction

Curvature penalization

Vector field

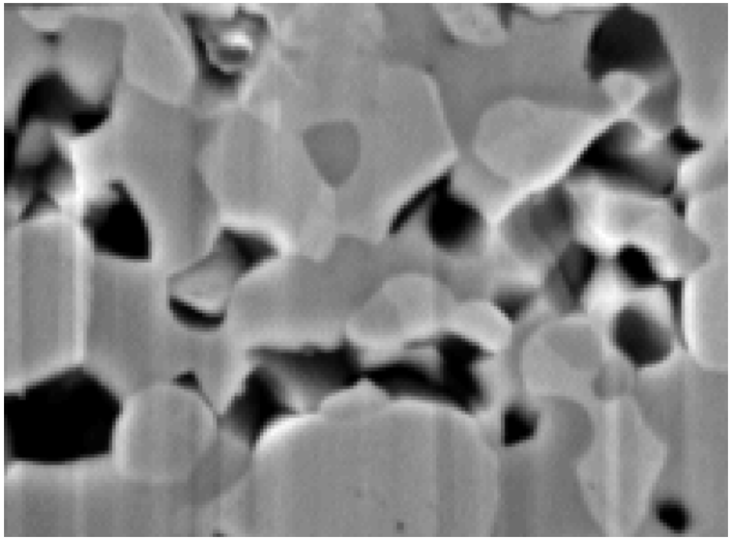


Vector velocity analogy

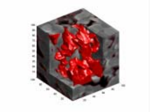


Designing the terms

- Defined the scalar and vector fields from the image data.
- The scalar field is controlled by the image intensity in the image.
- The vector field V is controlled by the intensity gradient in the image.
- Introduce a curvature penalizing term to enforce smooth surface.



$$\phi^{n+1} = \phi^n - \Delta t(\vec{V} \cdot \nabla \phi^n + a - b\kappa)$$



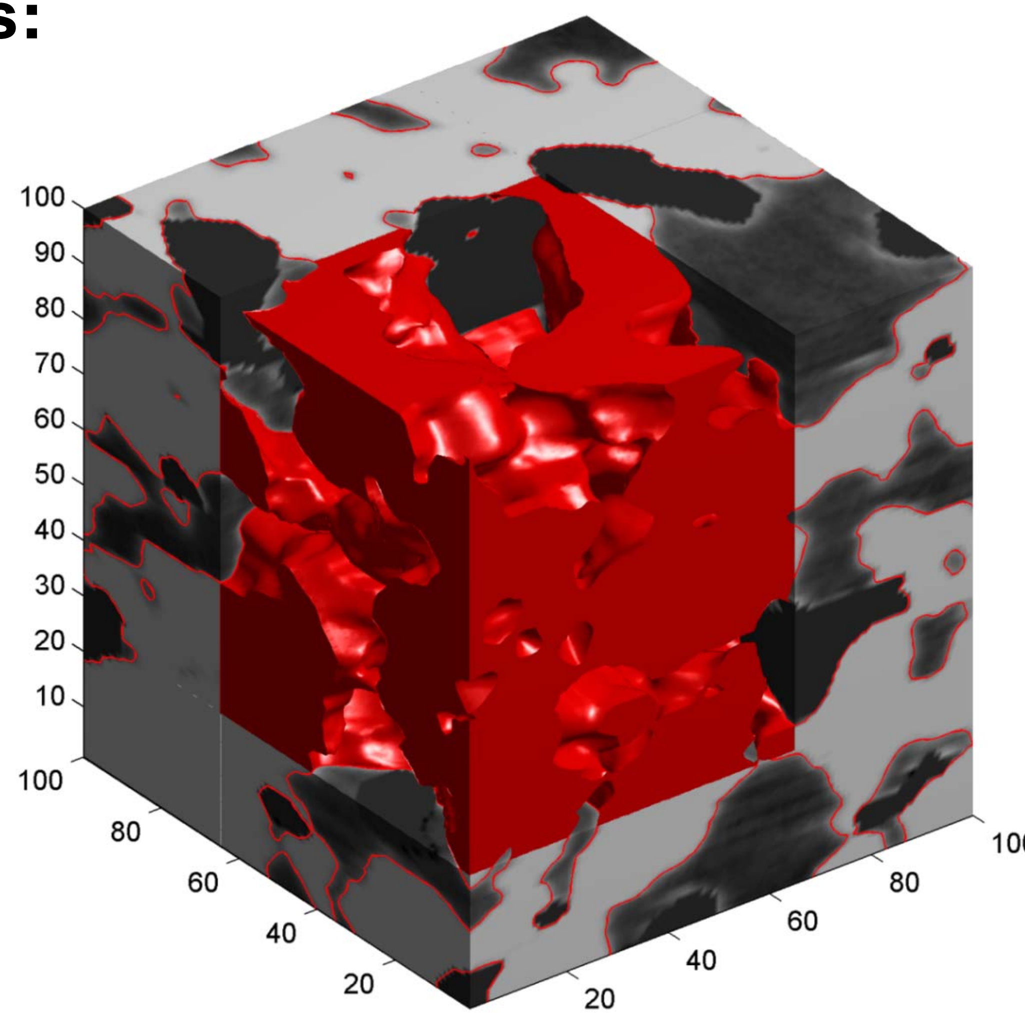
Level set advantages:

Forms coherent structures

Smooth surfaces due to curvature penalization.

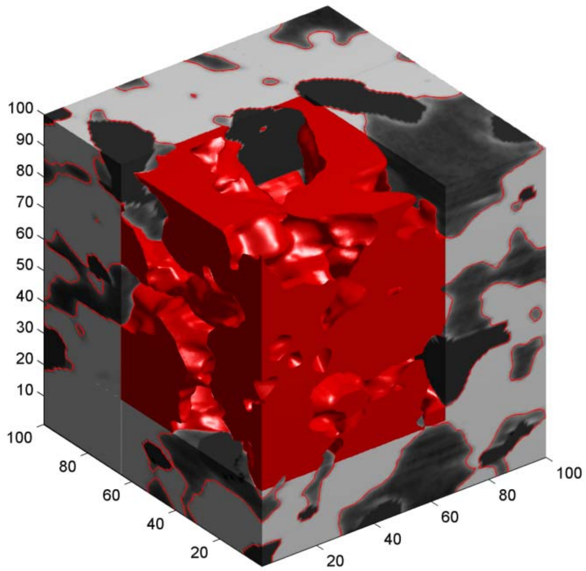
Subvoxel precision of interface locations.

Complexity can be scaled by adding or removing terms in the update rule depending on the segmentation task.



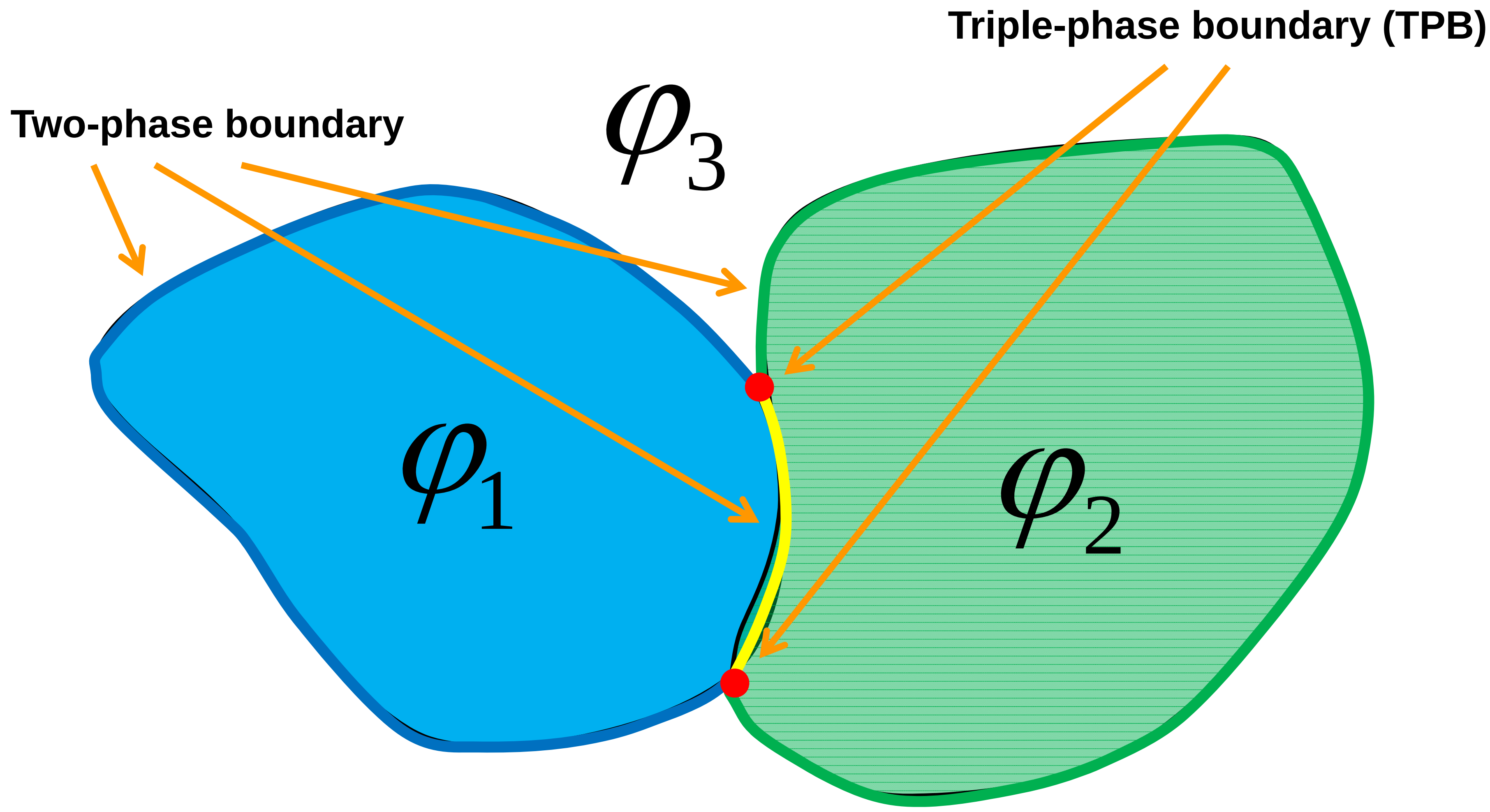
LSC/CGO sample 5.86 x 5.86 x 4.46 μm

3D measurements

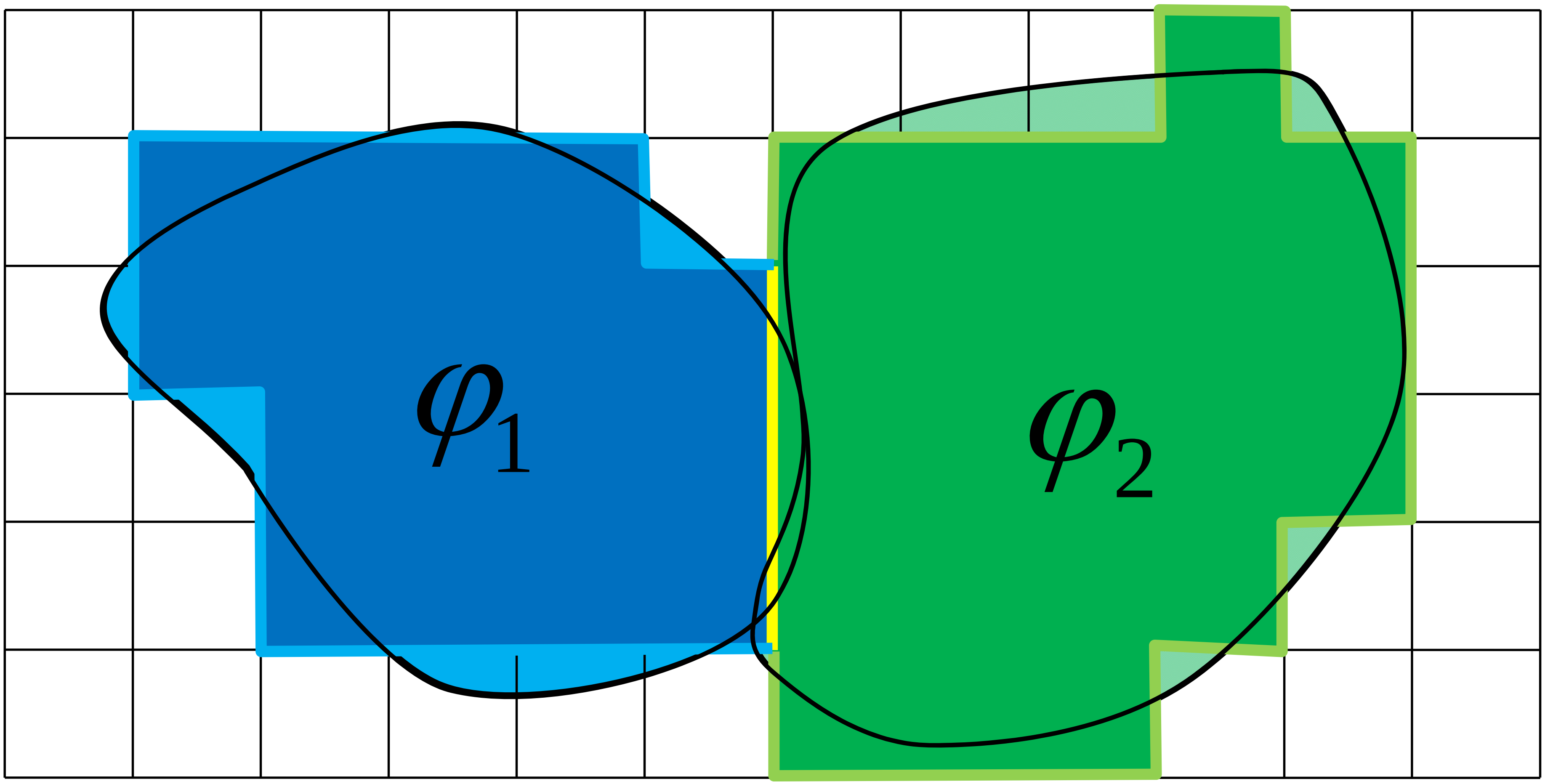


→ TPB = ???

Phase boundaries



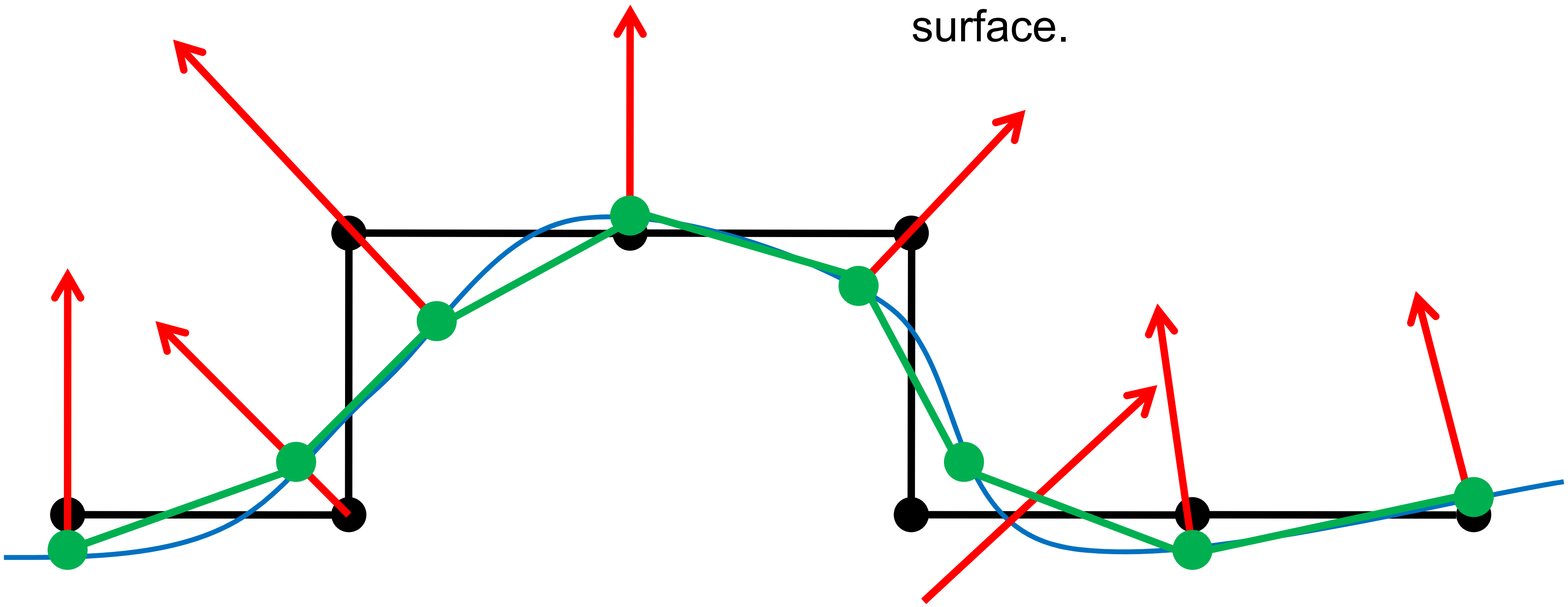
3-phase polygonization



Refining the surface

Interpolate surface normal from the apropriate signed distance function.

The value of the signed distance function gives the distance to the surface.

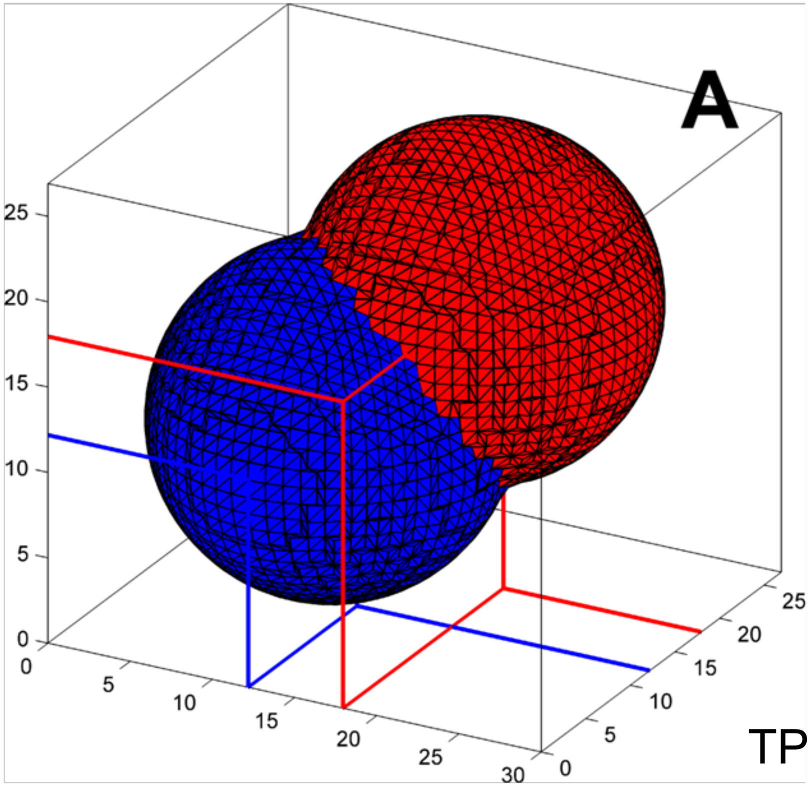


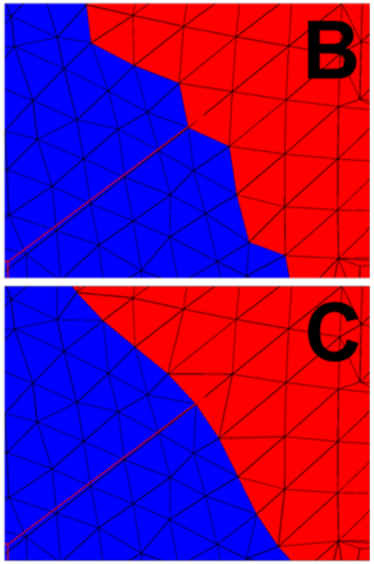
TPB and surface area calculations

Voxel based level set segmentation is Converted into a polygonal mesh structure.

•Sum over polygon areas for surface area calculations.

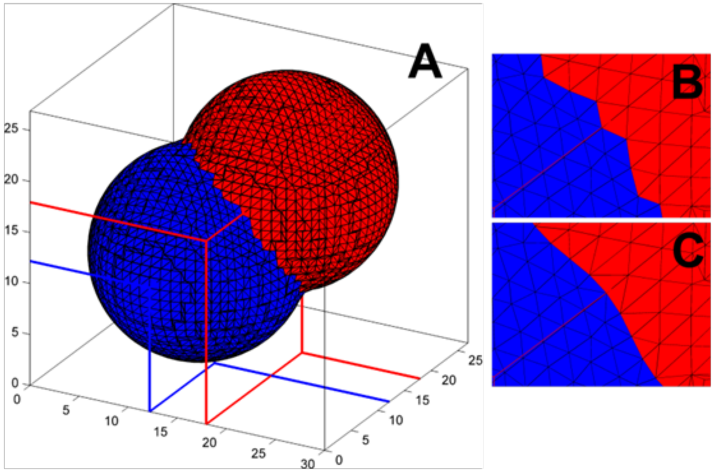
•Sum over smoothed polygon edges for TPB length calculations.

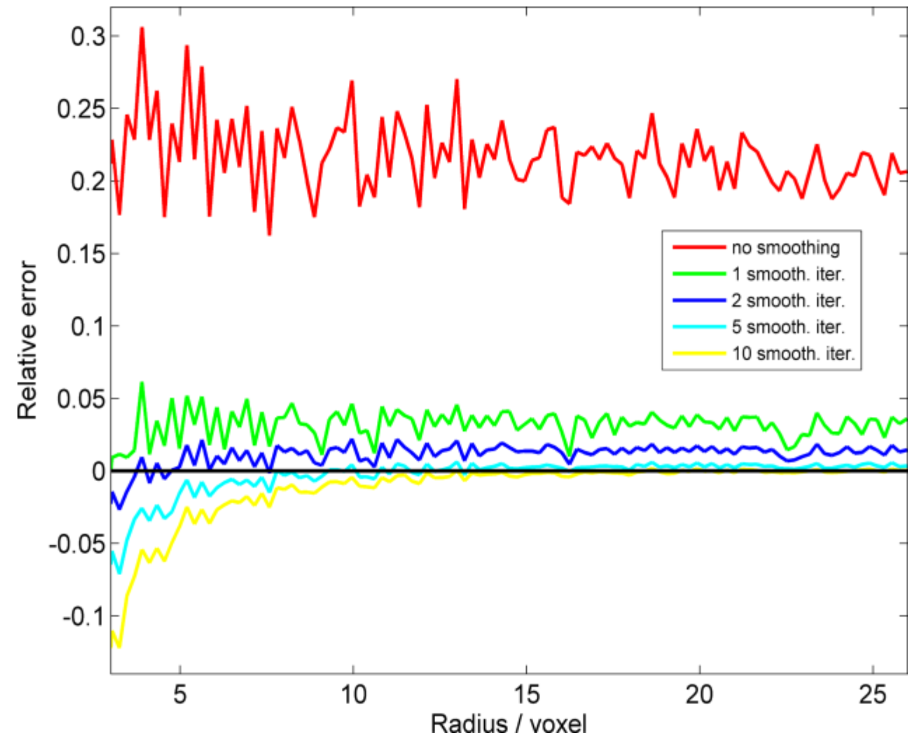


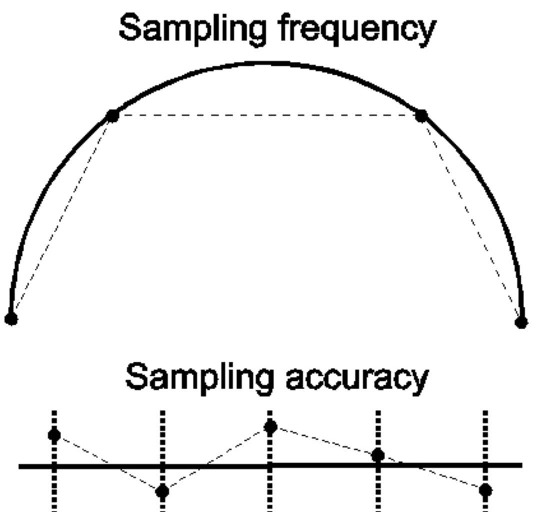


TPB curve smoothing

Accuracy analysis





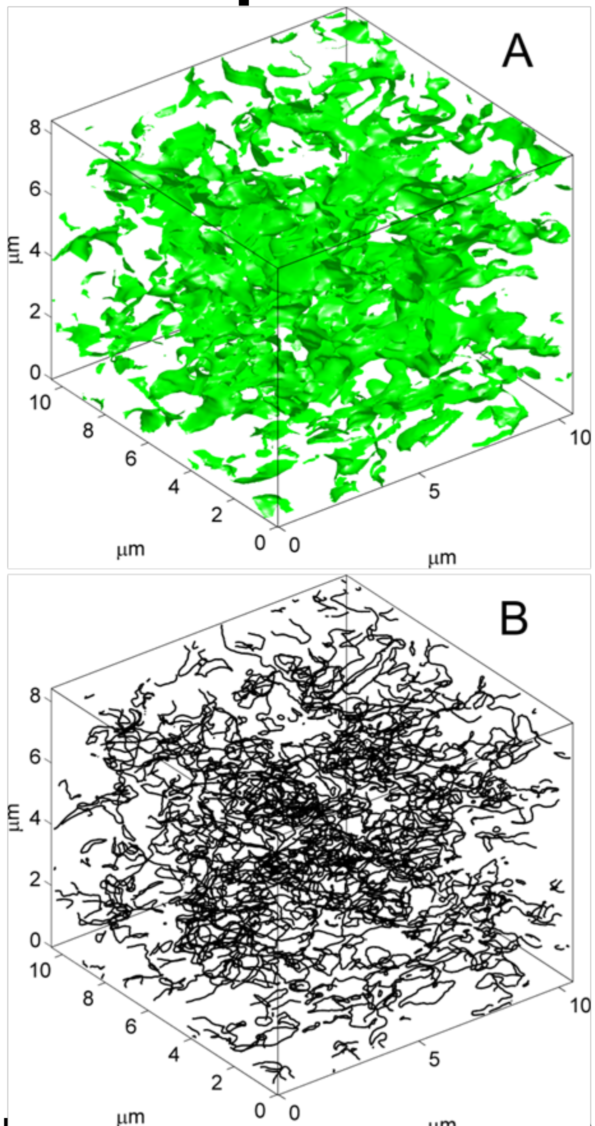


- The interface reconstruction is run on artificial spheres of varying radius.
- The reconstructed TPB length is compared to the exact geometrical value.

LSC / CGO example results

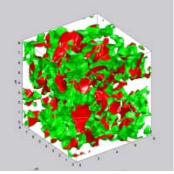
Pore/LSC interface surface

TPB curves



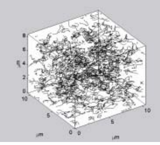
L_v (TPB)
L_v (Percolating TPB)
S_v (Pore surface area)
S_v (CGO surface area)
S_v (LSC surface area)
S_v (Pore/CGO surface area)
S_v (Pore/LSC surface area)
S_v (CGO/LSC surface area)

Volume specific measurements



Interface video

Ni/YSZ sample
red: Pore/Ni interface
Green: Pore/YSZ interface

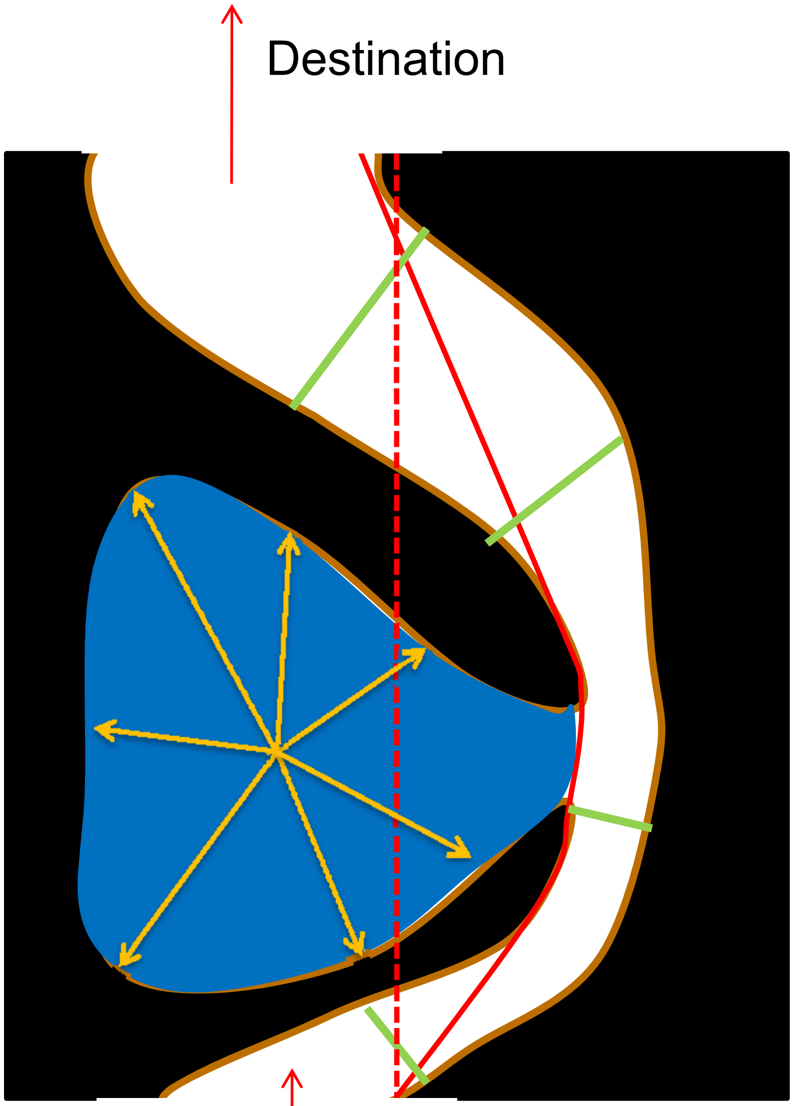


TPB video

LSC/CGO sample

Network characterization

- Shortest path tortuosity
  - Important for gas diffusion
- Transport network thickness
  - Diameter distributions
  - Bottleneck identification
- Dead ends
  - how large a fraction of the phase is percolating but not part of the main pathways
  - Identification of inefficient phase usage.
- Cavity sizes
  - cavity size distribution



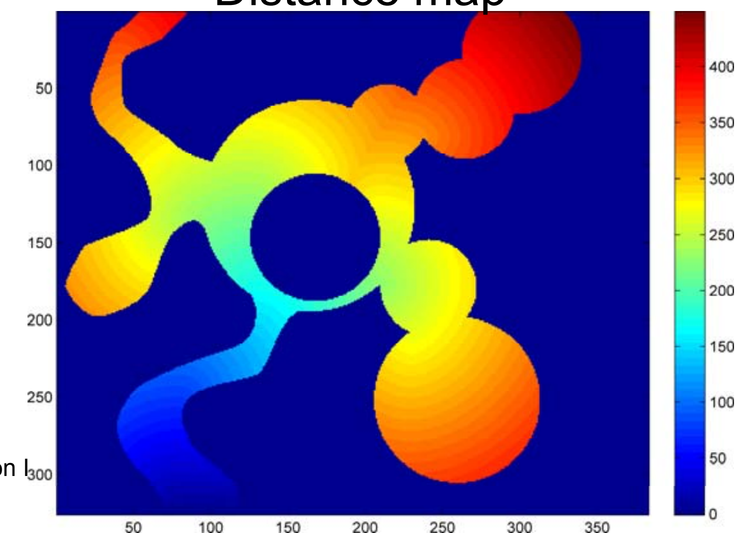
Calculation of tortuosity

•Calculate the distance map from the source to the destination side by the fast marching method.

•Calculate destination tortuosity as the distance value at the destination side divided by the distance between the sides.

2D artificial structure

Distance map

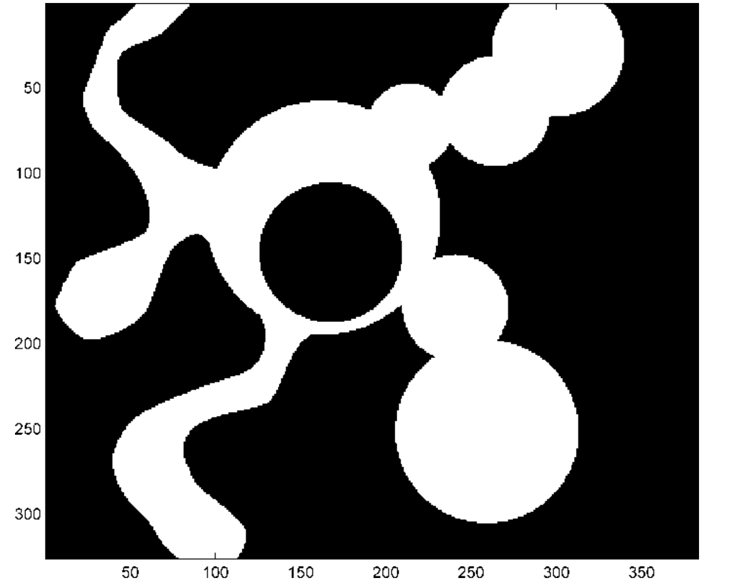


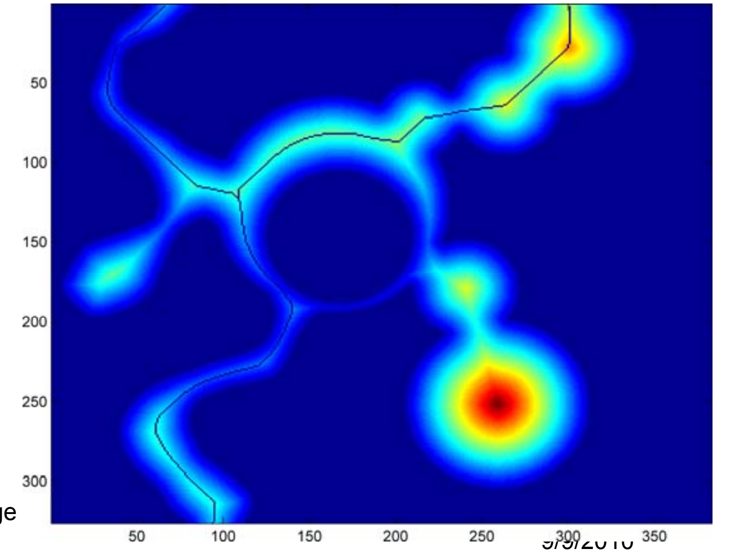
Path diameter and cavity size

•Calculate the distance map from the interface

•Identify the widest paths between two sides.

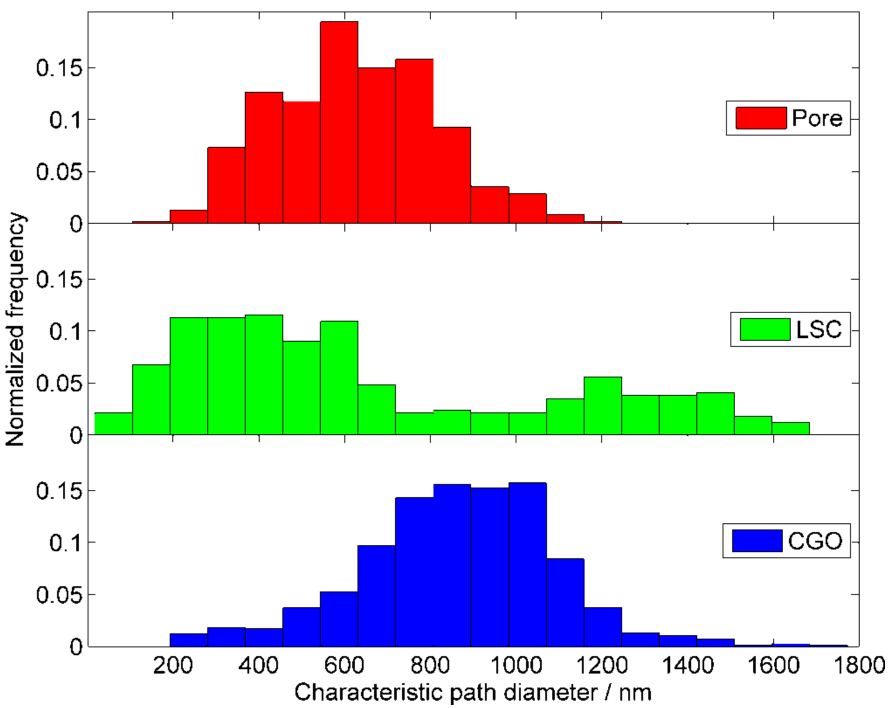
•Extract the path diameter distribution from the distance values (the radius) in the widest paths.

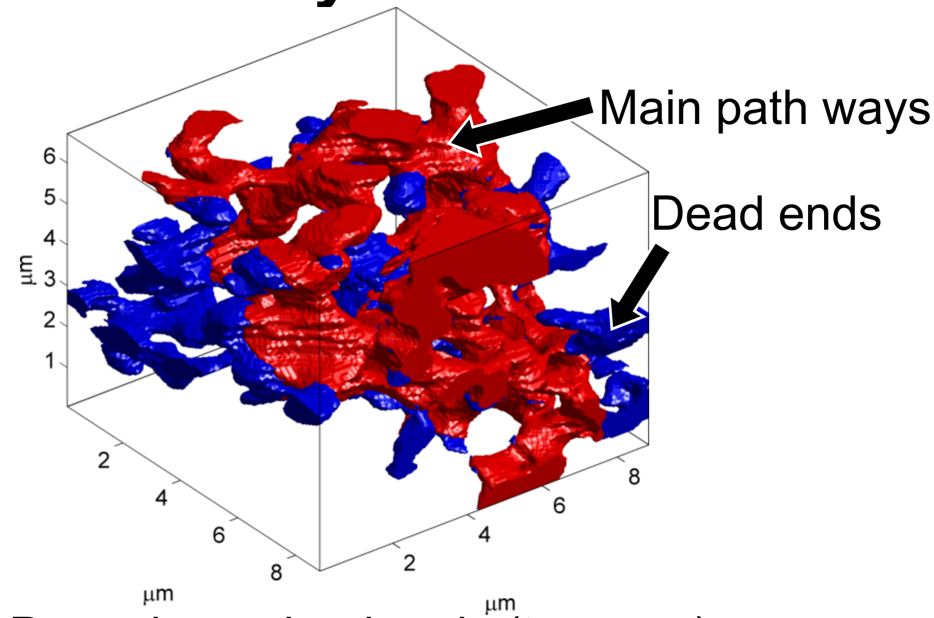




Selected LSC/CGO network analysis results

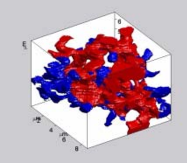
Pathway diameter distribution





Pore phase dead ends (two way)

	Dead end fraction	Average dead end distance
Pore	0.278	1.72 µm
LSC	0.245	1.13 µm
CGO	0.047	0.69 µm



Dead ends visualization

LSC/CGO sample

Automation overview

- Data acquisition
- Image alignment
- Illumination correction
- Segmentation
- Quantitative characterization

Automatic

Semi-automatic (parameter tuning)

The segmentation is currently where the most bias is introduced. It is the most critical step for the overall accuracy of quantitative measurements.

Other examples of degradation investigation methods

Low-voltage SEM

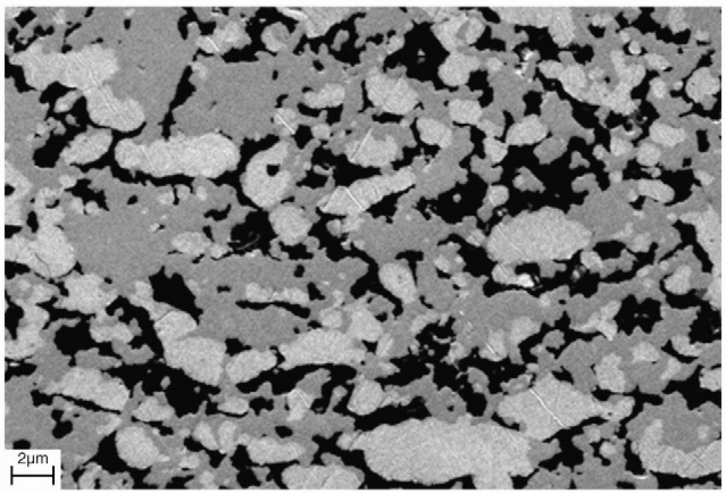


Fig. 3. Image recorded by use of a low voltage and the lateral SE detector. The bright phase is Ni, the grey phase is YSZ and the dark phase is epoxy. The image was acquired with a slow scan rate, an acceleration voltage of 1 kV and a working distance of 10 mm.

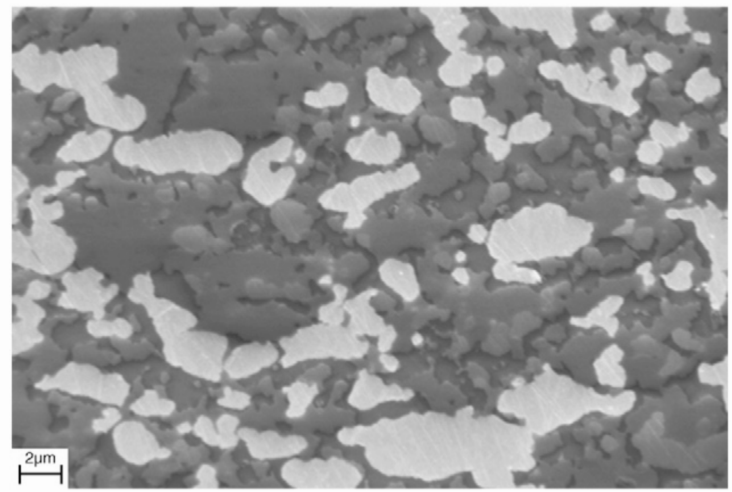
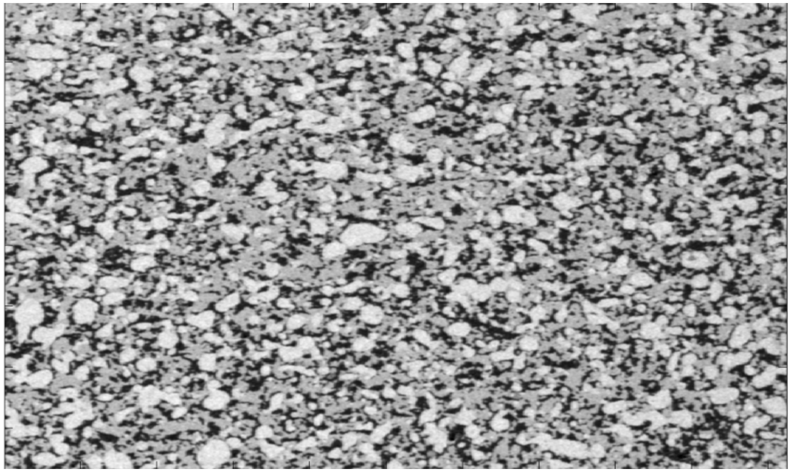
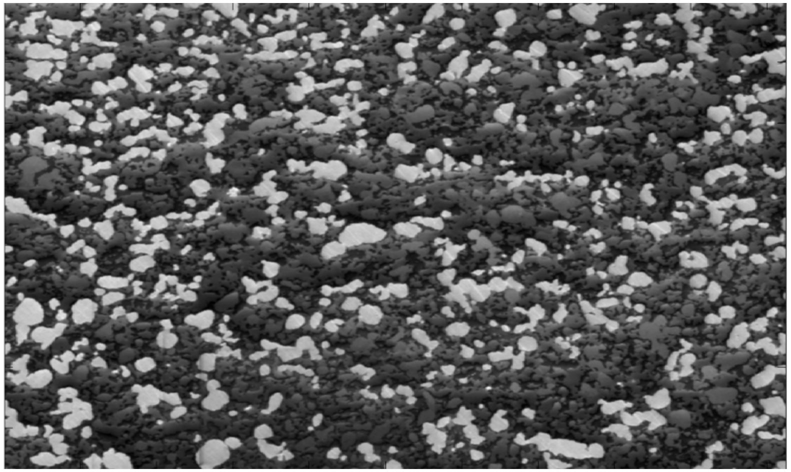


Fig. 4. Image recorded by use of low voltage and the inlens SE detector (same area as in Fig. 3). The bright phase is percolating Ni and the dark parts are non-percolating Ni and non-conducting phases.

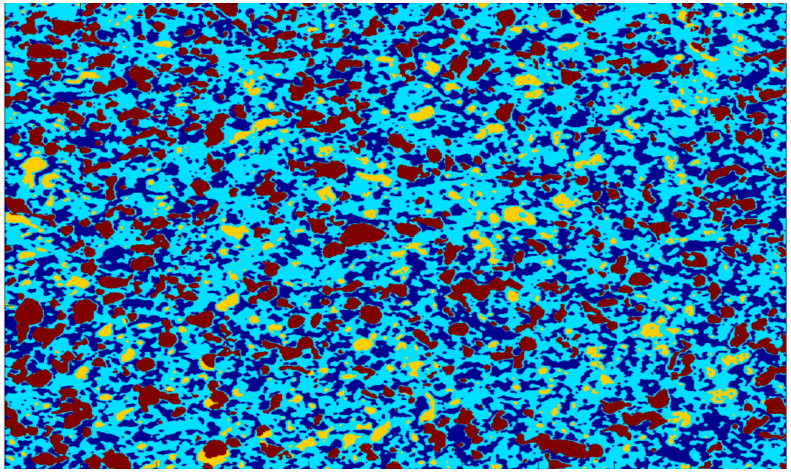
Thydén et al., Solid State Ionics 178 (2008) 1984–1989

Bi-modal 2D image segmentation

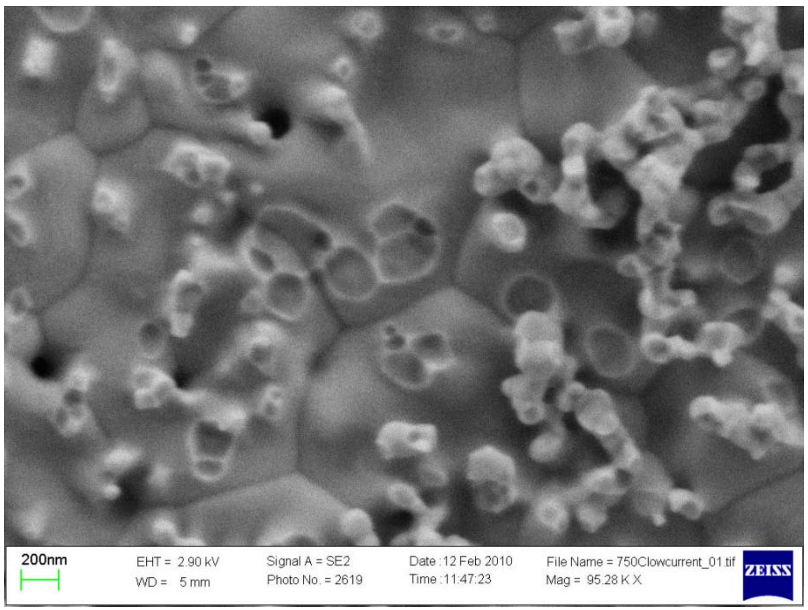




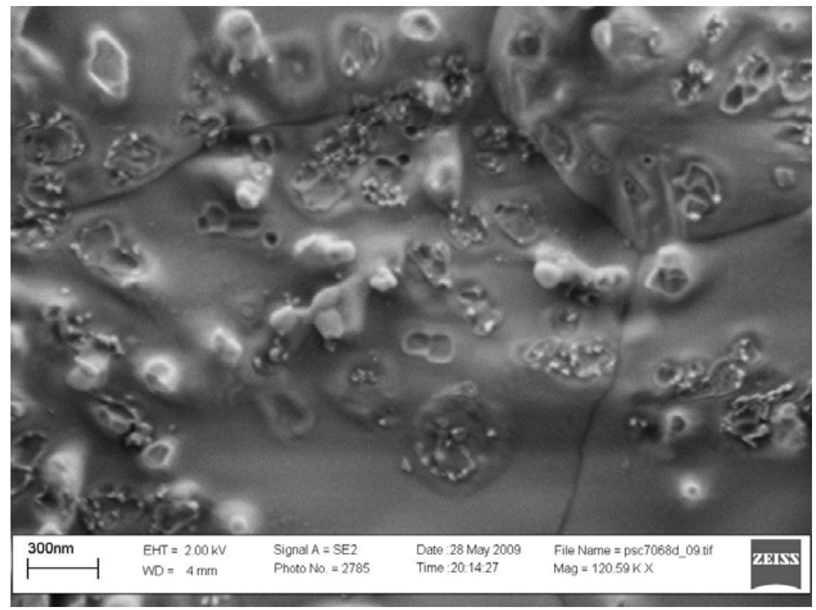
Percolating Ni
Ni
YSZ
Porosity



Etched LSM-YSZ SOFC cathodes tested in dry air @ 750°C



Low current → mild degradation



High current → severe degradation

Liu, Y.L. et al., Solid State Ionics, 180 (2009) 1298.

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