

## Cluster analysis for FIB tomography of nanoporous materials

Rogge, F.<sup>1</sup> and Ritter, M.<sup>1</sup>

<sup>1</sup> Hamburg University of Technology, Germany

Focused ion beam (FIB) tomography is often the method of choice for analysing the 3D structure of nanoporous materials. Especially nanoporous gold (npg) is of interest as a functional material but also as a model system for nanomaterials. It is created by de-alloying, i.e. dissolving silver from gold-silver bulk alloy via electro-corrosion and forming a uniform network structure.

Because of "shine-through" artifacts [1], npg is in most cases infiltrated with epoxy resin before FIB tomography is applied in order to lower the influence of this effect. However, resin infiltration might lead to unwanted impact on the material, such as deformation due to cure shrinkage. Here, we examine the effect of such "shine-through" artifacts on several segmentation algorithms that have to be used for the 3D reconstruction of FIB tomography data. To allow for comparison of infiltrated versus non-infiltrated materials, we simulated FIB slices by creating backscatter electron images of npg with MC X-ray software [2]. MC X-Ray allows for defining dozens of regions with various shapes and all these regions can have a different composition. In our case, we created a npg network using randomly distributed gold cubes, and voids or epoxy cubes, respectively in a 40 nm x 40 nm x 40 nm voxel mesh volume. Virtual FIB slices with a thickness of 7 nm were virtually cut and backscatter electron images (BSE, 2 kV, 25 pA, 3.98 nm pixel resolution) of the a) npg-epoxy and b) npg-void volumes were calculated. Figure 1 shows the first 7 slices of each, the npg-epoxy (Fig. 1a) and npg-void (Fig. 1b) BSE images.

For the comparison, several segmentation algorithms were used prior to 3D reconstruction, some already implemented in commercial software (Avizo, ThermoFisher) or in open source software (ImageJ). In addition, k-means [3], a cluster analysis algorithm related to machine learning, was applied for segmentation and also for clustering. Three clusters ( $k=3$ ) were defined with the intention that information in the images is either gold, void or resin, or "shine-through", respectively. As expected, segmentation and volume reconstruction results yield a large error in the simulated data of non-infiltrated npg (see Table 1). Presumably, this is mostly due to the "shine-through" effect, which is larger in non-infiltrated nanoporous samples than in resin filled npg, especially visible in slices 5 and 6. Our cluster analysis approach, however, proves to be more robust against the effect, as the volume comparison of infiltrated vs. non-infiltrated npg shows a 6% increase in deviation.

### References:

- [1] T. Prill et al., *Scanning* 35, 189-195, (2013).
- [2] G. Gauvin et al., *Microsc. Microanal.* 15(Suppl 2), 488-489 (2009).
- [3] S. P. Lloyd, *IEEE Trans. Inf. Theory* 28(2), 129-137, (1982).

### Acknowledgements:

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MonteCarlo BSE simulation of npg (resin infiltrated)

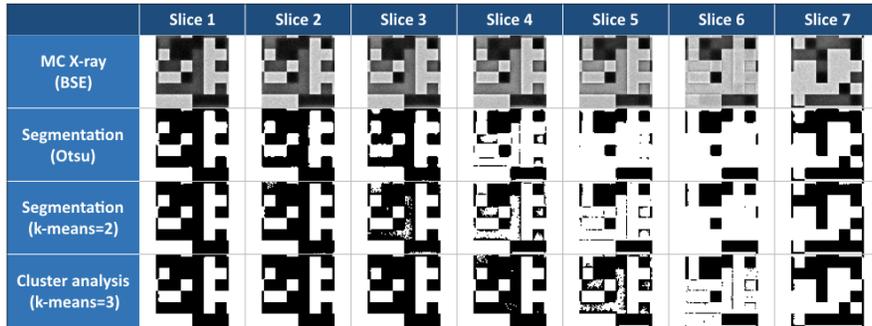


Fig. 1a: MC X-Ray BSE simulation of resin infiltrated npg. Segmentation and cluster analysis results.

MonteCarlo BSE simulation of npg (not infiltrated)

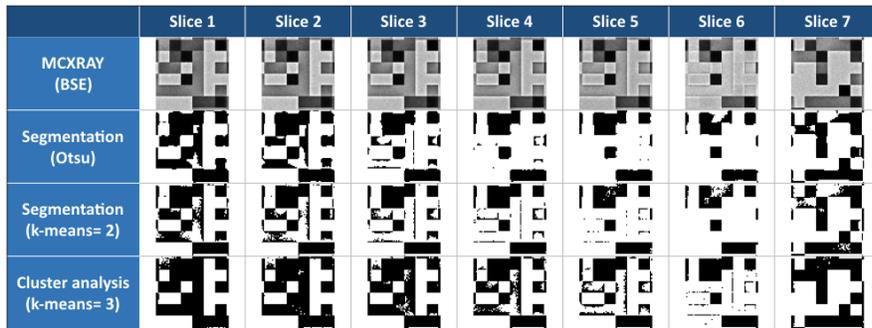


Fig. 1b: MC X-Ray BSE simulation of non-infiltrated npg. Segmentation and cluster analysis results.

	Otsu (Fiji)	Mean (Fiji)	Entropy (Avizo)	k-means (k=2)	k-means (k=3)
<b>npg epoxy infiltrated</b>					
Model volume gold [nm3]	8,12E+06	8,12E+06	8,12E+06	8,12E+06	8,12E+06
Segmentation result gold [nm3]	9,84E+06	9,41E+06	9,87E+06	9,93E+06	8,65E+06
Deviation [%]	21%	16%	22%	22%	7%
<b>npg non-infiltrated</b>					
Model volume gold [nm3]	8,12E+06	8,12E+06	8,12E+06	8,12E+06	8,12E+06
Segmentation result gold [nm3]	1,12E+07	1,02E+07	1,08E+07	1,11E+07	9,21E+06
Deviation [%]	38%	25%	33%	37%	13%

Table 1: Gold volumes of simulated npg data after segmentation or cluster analysis and deviation from gold model volume.