December 9th, 2014

Accurate 3D microstructure characterisation of porous materials by X-ray microtomography

A need for advanced and specialised image processing tools

Université de Liège

Erwan Plougonven

LGC Chemical Engineering

The Department of Applied Chemistry

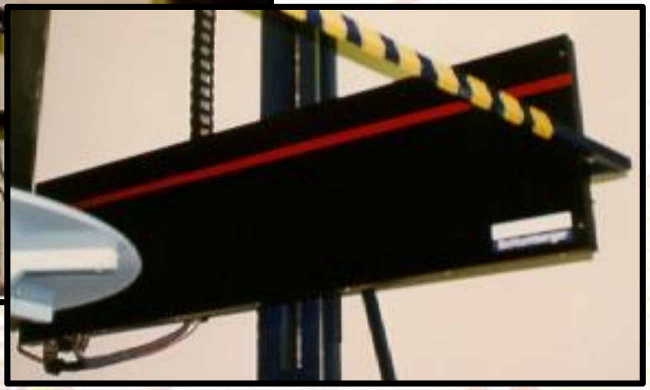
- Applied Sciences Faculty

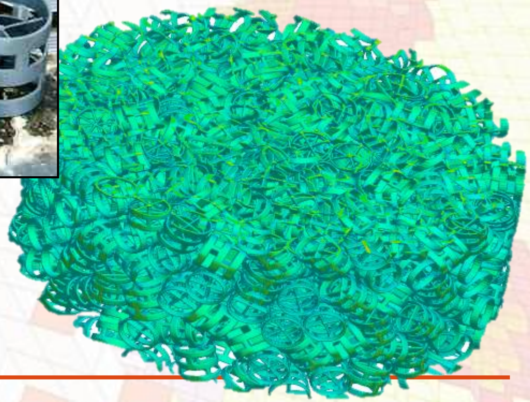
3 groups in the department (~55 people)

- Analysis and Synthesis of Chemical Systems – Cryotechnology laboratory
- Nanomaterials, Catalysis, Electrochemistry
- LGC – Environment, Energy, Reactors, separations

http://www.chimapp.ulg.ac.be/

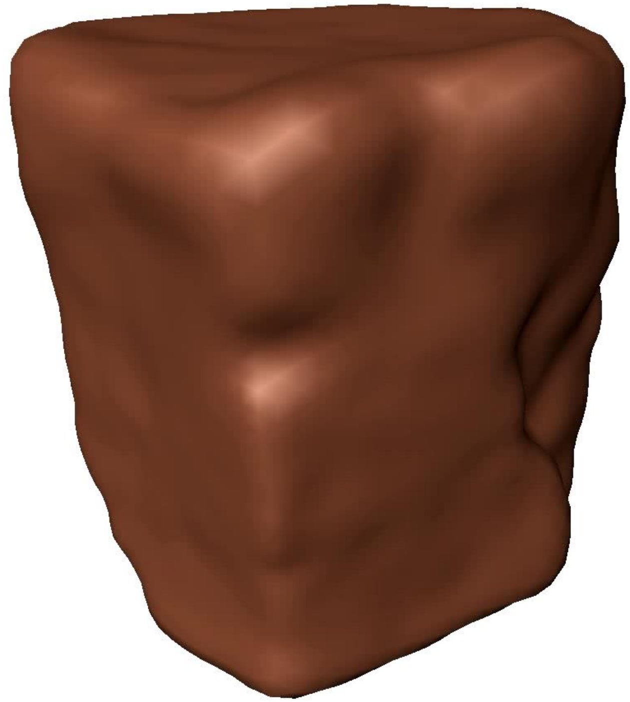
Tomography, a long history at the LGC

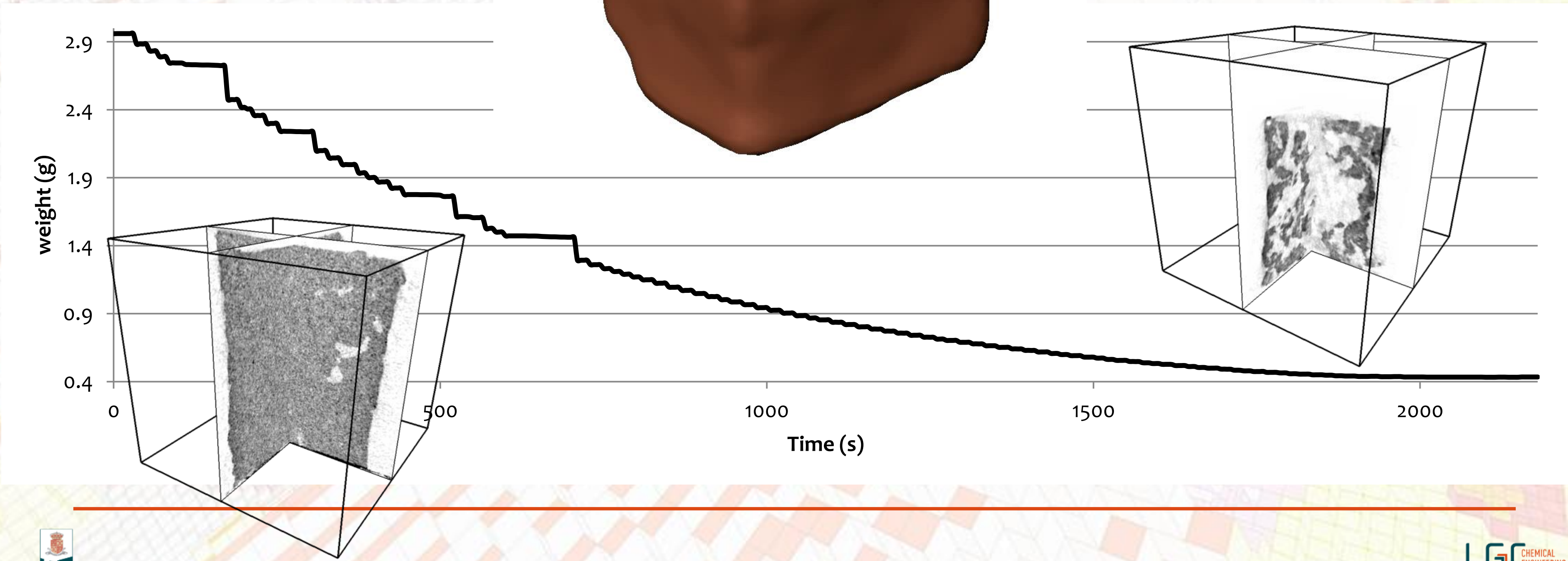




Tomography, a long history at the LGC

Skyscan-1074 portable µCT

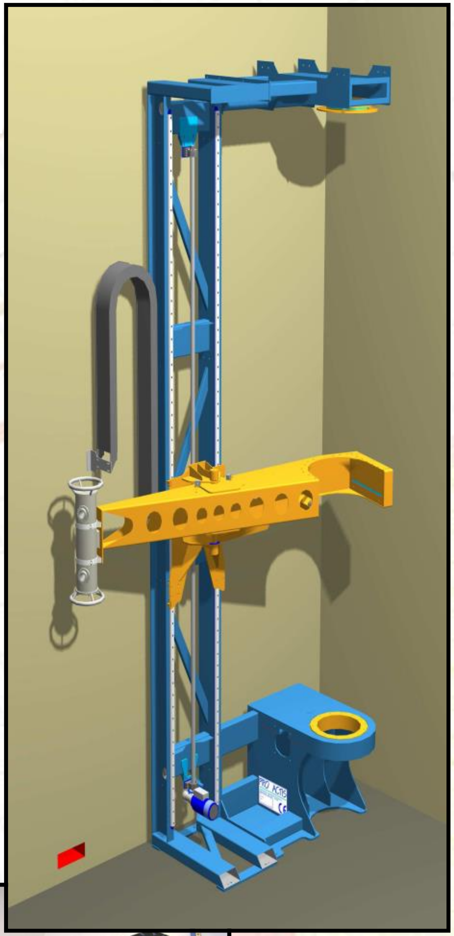


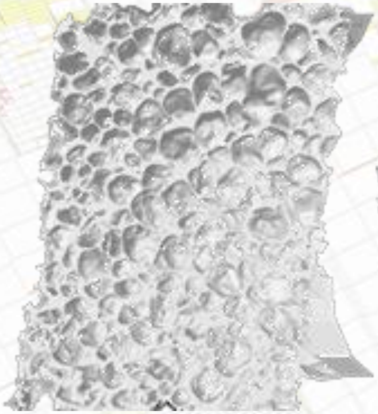


Micro and macro-tomography

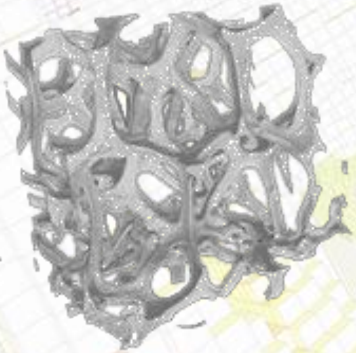
Skyscan-1074 portable µCT

Skyscan-1172 desktop µCT

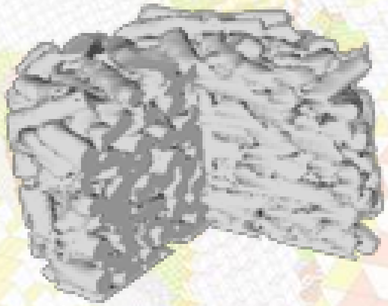




PVC foam



Metallic foam



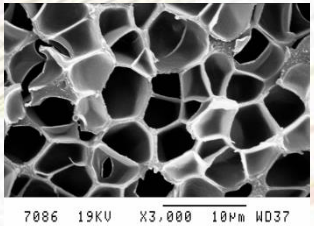
Extrudates bed

Human bone

Examples of analysis

- Nanocomposite polymer foams

  (*FNRS – ARC project*)



- Macadamia nuts

  (Collaboration with UNSW)

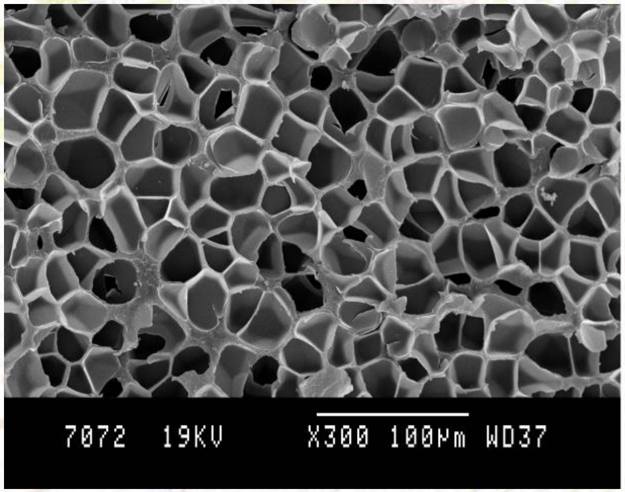
- Sintered soda-lime powder

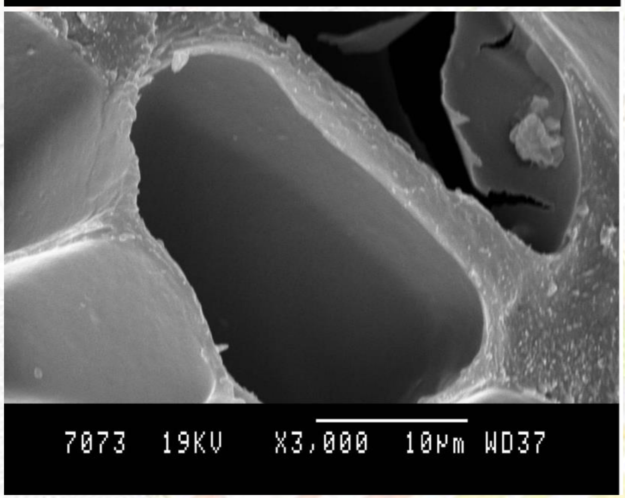
  (Phd funded by Corning)

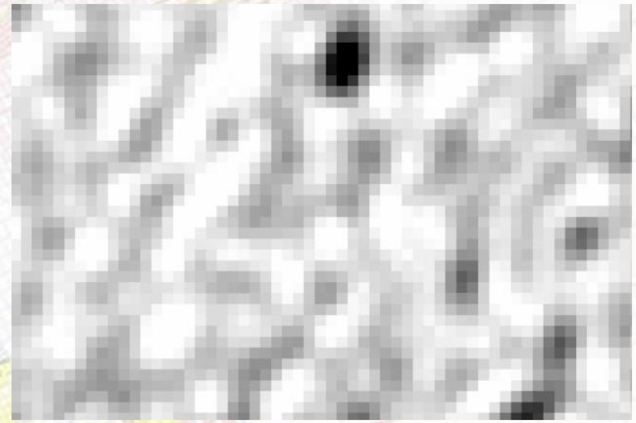
Nanocomposite polymer foams

Or what to do with poor quality 3D images ?

Polymer foams





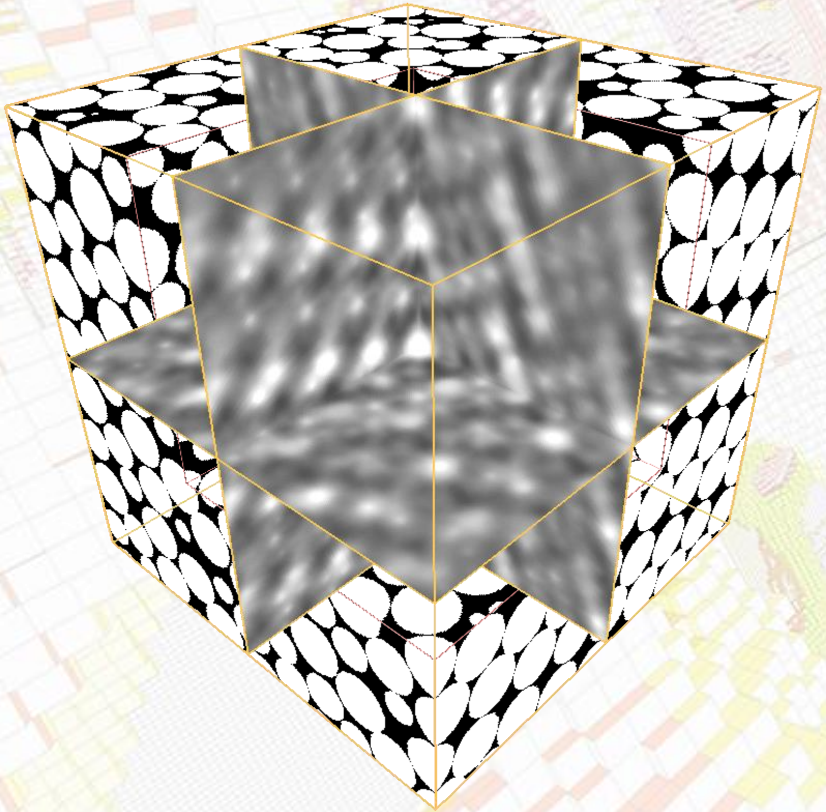


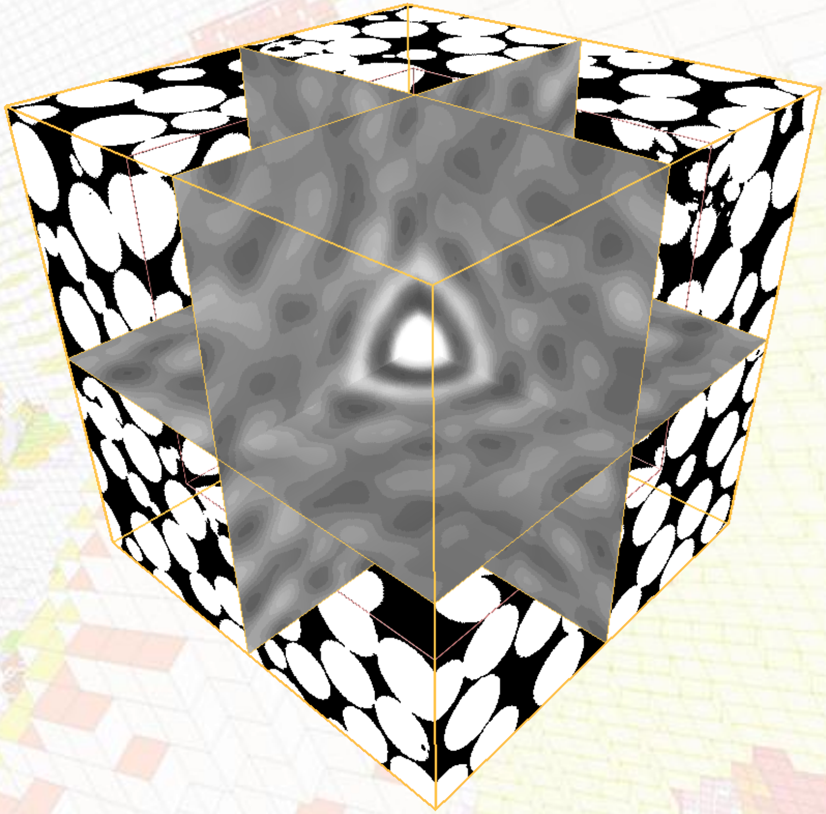
Polymer foams – characteristic length

- 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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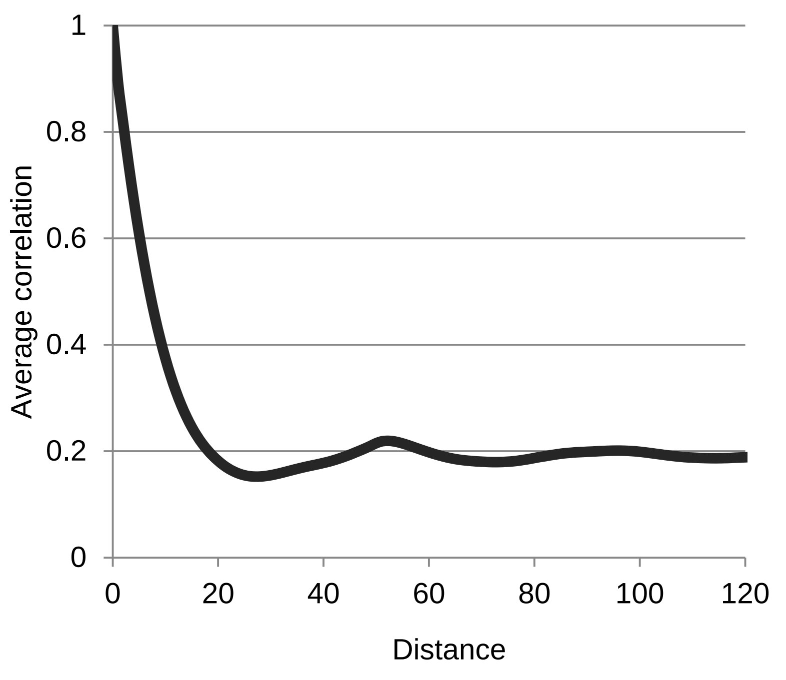
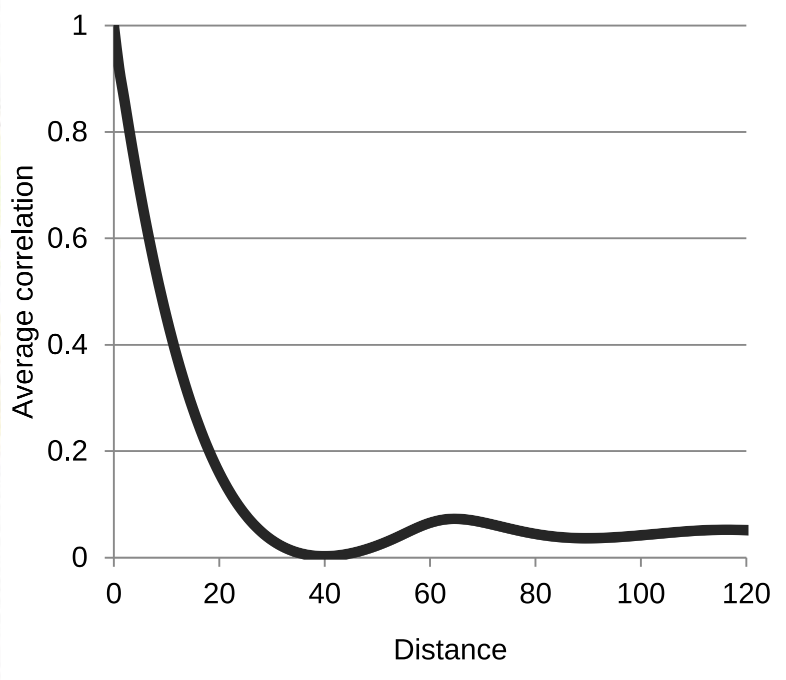
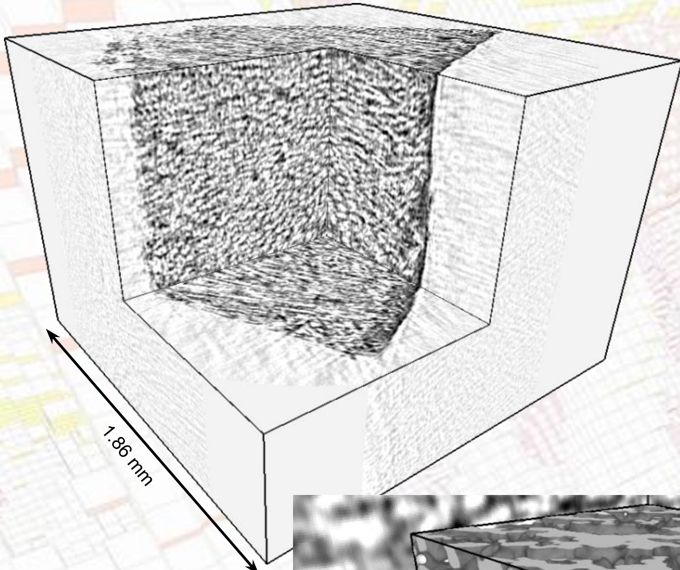
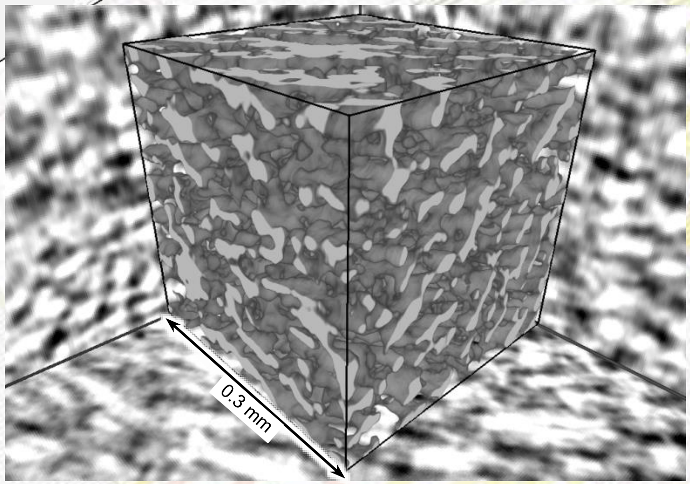


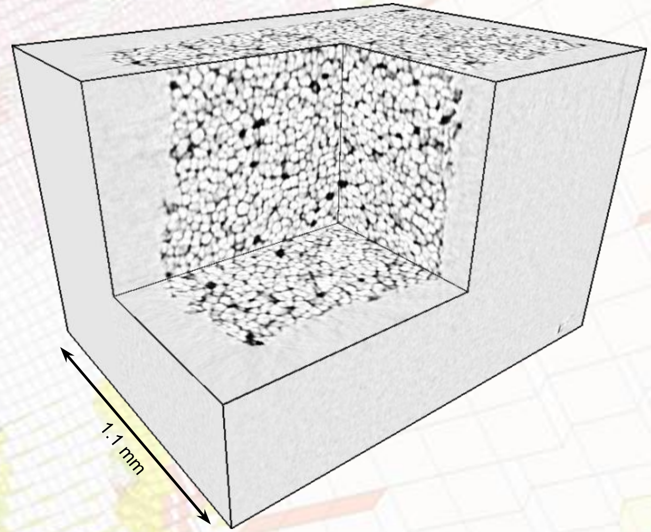


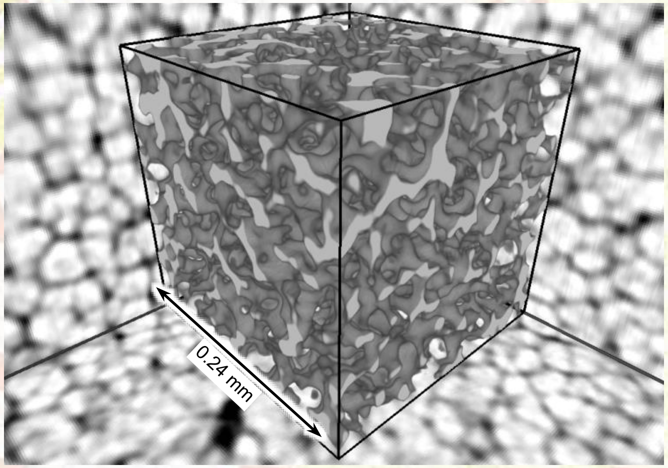
Polymer foams





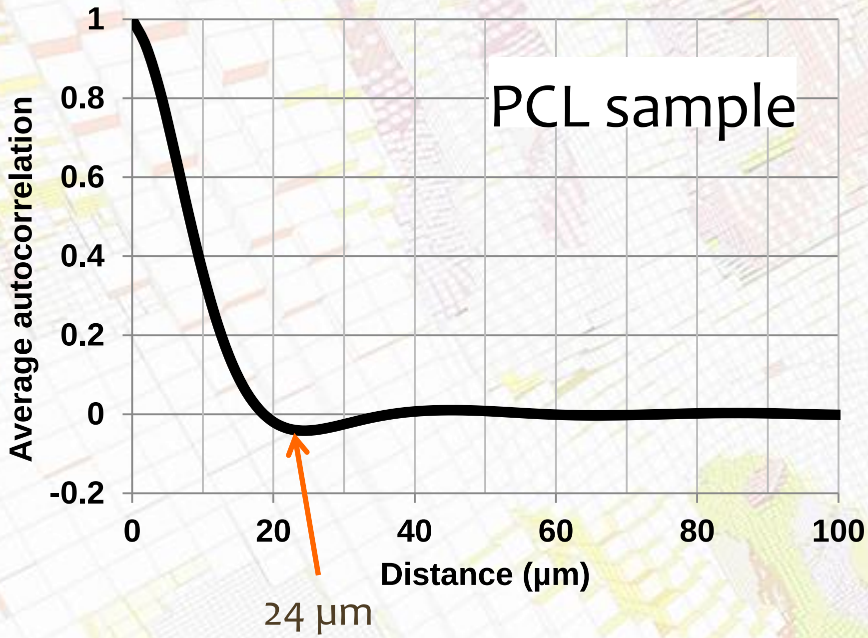
PCL sample

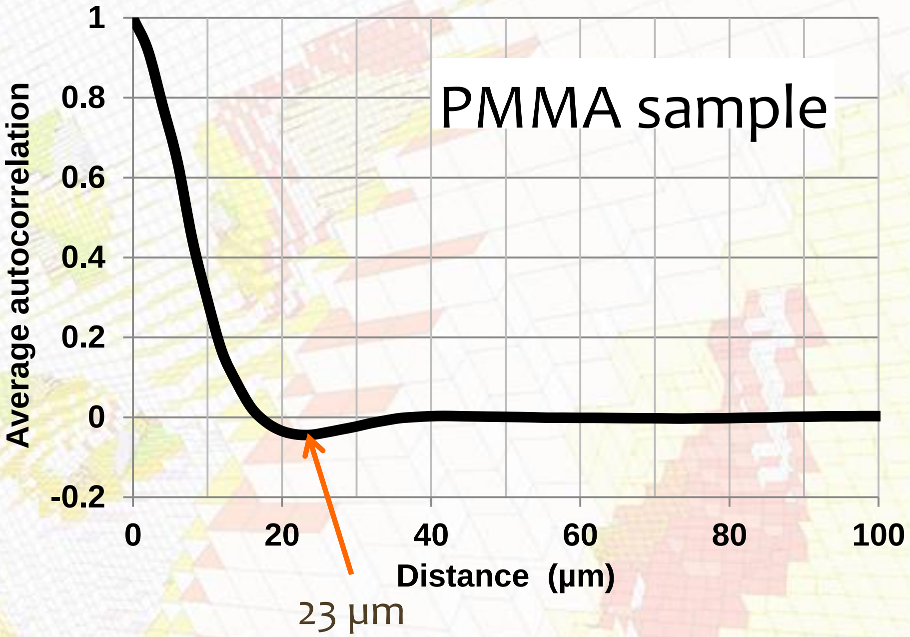




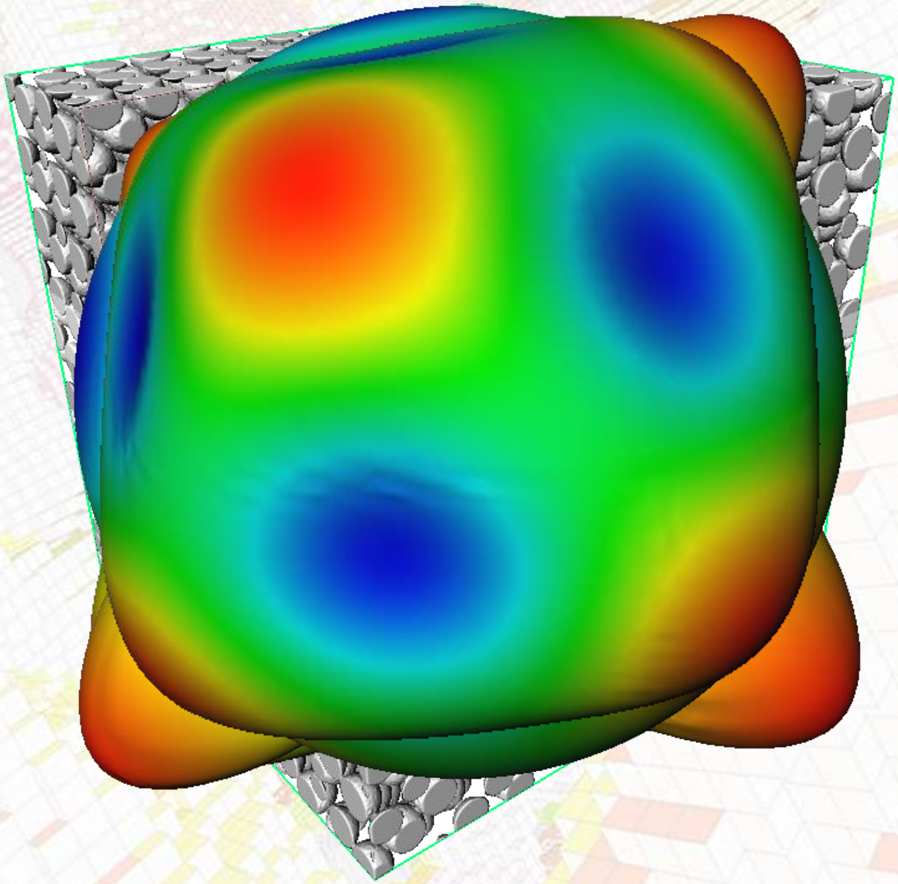
PMMA sample

Polymer foams – characteristic length

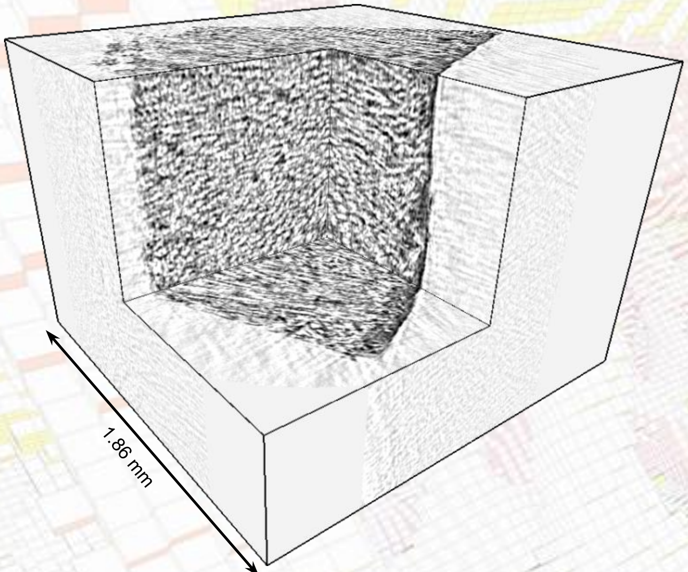




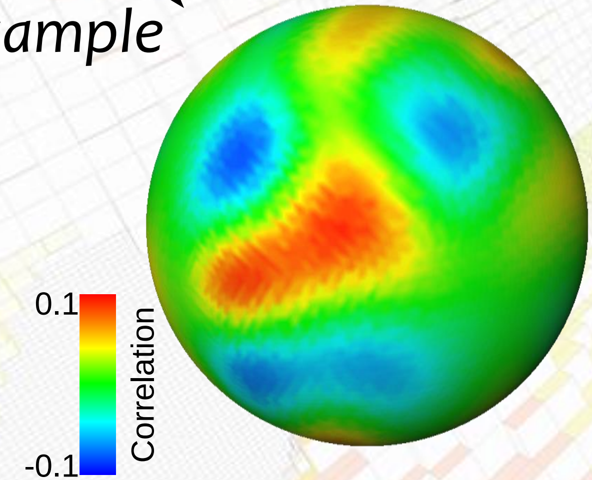
Rose diagram

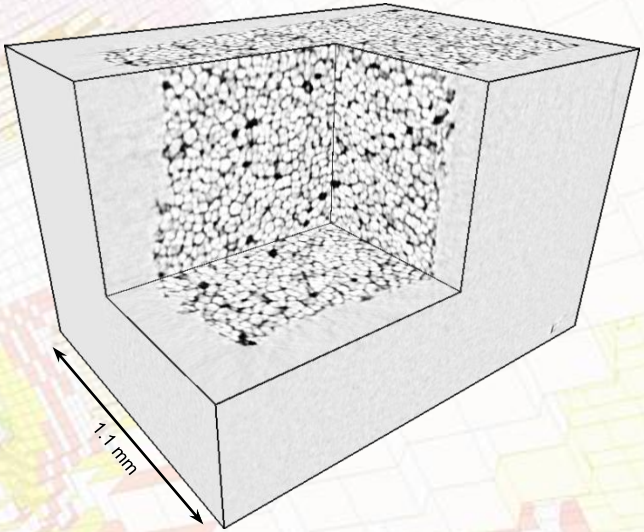


Polymer foams – anisotropy

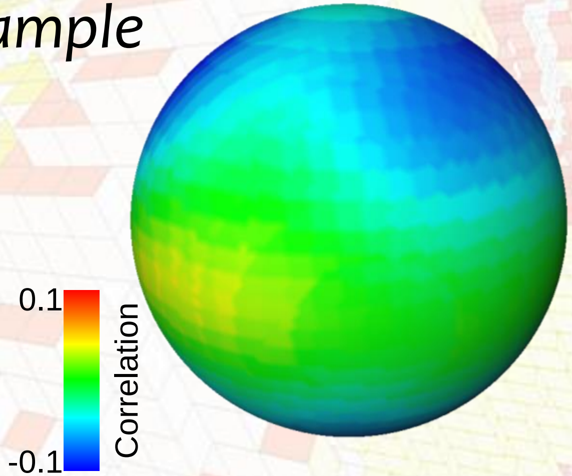


PCL sample

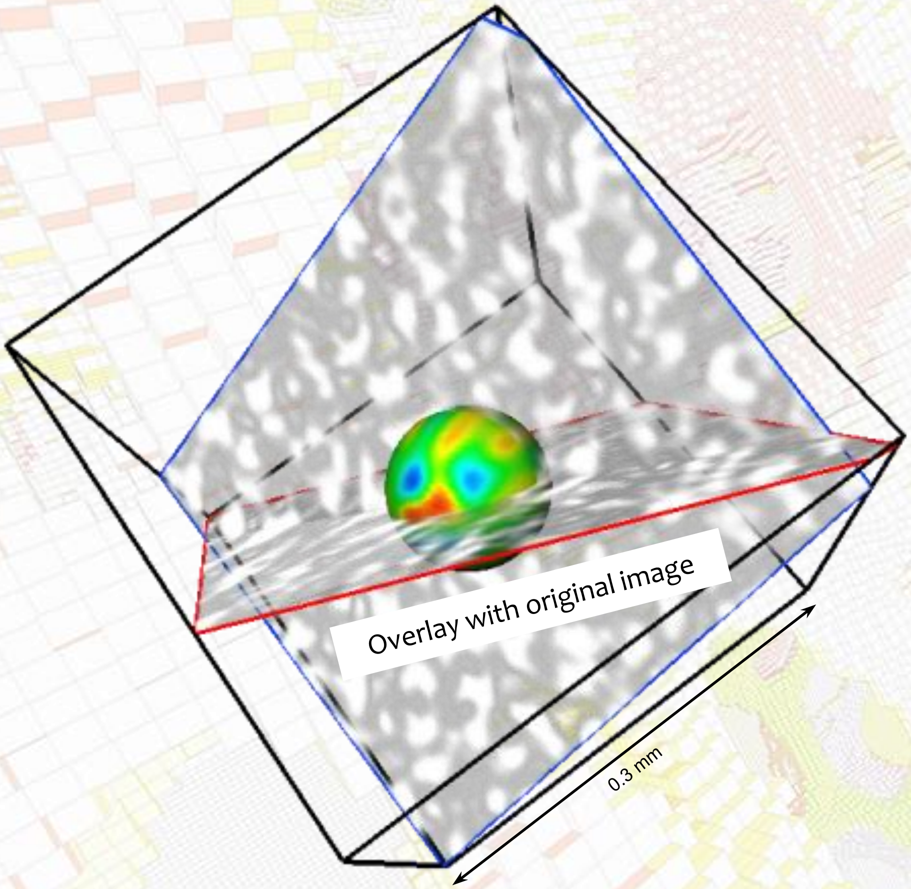


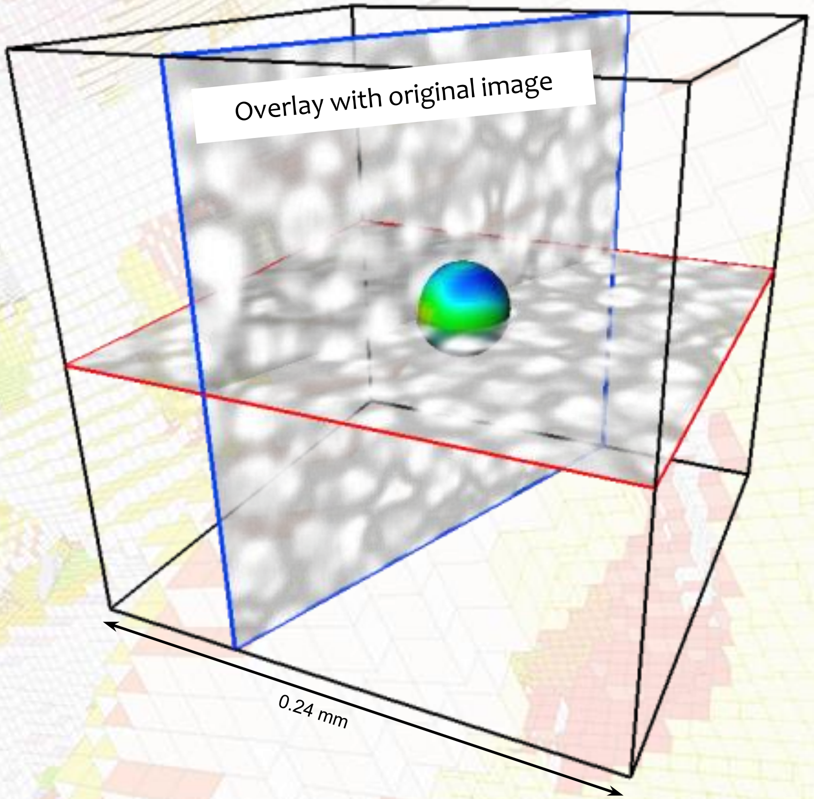


PMMA sample

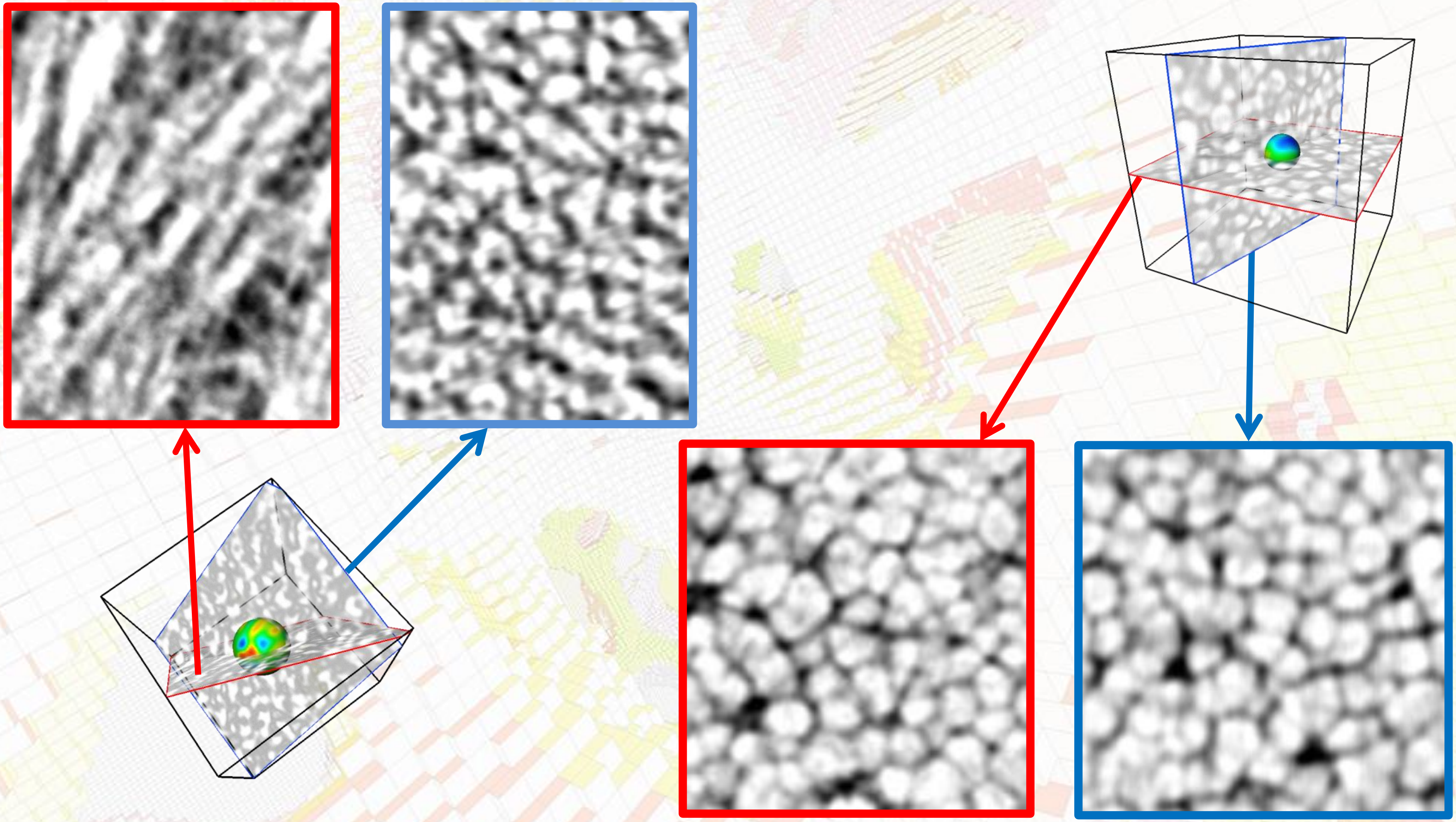


Polymer foams – anisotropy





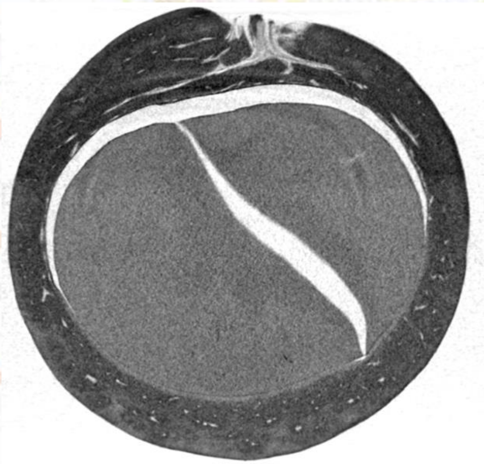
Polymer foams – anisotropy

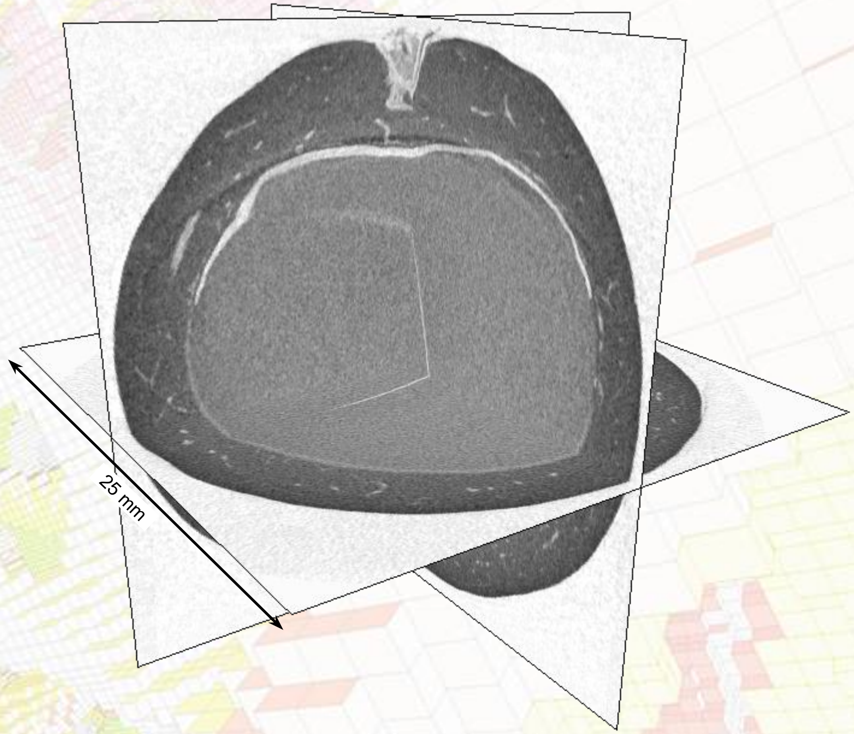


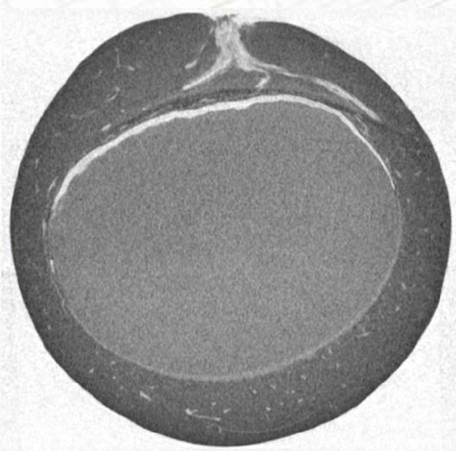
Macadamia nuts

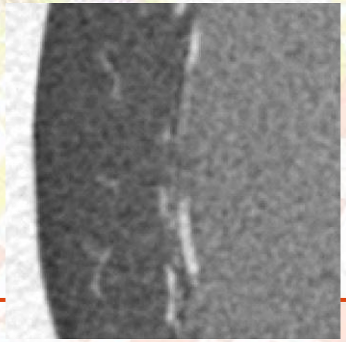
Image analysis AND taste test

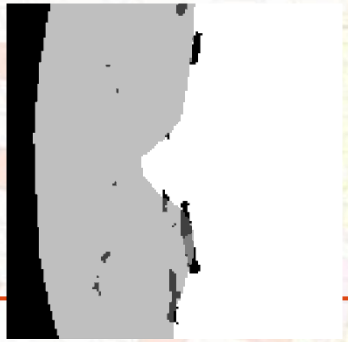
Macadamia nuts



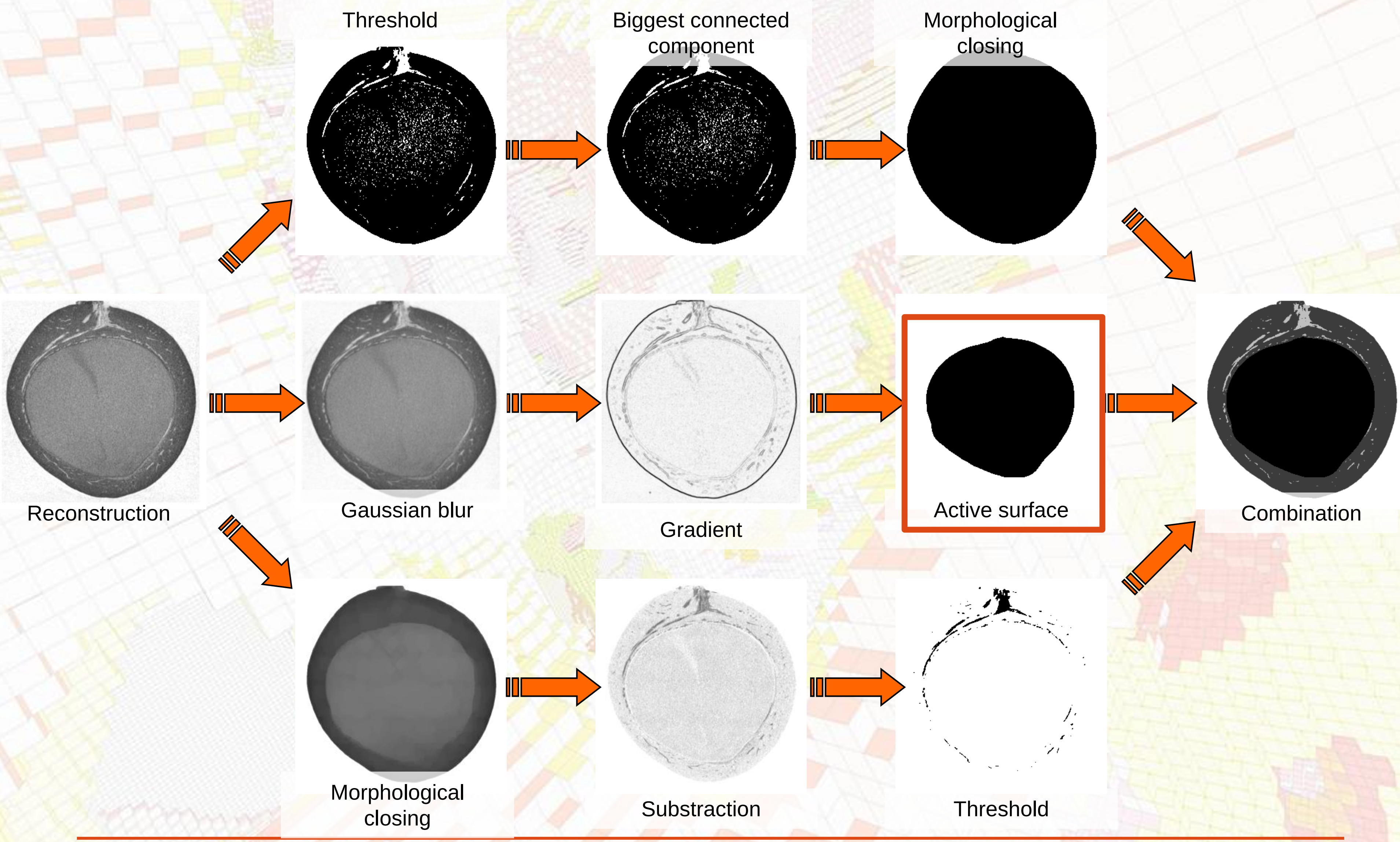








Macadamia nuts - Segmentation



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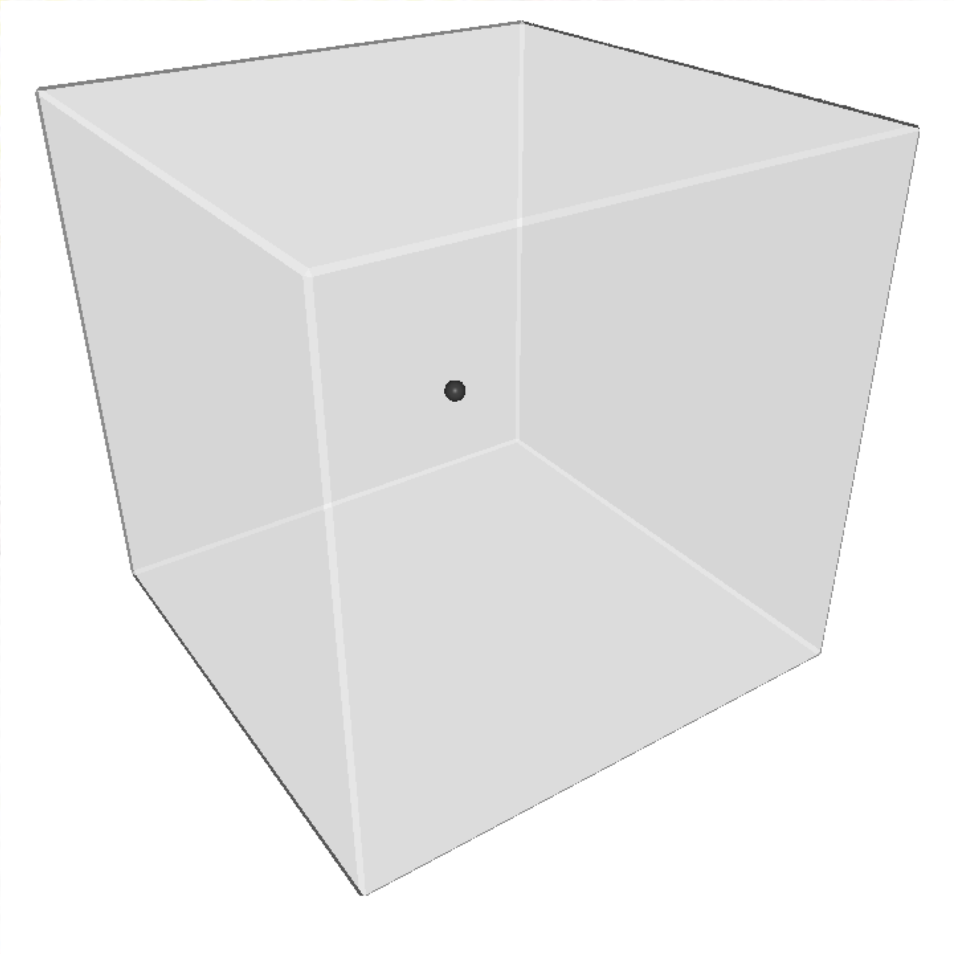
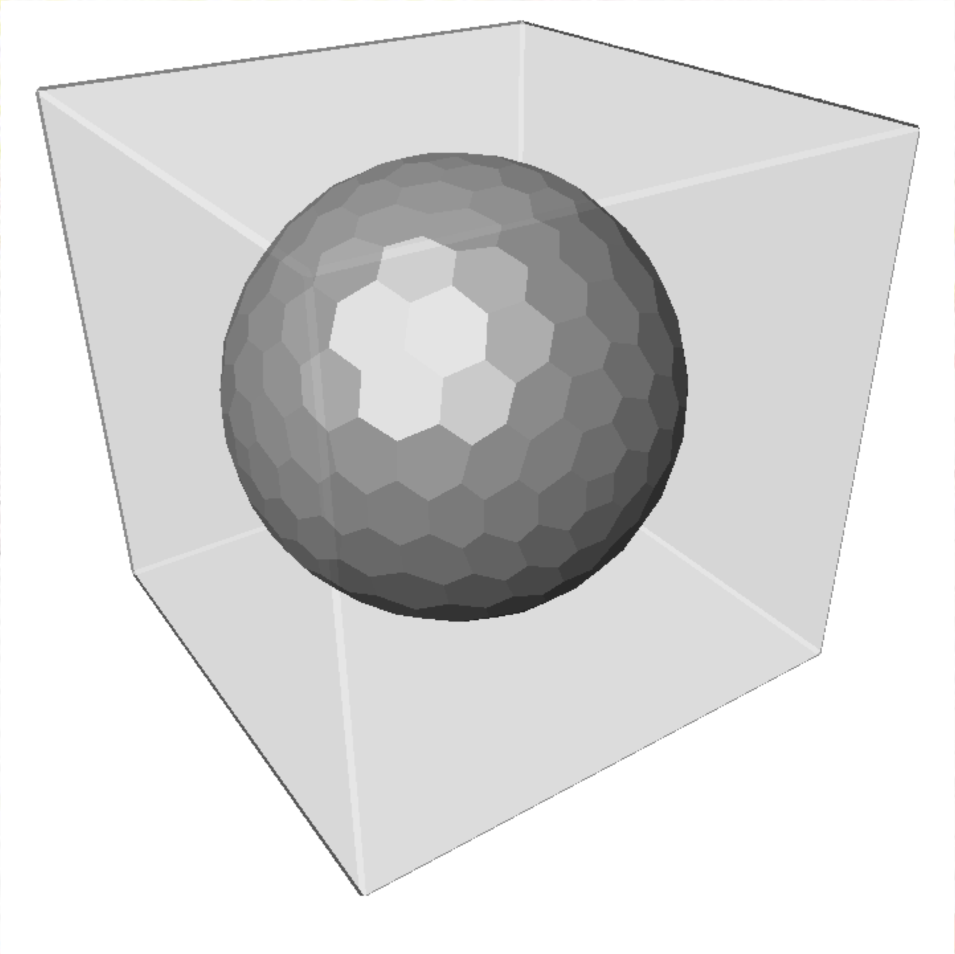
Kernel segmentation : active surface



Hervé Delingette, *Modélisation, déformation et reconnaissance d'objets tridimensionnels à l'aide de maillages simplexes*, PhD thesis, Ecole centrale de Paris, 1994

Active surface



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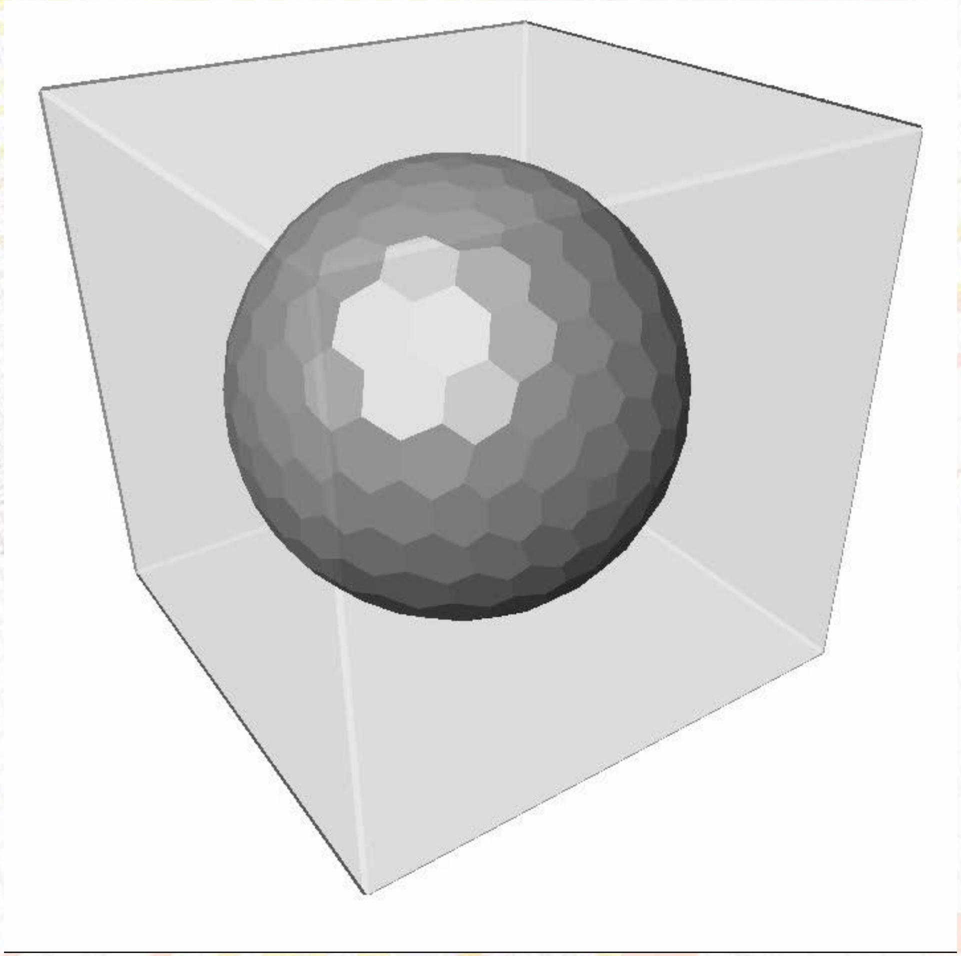
Active surface



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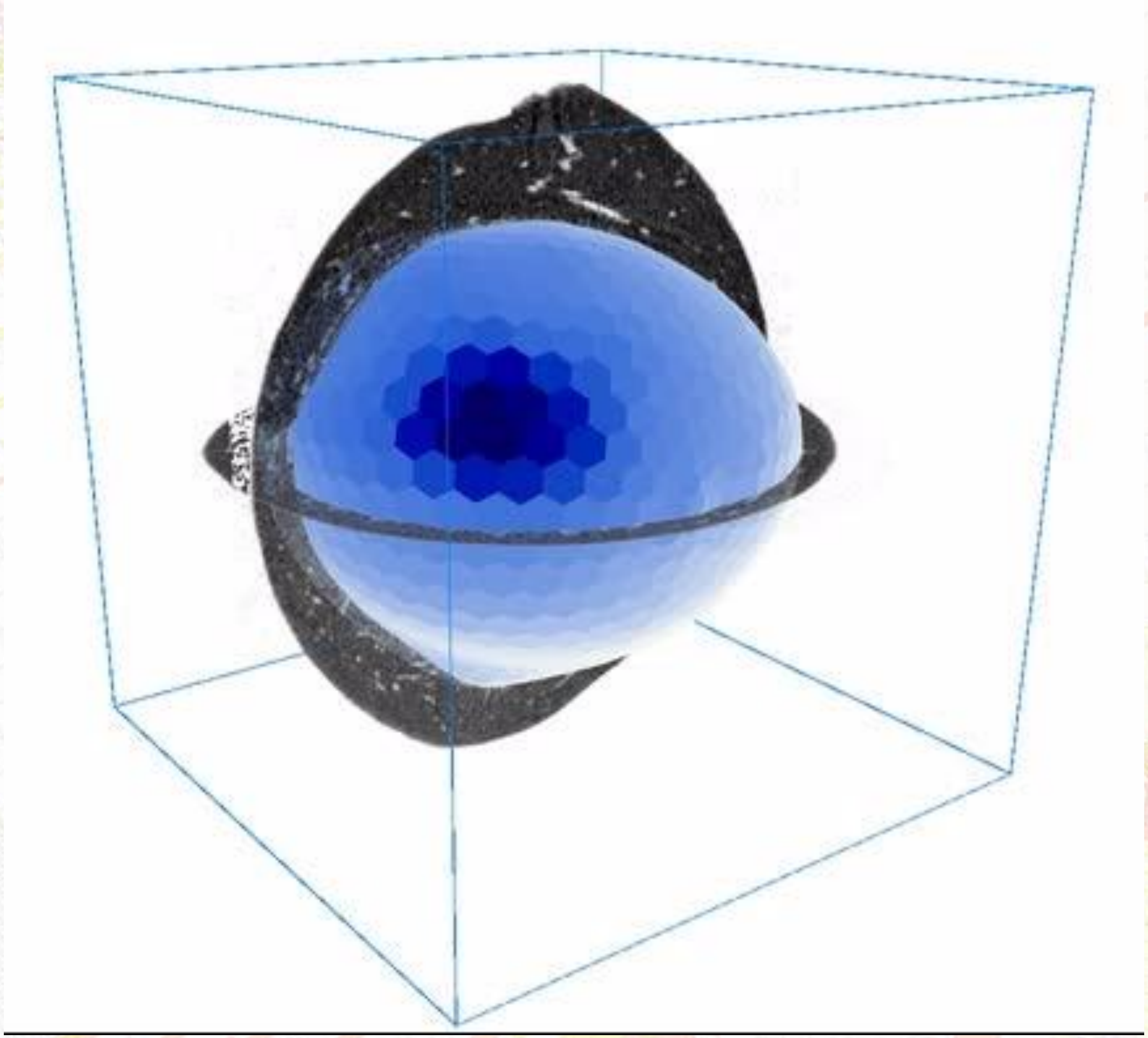


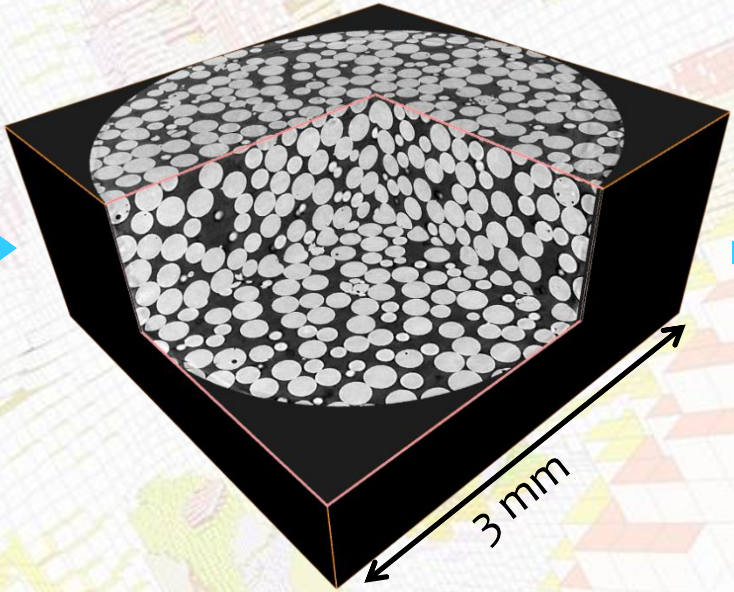
Segmentation result

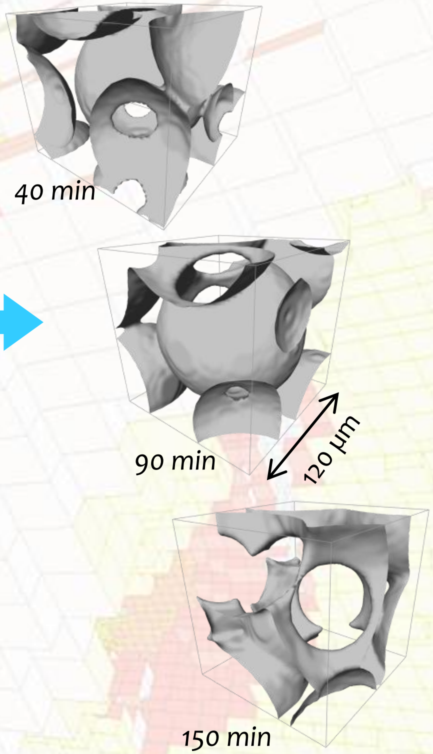
Additional component ?

Sintering of soda-lime glass

Or what is a pore ?

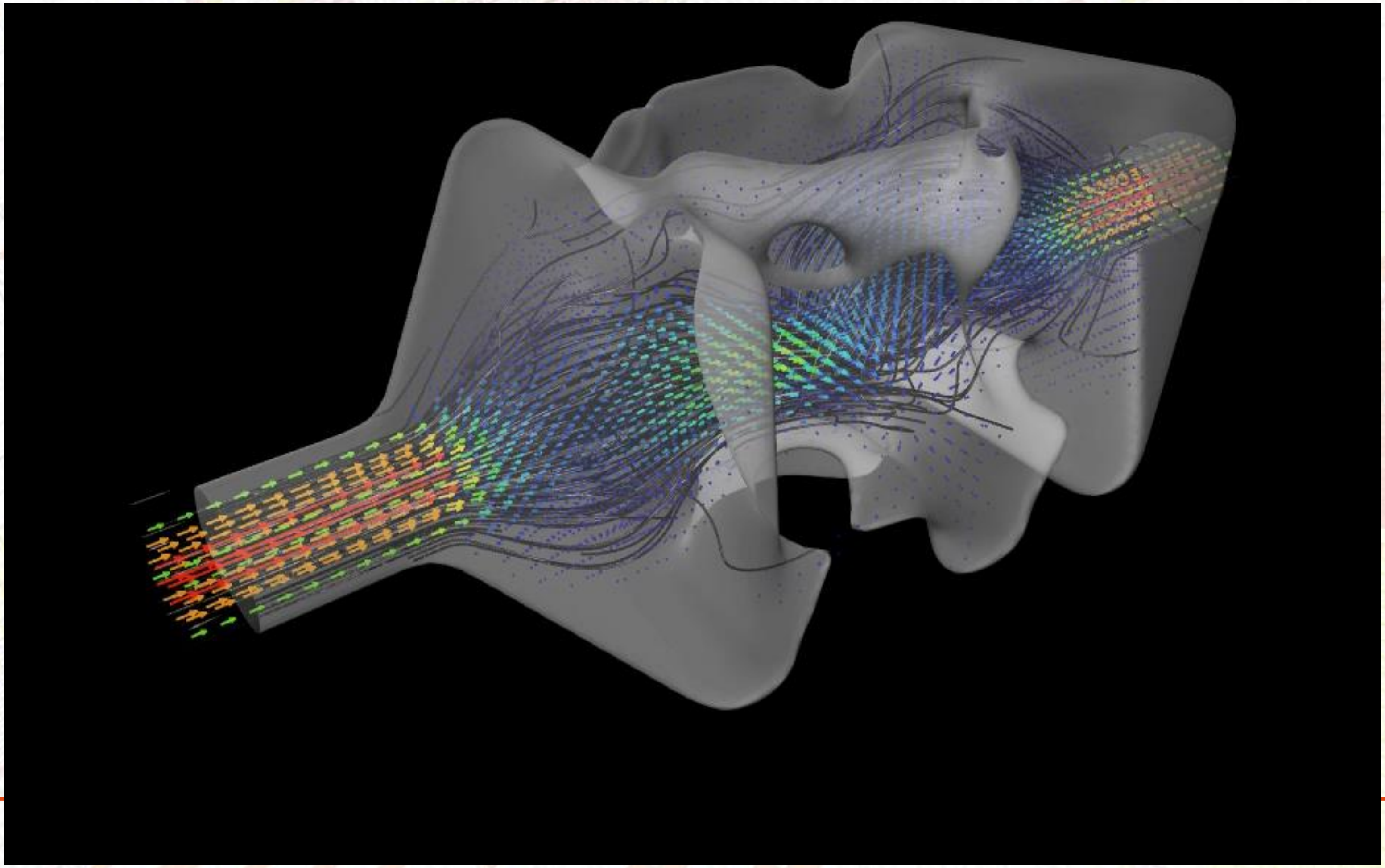
Sintering and transfer properties





Pore network models

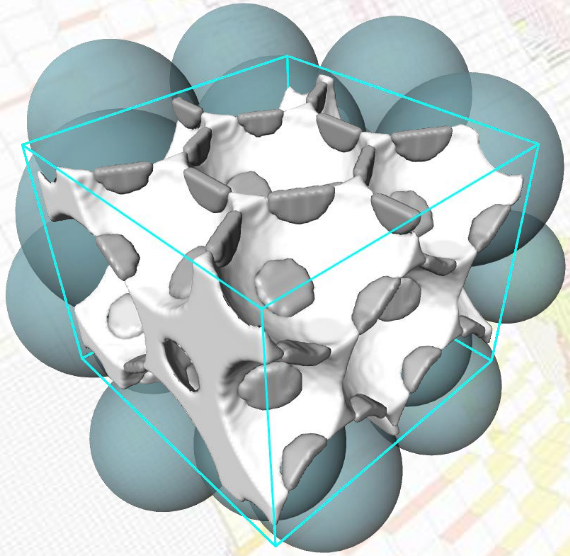
$$\begin{cases} \mu\vec{\nabla}\vec{\nabla}.\vec{V} - \vec{\nabla}P & = & 0 \\ \vec{\nabla}.\vec{V} & = & 0 \end{cases}$$

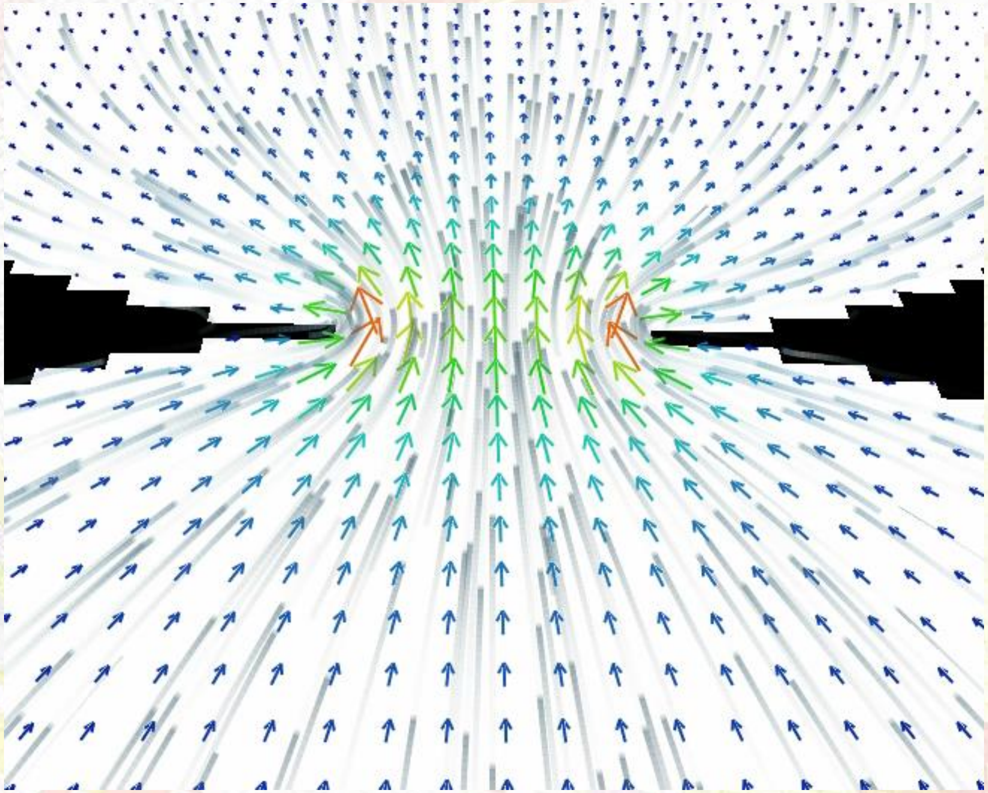


Pore network models

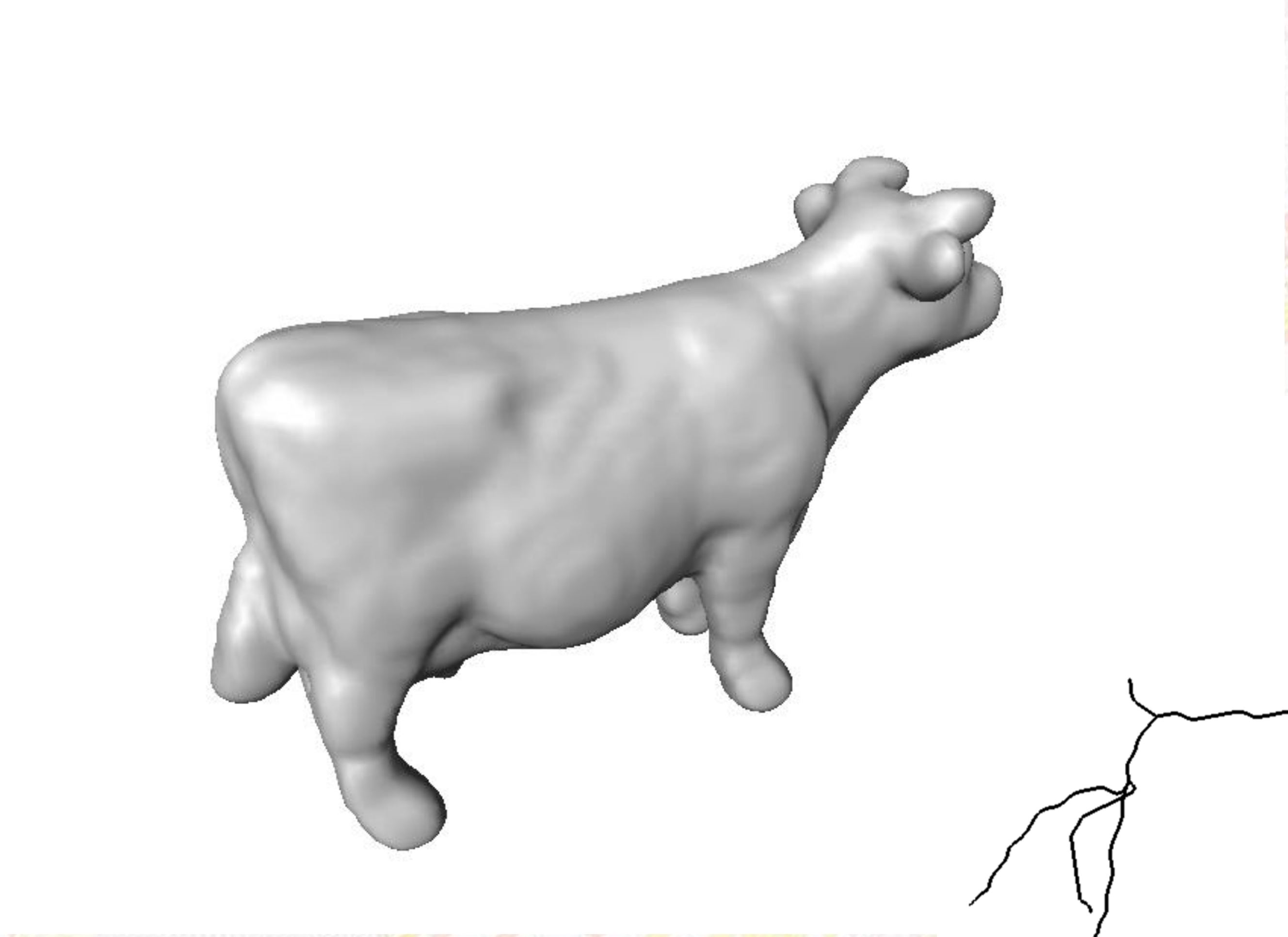
Classical definition: "part of the pore space bounded by the solid and planes erected where the hydraulic radius is minimal"

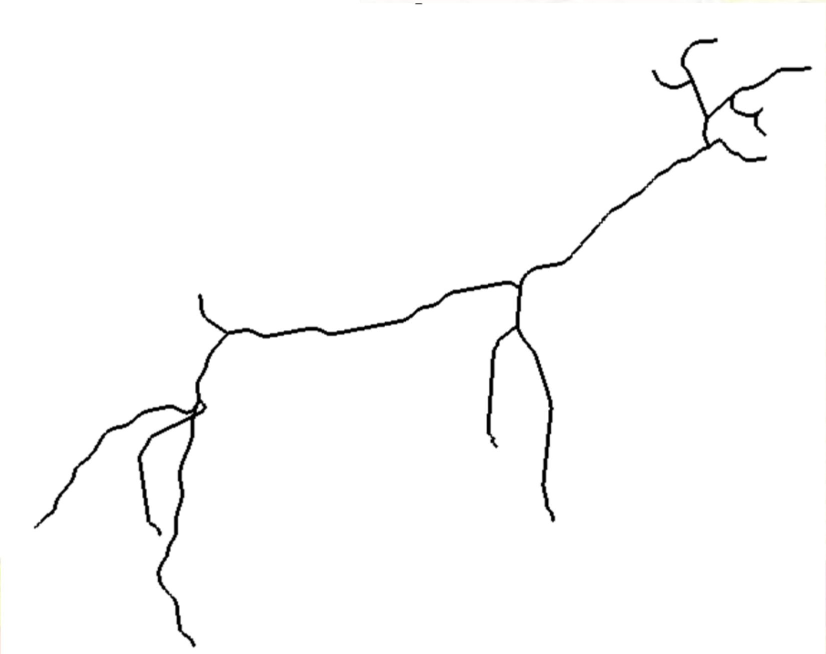
F.A. Dullien, *Porous Media: Fluid Transport and Pore Structure,* 1991



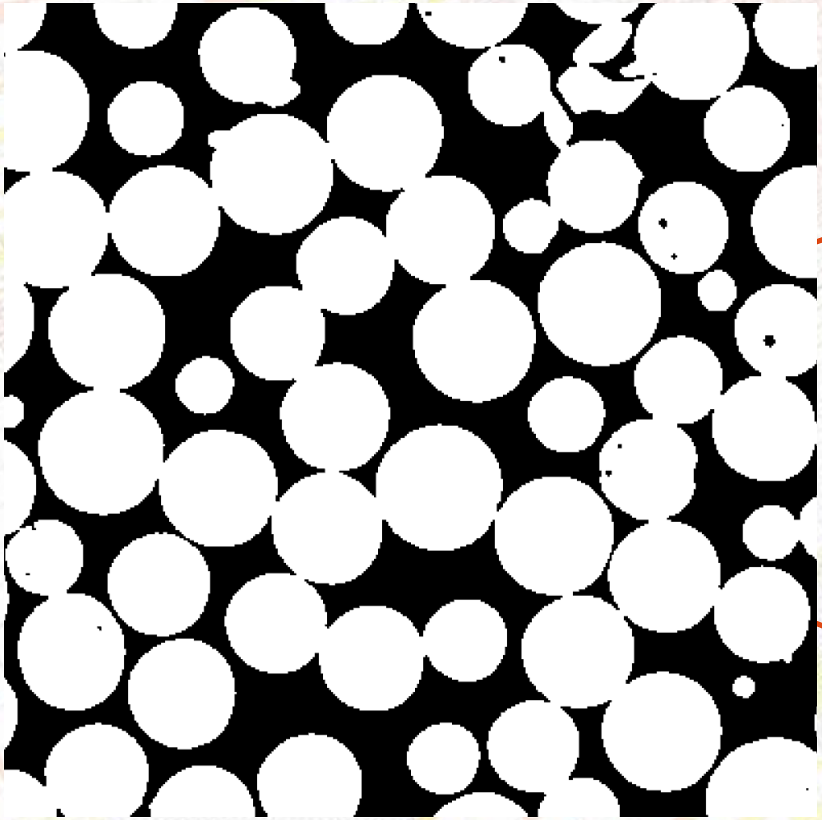


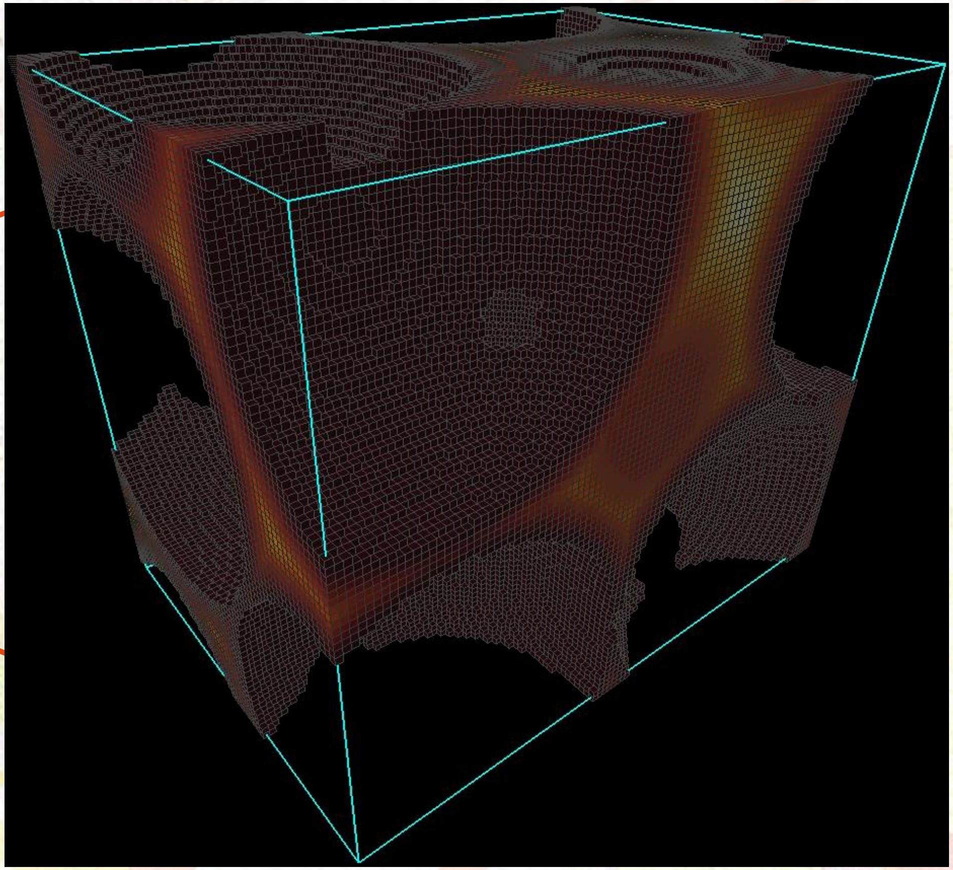
Pore decomposition - skeletonisation





Distance-ordered homotopic thinning





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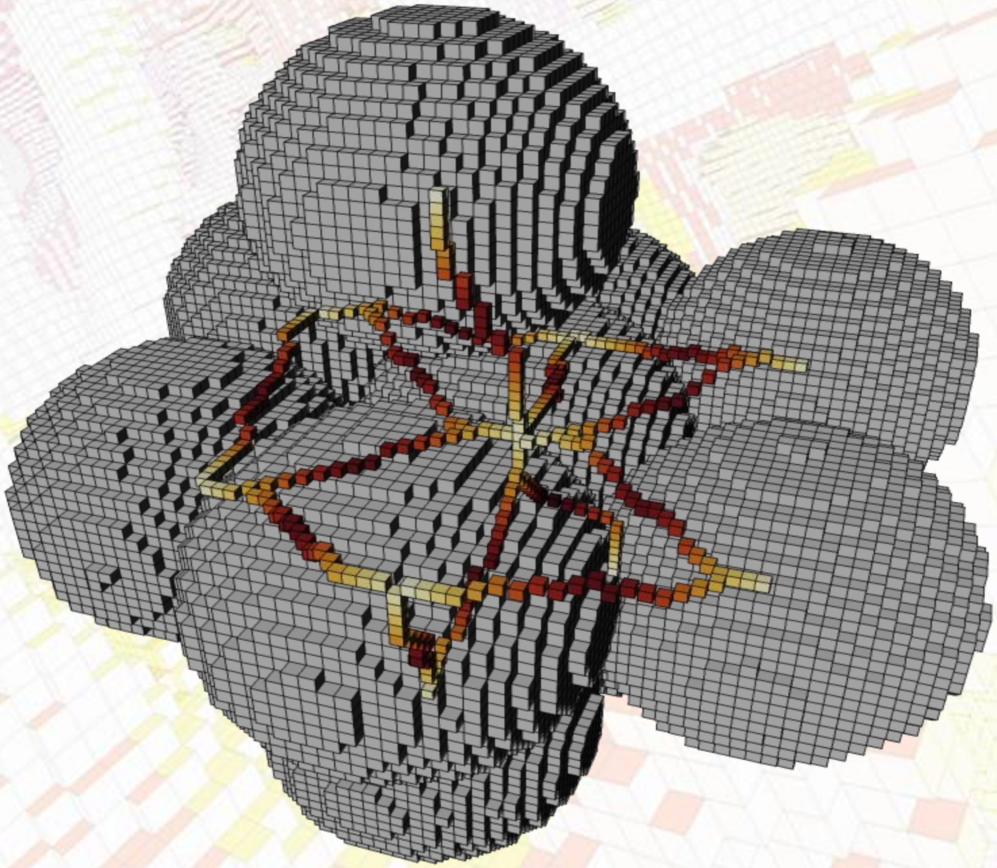
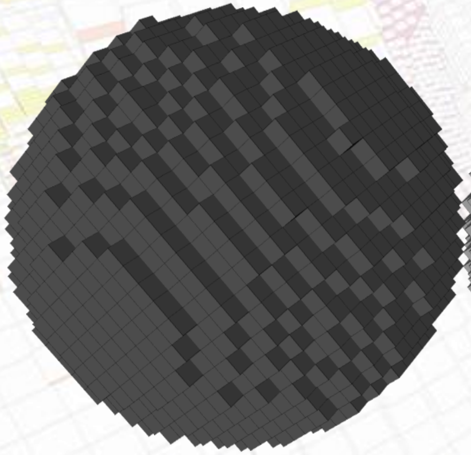
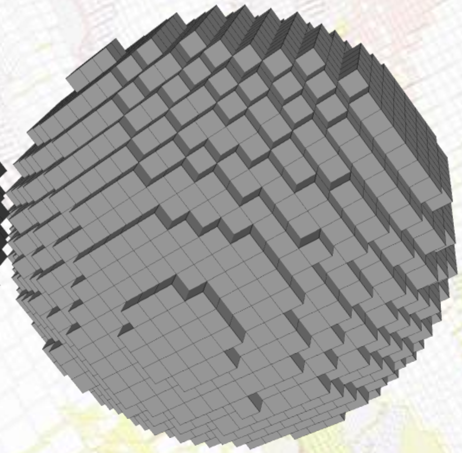
Before skeletonisation

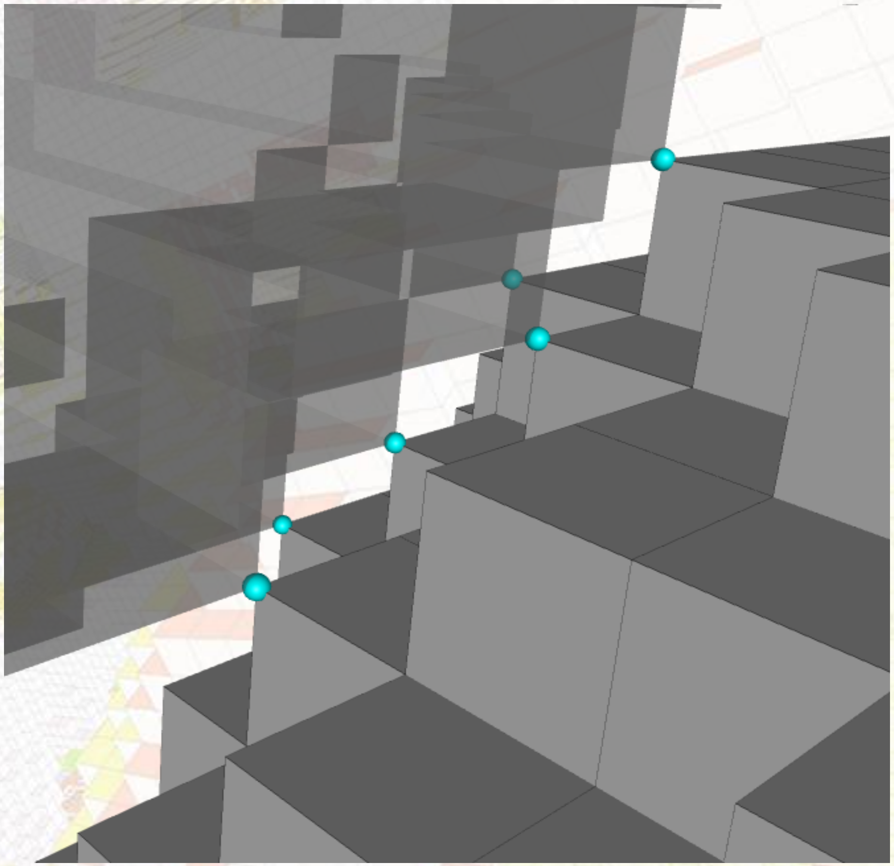
Inconvenience : sensitivity to small features (noise, misclassified pixels, digitisation, etc.)



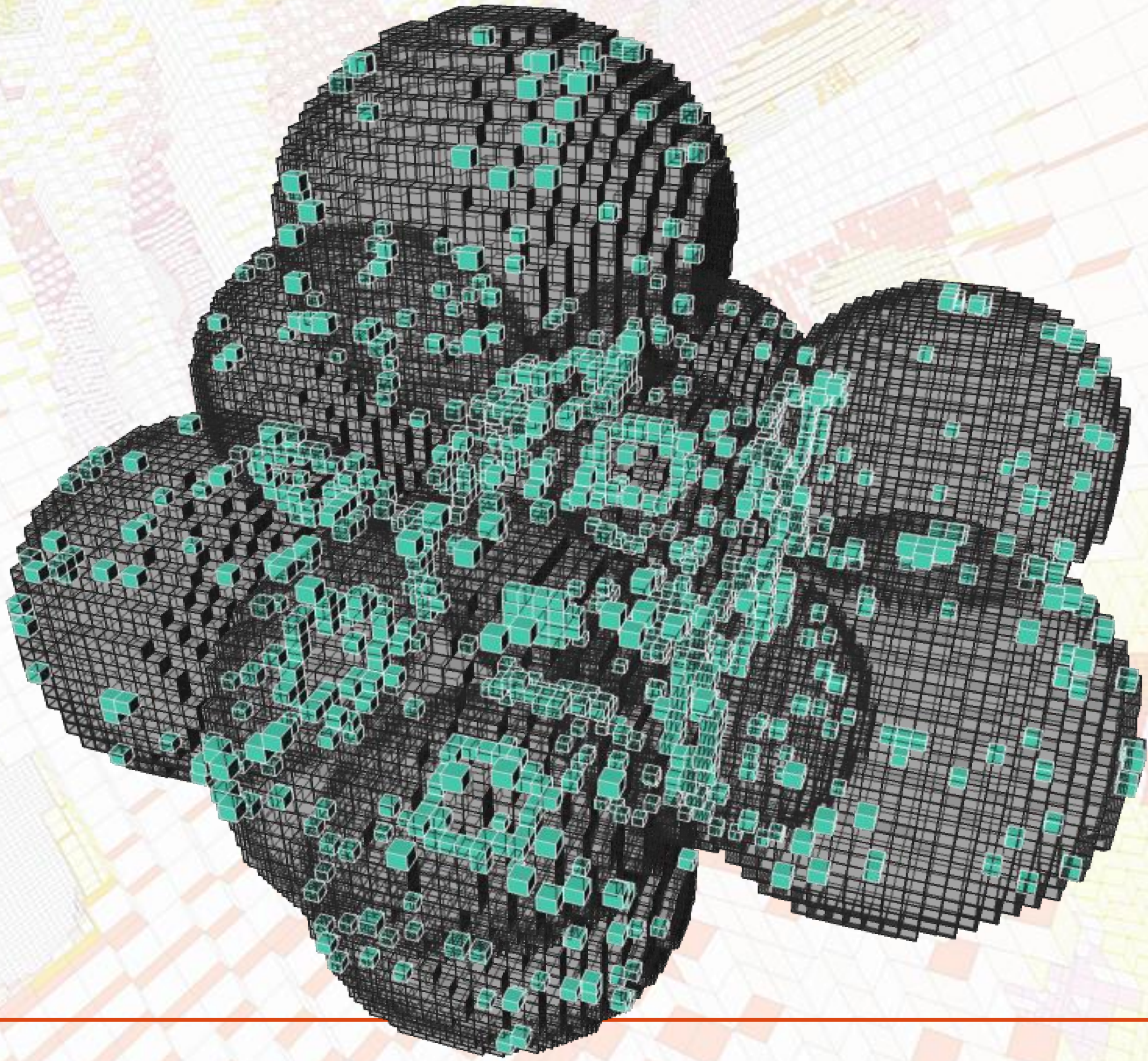
Topological artefacts



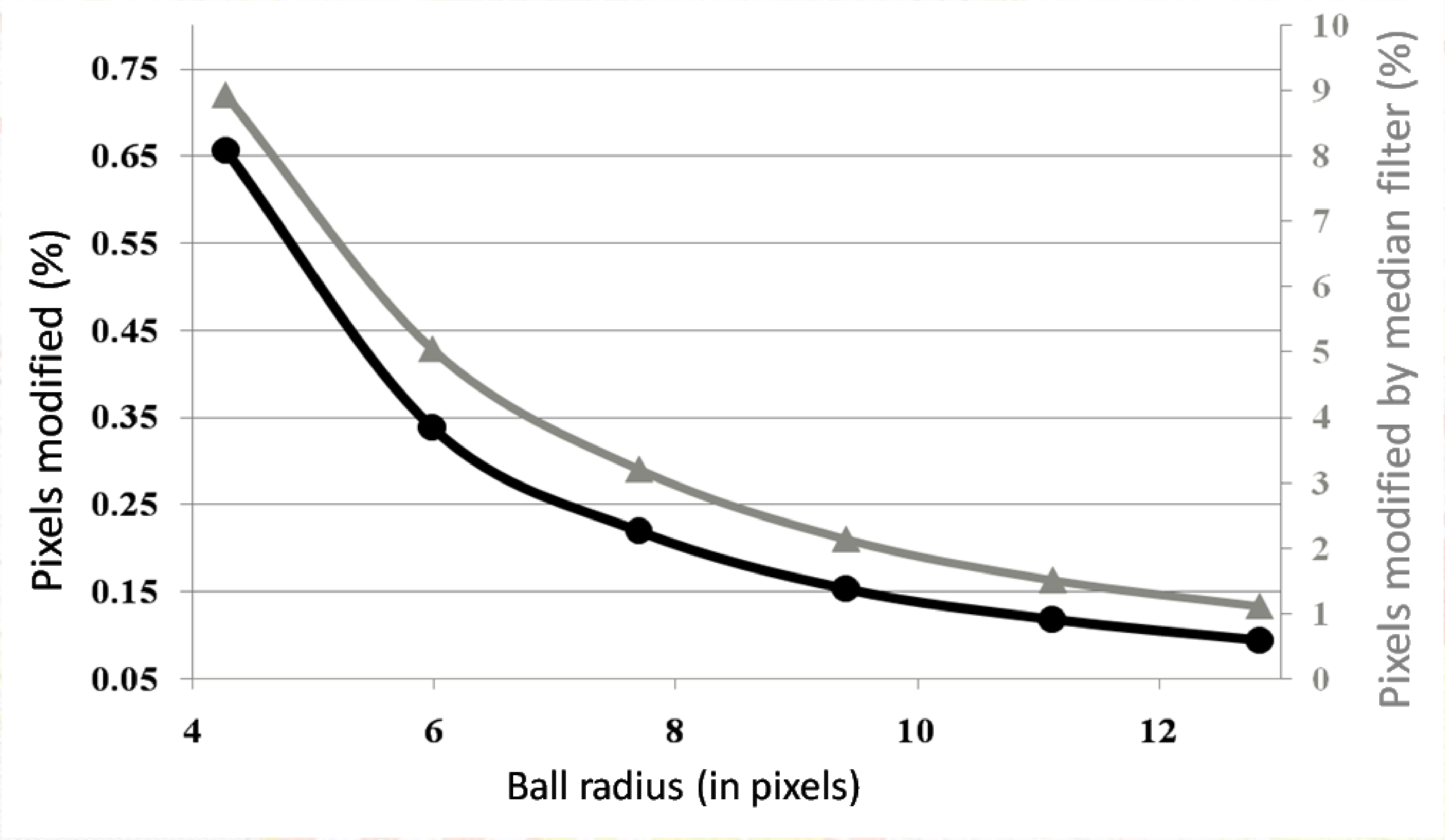




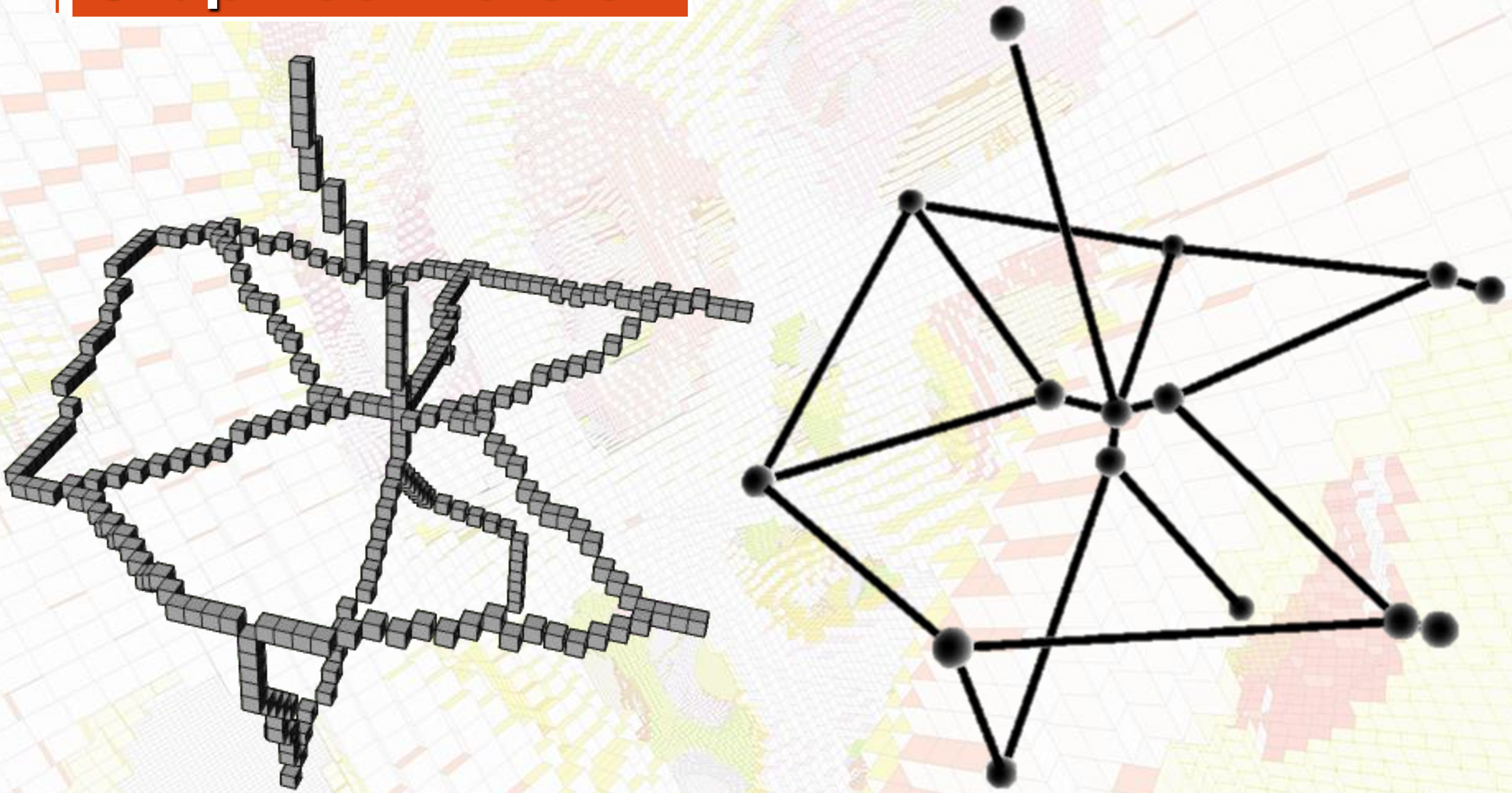
Filtering



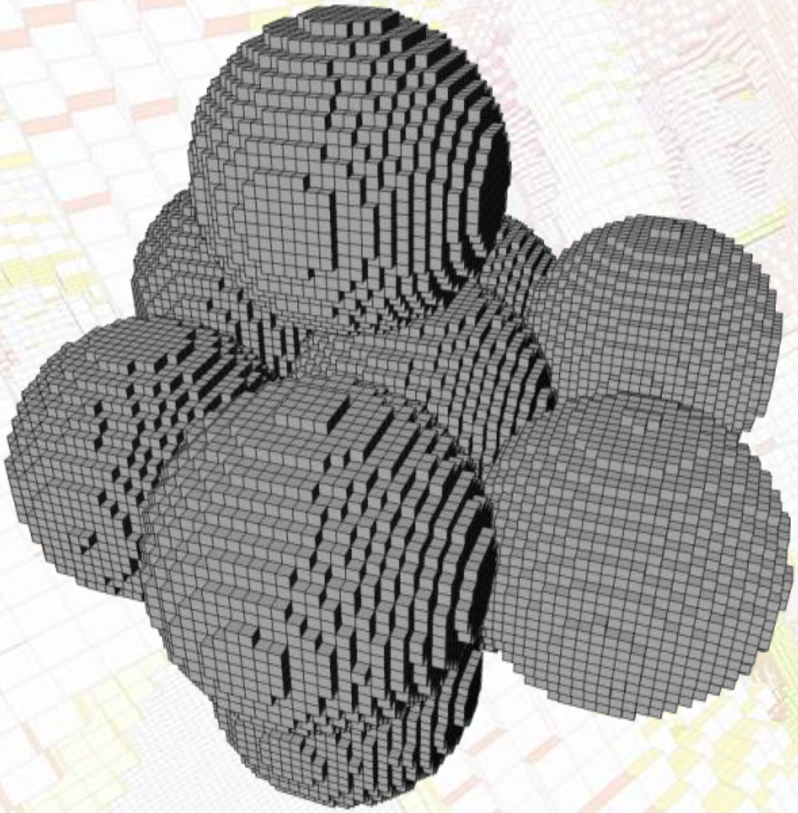
Filtering

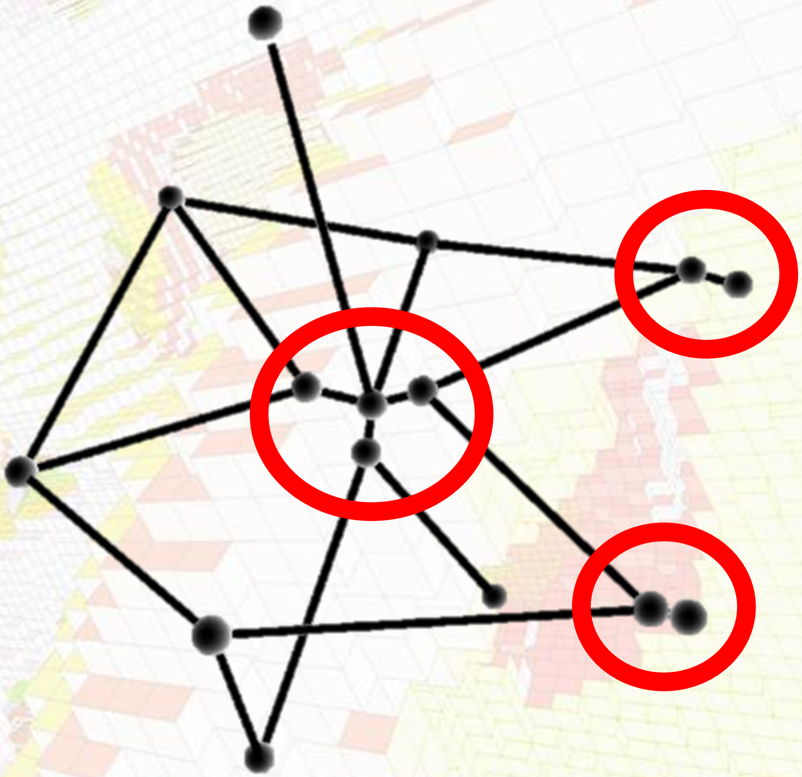

0.75
0.65
0.55
0.45
0.35
0.25
0.15
0.05
Pixels modified (%)
4
6
8
10
12
Ball radius (in pixels)
10
9
8
7
6
5
4
3
2
1
0
Pixels modified by median filter (%)

Graph conversion

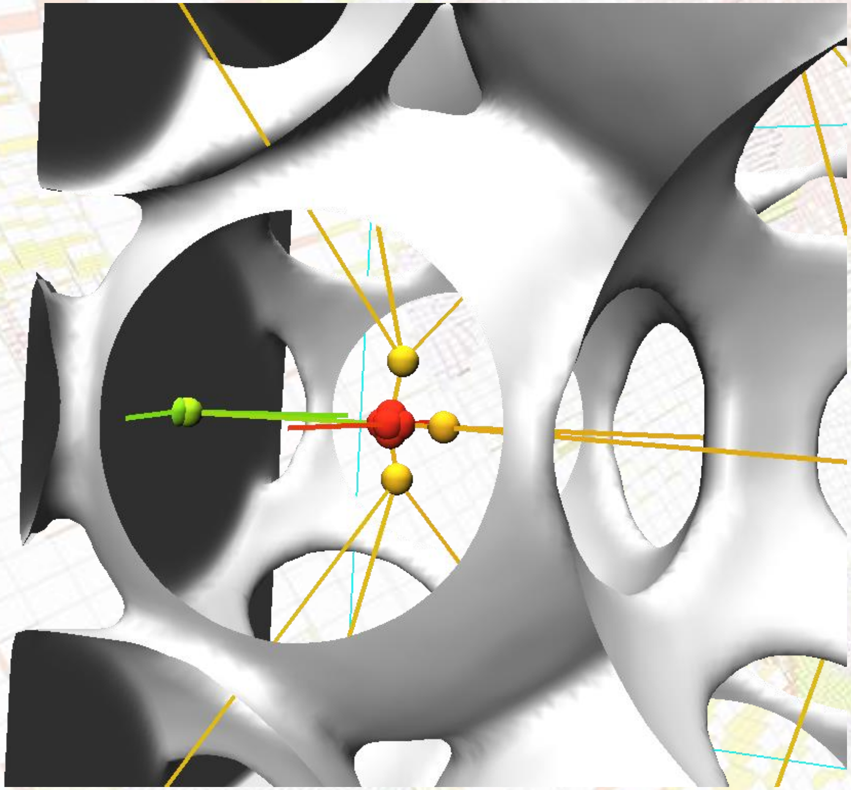


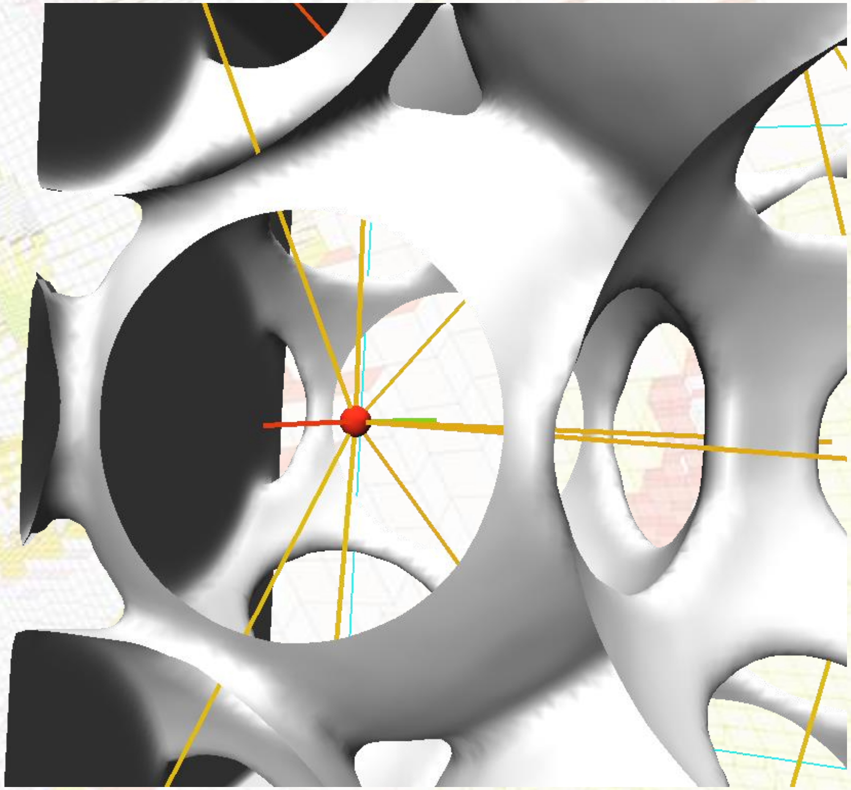
Graph post-processing



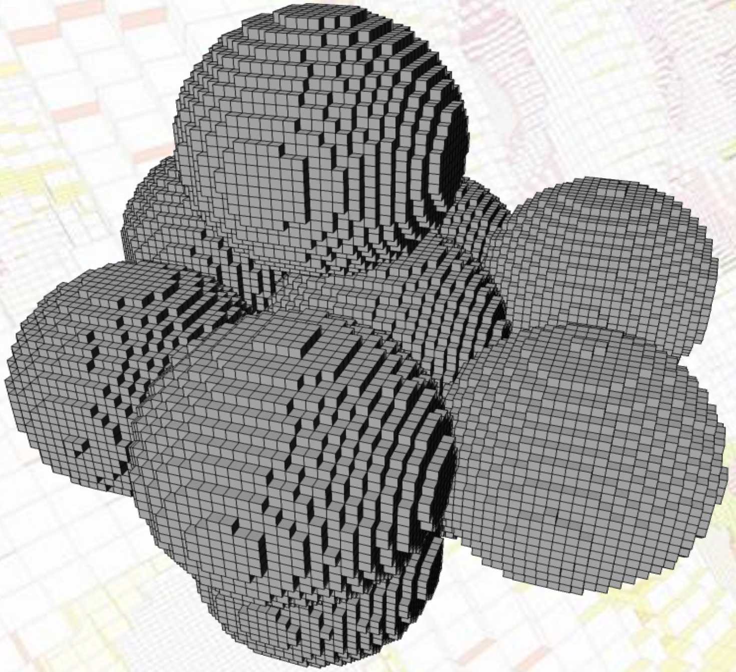


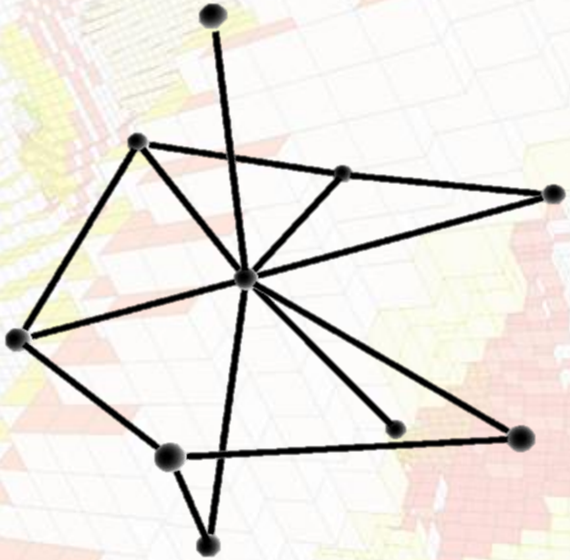
Graph post-processing





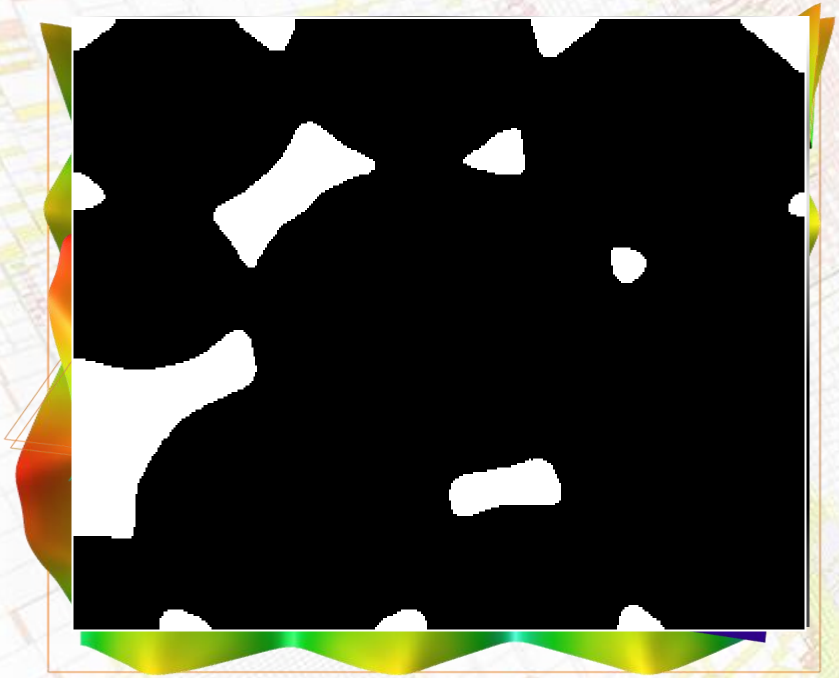
Graph post-processing

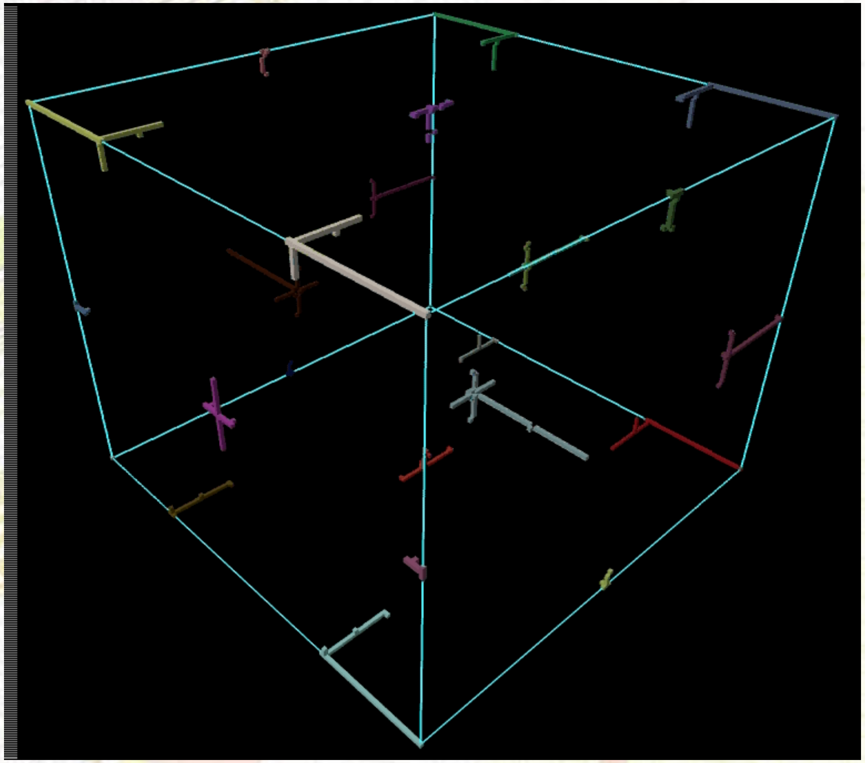




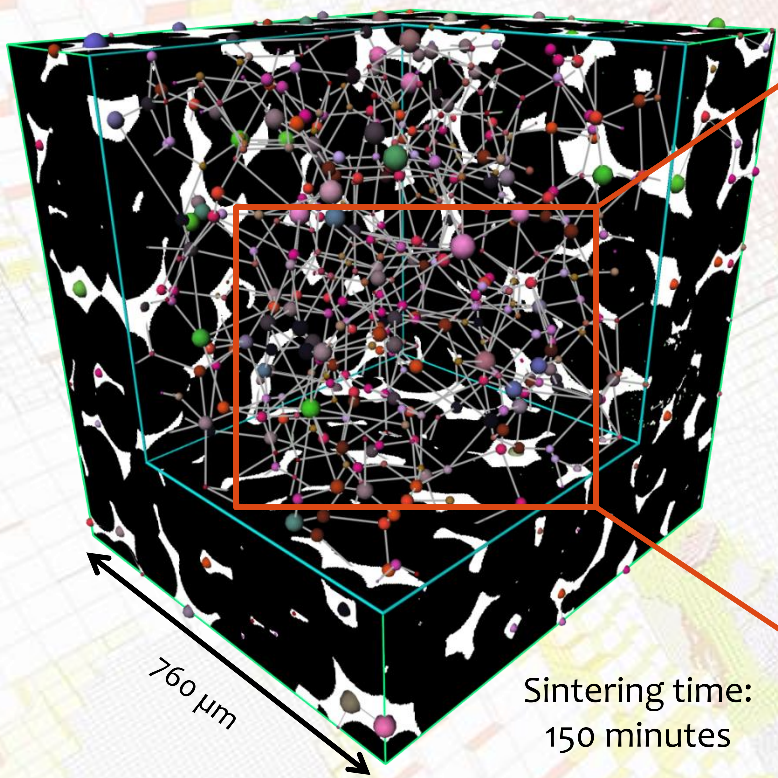
Pore delimitation

Region growth approach : the watershed

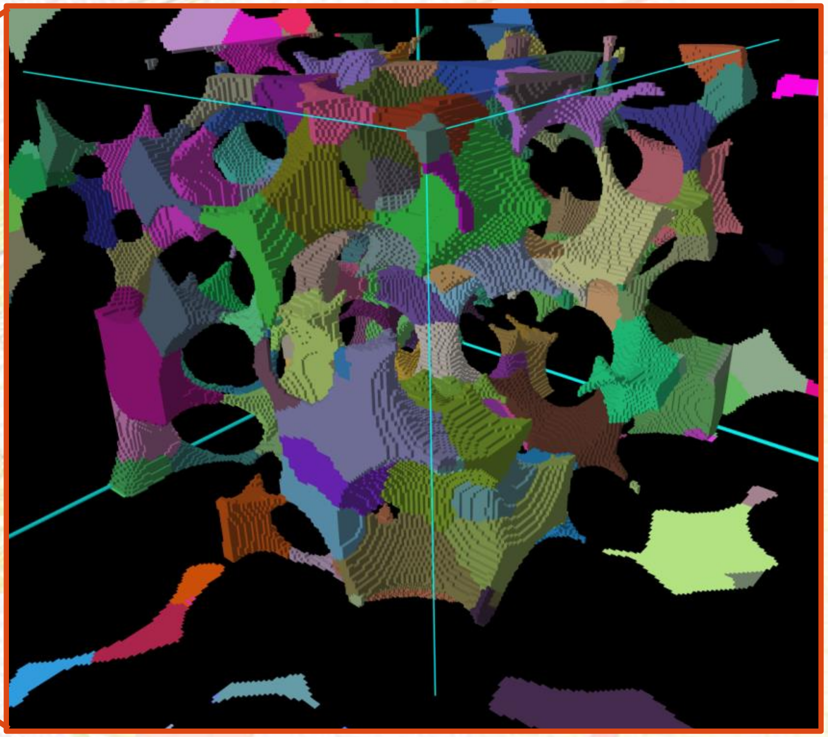




Resulting pore decomposition



Sintering time:
150 minutes



Statistics

Pores																			
Label	Number of pixels	On border	Mean x-coordi	Mean y-coordi	Mean z-coordin	Surface faces	Connection faces	a	Ax	Ay	Az	b	Bx	By	Bz	c	Cx	Cy	Cz
13077	78971	1	384.73538	42.296162	32.046627	40998	0	1.67E+08	0.13036	0.9812	-0.14231	1.53E+08	-0.00877	-0.14239	-0.98977	2.7E+07	0.99143	-0.13027	0.009
13005	4891	1	512.33368	36.082806	13.758331	3910	0	1568090	-0.30067	-0.88227	0.36221	1528012	-0.46461	0.46717	0.75226	107330	-0.83291	0.05789	-0.550
12669	769	1	544.16644	44.936279	4.0650196	976	0	40019.1	0.26266	-0.1563	-0.95214	37551.6	-0.31392	-0.94695	0.06885	5640	-0.91239	0.28081	-0.297
1	1	1	318	44	0	6	0	0	0	1	0	0	1	0	0	0	0	0	
8250	11	1	319.27274	47	0.27272728	38	0	17	-0.70711	-8.05E-08	-0.70711	15.3636	0.70711	-1.00E-07	-0.70711	4.36364	1.39E-08	1	-1.28E-
8794	15	1	557.73334	47.466671	0.40000004	44	0	21.0552	0.03768	0.20284	0.97849	15.578	-0.96434	0.26409	-0.01761	11.9001	0.26198	0.94293	-0.205
6153	4	1	412.5	54.5	0	16	0	2	0	0	-1	1	0	-1	0	1	-1	0	
8524	13	1	465.69232	55.769234	0.30769232	40	0	17.2661	0.11361	0.09262	0.9892	14.8505	0.85965	-0.50831	-0.05114	7.57568	0.49808	0.85618	-0.137
12508	506	1	589.85577	60.314232	1.7786562	702	0	13460.5	-0.07911	0.09292	0.99253	8202.04	0.79838	-0.5903	0.1189	7346.93	-0.59693	-0.80182	0.027
12672	779	1	281.87164	59.671375	4.5879335	862	0	20703.9	-0.04217	0.32088	0.94618	16296.3	0.55937	-0.7771	0.28847	9923.33	0.82784	0.54143	-0.146
10363	37	1	363.91891	65.972977	0.4054054	92	0	175.741	0.0186	-0.00632	0.99981	162.459	0.78401	-0.62047	-0.0185	31.0974	0.62047	0.7842	-0.006
13065	14863	1	577.98248	54.021191	29.91011	11012	0	5690970	0.18625	-0.85668	-0.48104	4543964	0.00355	-0.48902	0.87226	2542938	0.9825	0.16417	0.088
4189	2	1	280.5	69	0	10	0	0.5	0	-1	0	0.5	0	0	-1	0	1	0	
8069	10	1	368.60001	72.300003	0.30000001	34	0	10.6515	-0.63481	0.11141	0.76459	10.2939	-0.74673	0.16578	-0.64414	4.25464	-0.19851	-0.97985	-0.022
2	1	1	470	73	0	6	0	0	0	1	0	0	1	0	0	0	0	0	
11709	126	1	300.4921	77.150795	1.484127	206	0	923.835	0.29354	0.61994	-0.72767	807.977	0.07197	-0.77338	-0.62984	378.378	0.95323	-0.13251	0.271
12680	805	1	450.37018	79.18882	3.7031054	934	0	35843.6	0.524	0.61173	-0.59262	31689.6	0.20491	0.58482	0.78486	7372.87	-0.8267	0.5327	-0.1
12276	291	1	588.1615	77.920967	1.862543	384	0	5224.39	0.20872	0.11476	-0.97122	4774.38	-0.49324	-0.84518	-0.20587	1411.4	-0.84448	0.52201	-0.11
7377	7	1	289.28574	76.714287	0.2857143	26	0	5.56427	0.21848	0.69002	-0.69002	4.28571	0	-0.70711	-0.70711	2.72145	0.97584	-0.15449	0.154
12794	1220	1	472.11639	86.228691	11.755737	1416	0	94368	0.45619	-0.72449	0.51673	81307.4	0.88527	0.42855	-0.1807	21664.6	-0.09053	0.53988	0.836
14821	43130	1	281.77191	92.596497	29.613632	20046	1654	3E+07	0.62472	0.51076	-0.59064	2.6E+07	0.31557	0.52674	0.78928	9674455	-0.71424	0.67947	-0.167
14047	10743	1	235.56668	93.014145	9.6889133	5602	994	1724845	0.06974	0.36066	-0.93009	1514552	0.23459	0.90028	0.36669	704074	0.96959	-0.24376	-0.021
13130	4368	1	649.6687	95.20536	19.117903	5152	202	1518851	0.80404	-0.41887	-0.42198	1290993	-0.49159	-0.86755	-0.07551	304547	0.33446	-0.26816	0.903
7628	8	1	489.5	83	0.25	28	0	6	0	0	-1	5.5	-1	0	0	3.5	0	-1	
14383	3637	1	420.0737	109.3129	5.6565852	2462	67	312231	0.42084	-0.52019	0.74317	282207	-0.71191	0.31835	0.62597	102531	-0.56221	-0.7925	-0.236
14050	3750	1	216.0312	105.24773	11.266666	2932	864	339913	0.10199	-0.96556	0.23936	325572	0.59309	-0.13416	-0.79388	138532	0.79865	0.22293	0.558
3	1	1	408	110	0	6	0	0	0	1	0	0	1	0	0	0	0	0	
7062	6	1	178.33334	118.33334	0.5	22	0	3.5	-0.70711	-0.70711	0	2.66667	0	0	-1	2.16667	0.70711	-0.70711	
14289	8325	1	693.12445	135.12709	17.977777	5206	514	935830	-0.23406	-0.84683	0.47759	728961	0.85112	0.05895	0.52166	672760	0.46391	-0.52859	-0.706
10232	34	1	261.14706	136.94118	0.61764705	80	0	94.5503	0.20582	-0.29875	0.93187	85.1173	0.12651	-0.93615	-0.32806	36.6853	-0.97038	-0.18542	0.154
14295	20026	1	584.87561	154.91336	19.458803	11190	40	7939989	0.12874	0.80746	0.5757	7524395	0.91593	-0.31934	0.24307	2856260	0.38012	0.49601	-0.78
14640	3070	1	455.52899	145.34755	10.971009	2114	408	151093	-0.31095	-0.84932	0.42658	120120	0.91779	-0.15171	0.36695	112834	0.24694	-0.50561	-0.826
12637	722	1	274.69391	149.17728	2.9141273	820	0	33732.4	0.36995	-0.87902	0.30078	32456.8	0.06093	0.34601	0.93625	5169.43	0.92705	0.32804	-0.18
14294	17	1	606.64703	145.05882	0.23529412	66	34	82.8501	-0.01442	0.08708	0.9961	79.6922	0.104	-0.99067	0.08812	9.22248	0.99447	0.10487	0.005
4	1	1	324	145	0	6	0	0	0	1	0	0	1	0	0	0	0	0	
8912	16	1	588.3125	147.6875	0.375	44	0	23.419	0.1365	0.23754	0.96174	19.0013	-0.1228	0.96739	-0.22151	10.8297	0.983	0.08786	-0.161
8913	16	1	594.8125	151.5625	0.25	48	0	28.6648	0.21989	0.0309	0.97504	22.7963	0.96916	0.1071	-0.22195	11.2889	-0.11129	0.99377	-0.00
12858	1724	1	690.20819	158.62645	14.168793	1462	0	93121.9	-0.38077	0.58498	-0.71611	79103.6	-0.67156	-0.70732	-0.22072	32293.5	-0.63563	0.39686	0.662
11694	124	1	296.05646	156.64516	1.4516128	204	0	800.713	-0.23415	-0.50009	-0.83371	597.111	0.94613	0.08003	-0.31373	453.579	-0.22361	0.86226	-0.454
4190	2	1	392	155.5	0	10	0	0.5	-1	0	0	0.5	0	0	-1	0	0	1	
8914	16	1	461.375	161.5625	0.3125	48	0	23.8323	0.04399	0.11637	-0.99223	17.7519	-0.96003	-0.26989	-0.07421	12.6658	0.27643	-0.95583	-0.099
13042	7979	1	642.8573	169.68369	22.433264	7110	0	5986547	0.50156	-0.79949	0.33052	4919289	-0.31111	-0.52318	-0.7934	1335178	-0.80724	-0.29511	0.51
12962	3168	1	741.43463	179.01199	6.8636365	3068	0	413524	0.88248	-0.32497	-0.34005	345913	-0.18113	0.4324	-0.8833	144309	-0.43408	-0.84109	-0.322
11359	87	1	750.32184	167.58621	0.98850572	180	0	1191.98	0.39184	0.53513	-0.7484	1163.43	-0.54198	-0.52306	-0.65777	150.75	-0.74345	0.66336	0.085
11943	168	1	300.3512	167.90477	1.6904762	250	0	1044.82	-0.99052	0.13236	0.03671	976.679	0.03759	0.00411	0.99928	795.819	0.13212	0.99119	-0.009
12158	234	1	763.64532	176.62395	1.4444445	330	0	2937.15	0.22208	-0.3903	0.8935	2378.21	-0.65366	0.62036	0.43345	1073.13	-0.72347	-0.68031	-0.117
12988	4083	1	332.96524	179.65663	9.057066	2448	0	293172	0.001	0.77625	-0.63042	233132	-0.52951	-0.53437	-0.65883	143426	-0.8483	0.33448	0.41
12220	262	1	373.94275	188.34351	1.4198474	386	0	4093.74	0.00905	0.26434	0.96439	3508.67	-0.91408	0.39321	-0.0992	1319.67	-0.40543	-0.88063	0.245
12052	137	1	314.89847	188.72589	1.2944162	268	0	1955.36	-0.07261	0.28036	-0.95715	1477.8	0.23797	0.93683	0.25636	919.013	0.96855	-0.20916	-0.134
14569	5437	1	513.65186	250.0732	15.986757	4780	161	3320039	0.36629	-0.15113	0.91815	3057508	0.92905	0.11445	-0.3518	355073	-0.05192	0.98187	0.182
14386	46686	1	444.50717	229.89954	33.964401	18546	3897	1.7E+07	-0.09798	0.91096	-0.40068	1.6E+07	-0.96856	-0.17981	-0.17195	1.4E+07	0.22869	-0.37124	-0.899
12997	4534	1	601.47662	205.55536	14.104764	2708	0	388958	-0.77705	0.61708	-0.12412	331101	0.60689	0.78681	0.11231	144425	-0.16697	-0.01194	0.985
11645	118	1	323.38135	205.45763	2.0254238	210	0	1033.1	-0.9804	-0.16842	0.10228	894.163	-0.13324	0.18425	-0.97381	348.843	0.14516	-0.96834	-0.203
14417	17257	1	320.33093	196.66621	29.949295	9580	214	1.8E+07	0.06729	-0.74973	-0.65831	1.8E+07	0.75965	0.46625	-0.45336	877079	0.64684	-0.46958	0.600
13459	5890	1	79.669441	250.87759	17.854839	6108	464	3610777	0.85523	0.3502	0.38204	3465844	0.14725	0.54258	-0.827	279522	0.4969	-0.76352	-0.412
14390	8950	1	372.86905	220.06281	14.401341	4444	2597	899342	0.05967	-0.88332	-0.46496	816862	0.96309	0.17342	-0.20586	544832	0.26247	-0.43552	0.861
14391	1780	1	383.84662	224.32529	7.069663	1380	695	57153	0.54169	0.47888	-0.69082	52188.8	0.05377	-0.8399	-0.54007	36480.7	0.83885	-0.25541	0.480
14441	9729	1	355.67108	225.08357	12.844999	4602	2254	1034488	-0.75453	0.43059	0.49525	778563	0.56761	0.80697	0.16317	679951	0.3294	-0.40422	0.853
14412	43039	1	344.05832	230.23145	32.035549	15816	4358	1.7E+07	-0.55985	-0.8116	0.16695	1.3E+07	-0.36729	0.42369	0.828	7931947	-0.74274	0.40224	-0.53
5437	3	1	536	221.33334	0.33333334	14	0	1.33333	-1	0	0	1	0	0.70711	0.70711	0.33333	0	0.70711	-0.70
14396	866	1	445.66858	233.1582	5.7725172	798	197	16580.6	-0.91792	-0.39506	0.03665	14871.9	0.08721	-0.29101	-0.95274	10946.2	-0.38706	0.87134	-0.30
14414	8116	1	318.81616	239.83257	10.56518	3598	1248	733385	-0.42461	-0.8868	0.17244	602225	0.70988	-0.20862	0.67272	458735	0.56194	-0.40806	-0.719
14392	1312	1	381.94815	235.07849	9.9893293	1302	295	68332.6	0.26495	0.9212	-0.28494	61419.4	-0.30428	-0.20053	-0.93124	17834.4	-0.91499	0.33343	0.22
14415	2025	1	298.40833	243.6163	4.1486421	1464	548	83018.1	-0.4815	0.23928	0.84315	76107.5	-0.59163	0.62103	-0.5141	42543.1	-0.64664	-0.74637	-0.157

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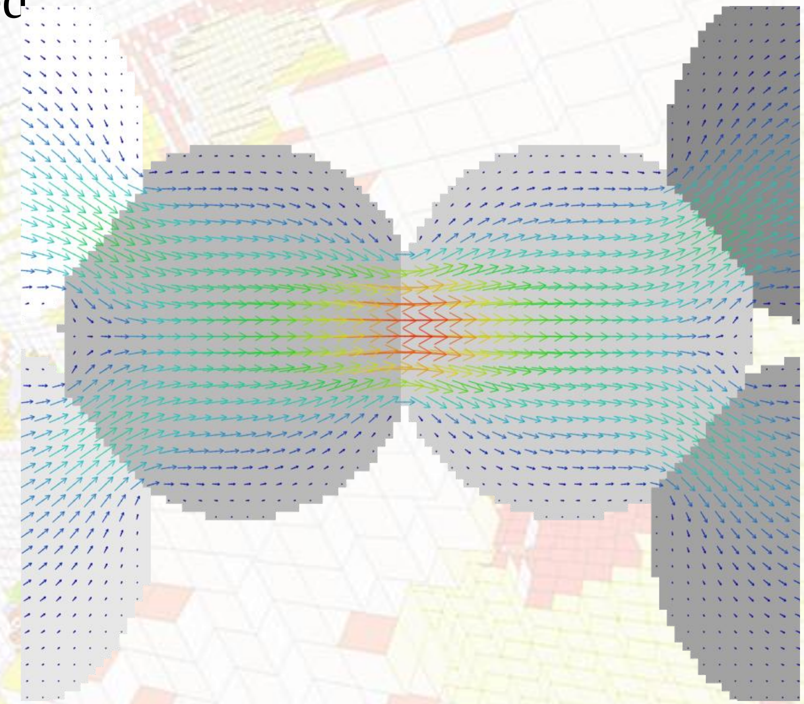
Pore network model

Pore-network model ≠ Pore positioning graph

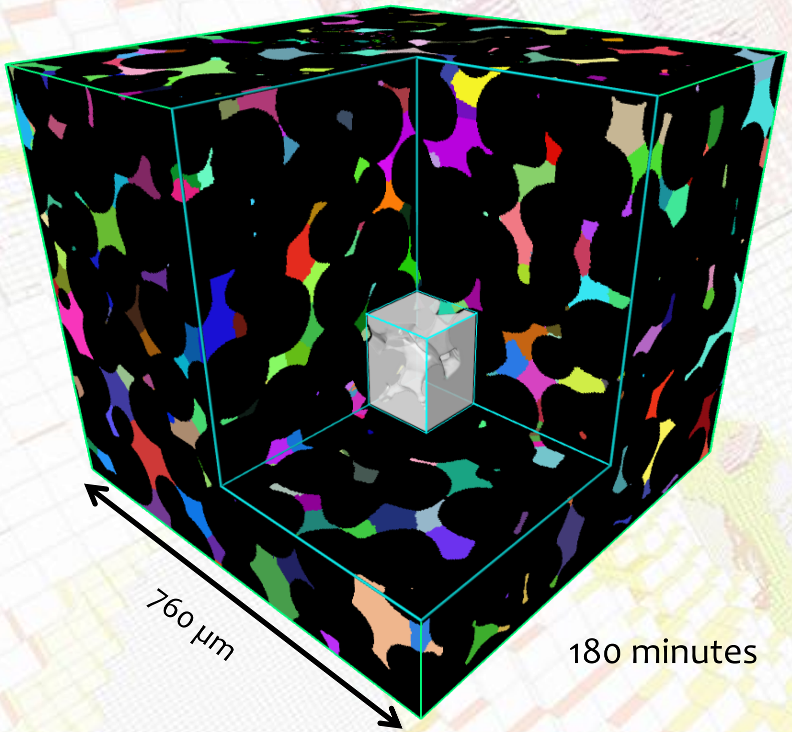
Assumption: Flow between two pores controlled *only* by their separation

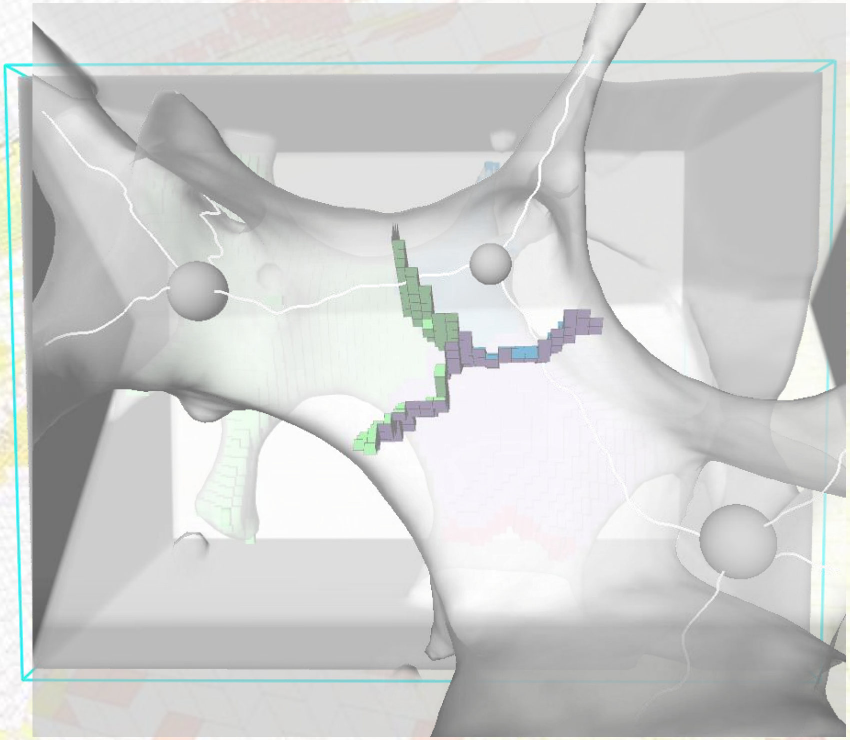


⇨ Pairwise pore connexions
⇨ Branch ↔ Throat

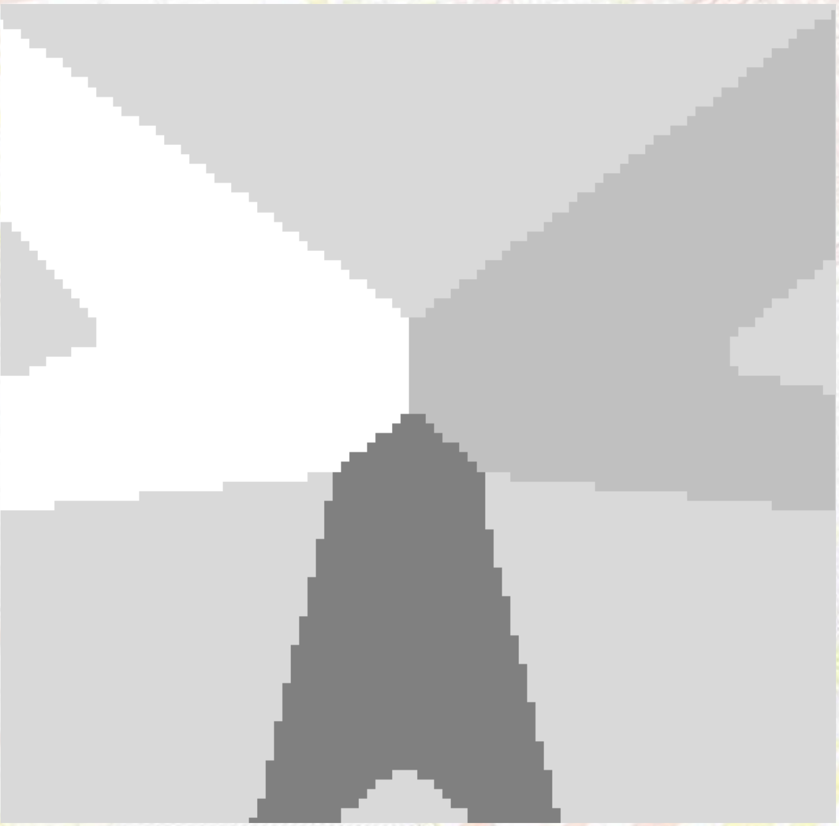
Pore network models

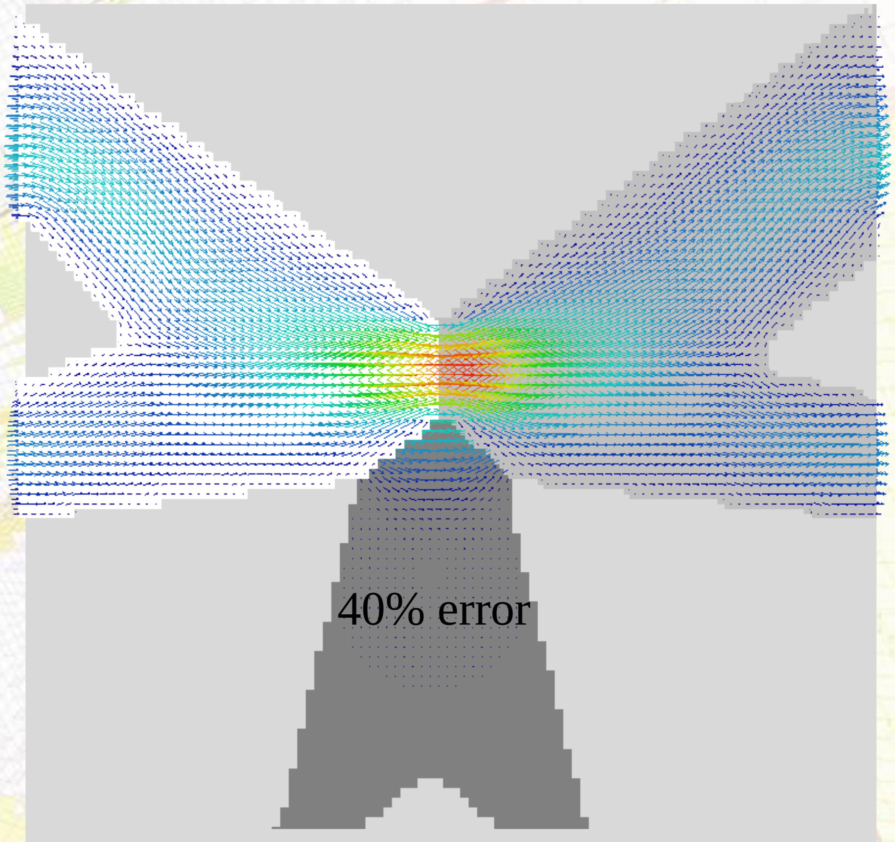
Existence of non-pairwise connexions



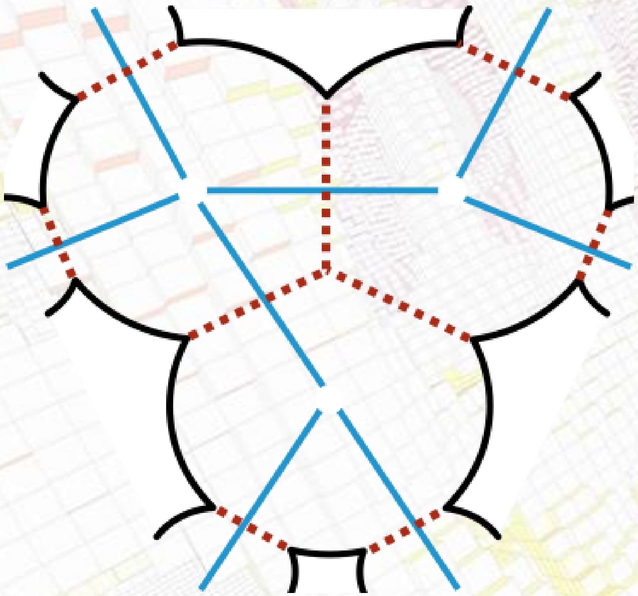


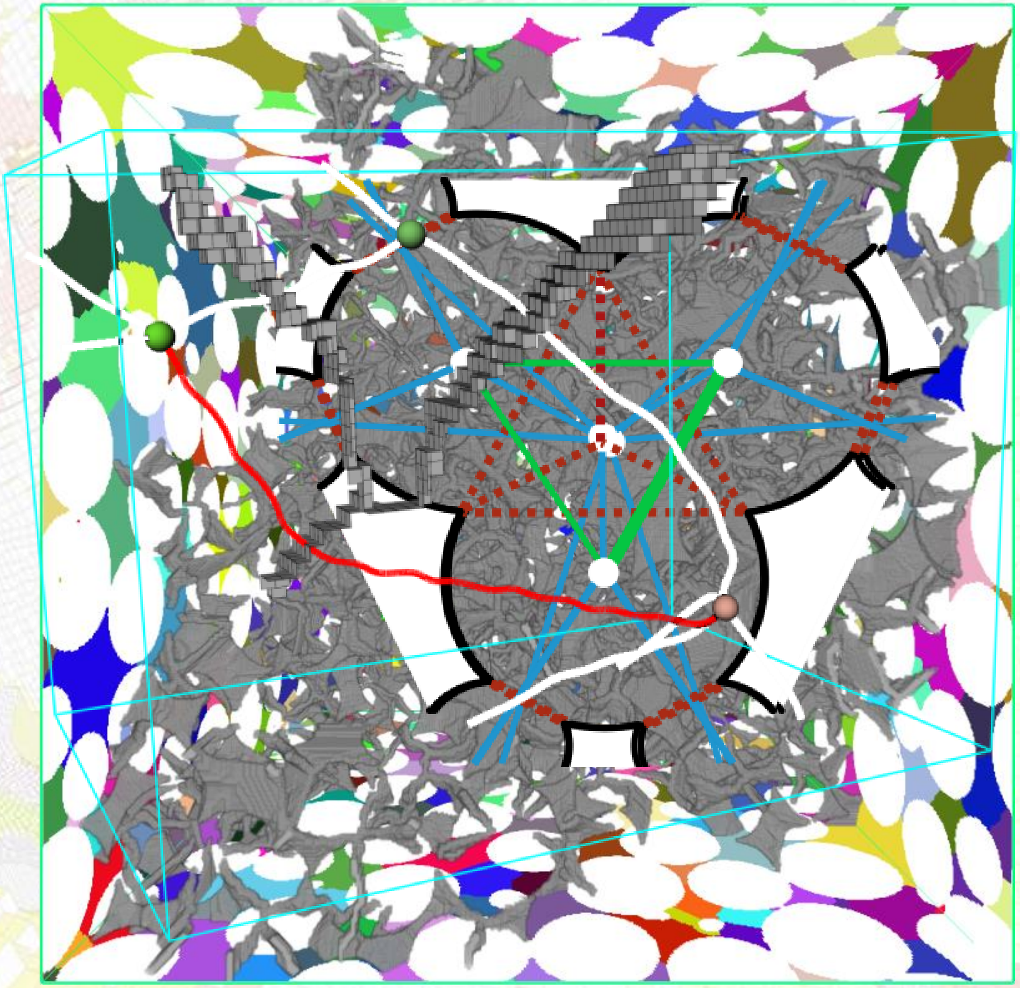
Multiple connexions





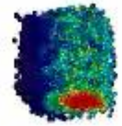
Multiple connexions

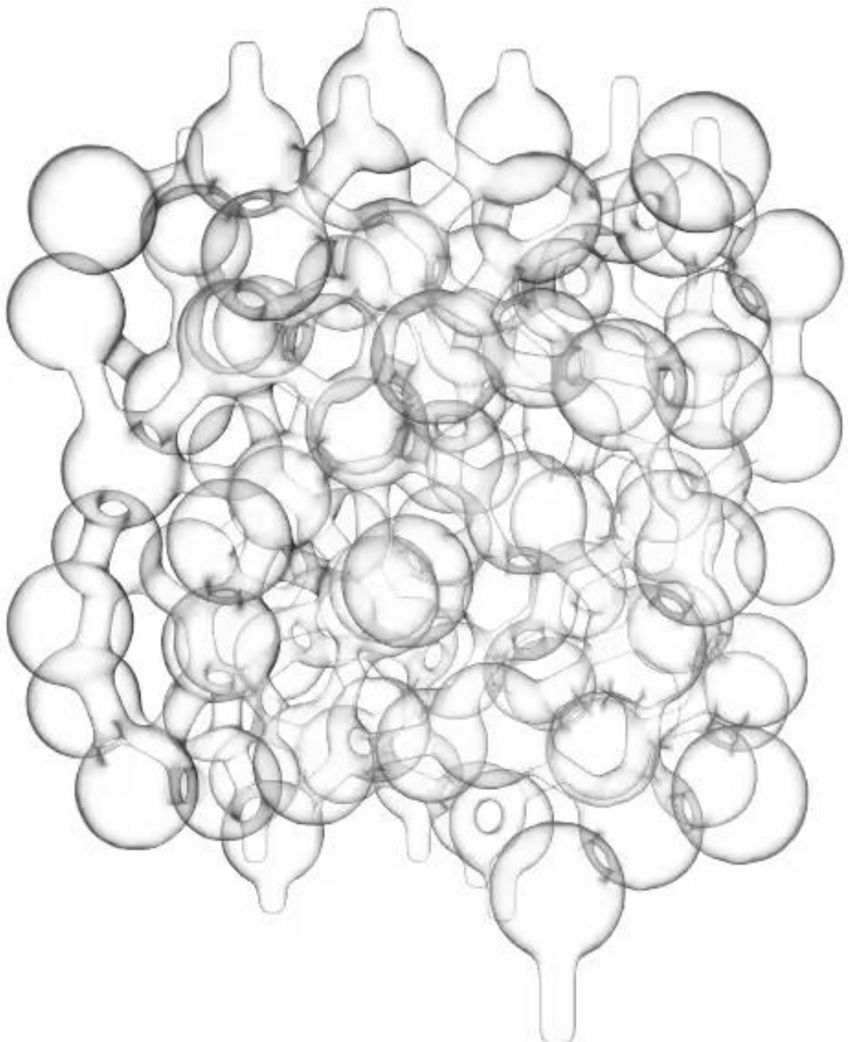




Conclusions

- Always the same basic problems (at first): segmentation, separation, labelling
- No method is universal
- Different material / acquisition / application ➔ different methodology
- More work needed for fast and robust analysis tools





Kernel segmentation : why not a watershed ?