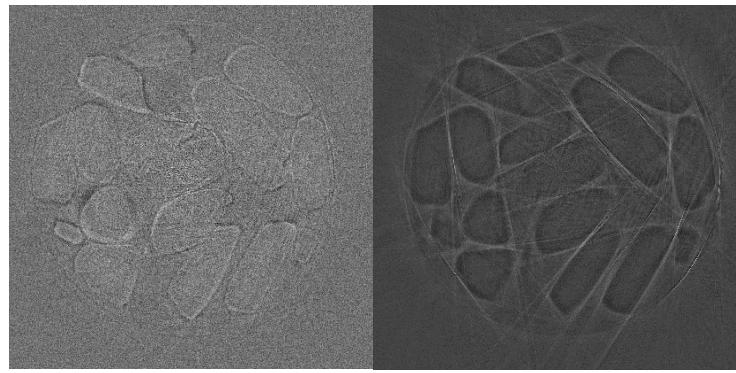


1 Supplementary Information S1: Edge artifact mitigation
2 In dark-field imaging, the DFEC values are sensitive to scattering effects associated with high-contrast
3 edges between structures larger than the scale of a single voxel and partial voluming. This is an
4 undesirable effect that hinders study of the sub-resolution structure. The effect is evident from the
5 edge contrast enhancement visible around the edge of particles in Figure 4 and Figure S1. We found
6 the artifact is aggravated by increasing correlation length ξ .

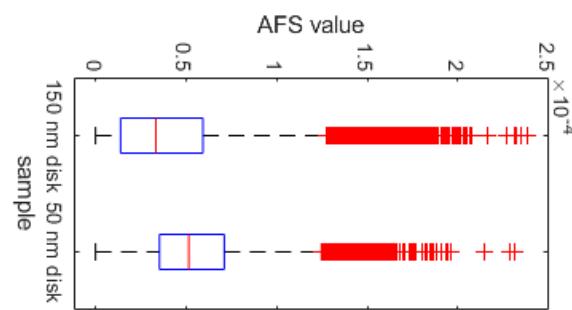
7



8 *Figure S1 Illustration of the edge artifacts in DFEC signals for the mixed sample, measured for a correlation length ξ of 24.83*
9 *nm (left) and 434.60 nm (right).*

10 The impact of these artifacts, as well as noise in general, is visible in Figure S2: in this graph, the AFS
11 value is extracted per voxel, rather than per object, and displayed as box plots. Per-voxel values are
12 evidently of very low precision, making it nearly impossible of reliably distinguishing a single 50nm disk
13 voxel from a 150nm disk voxel.

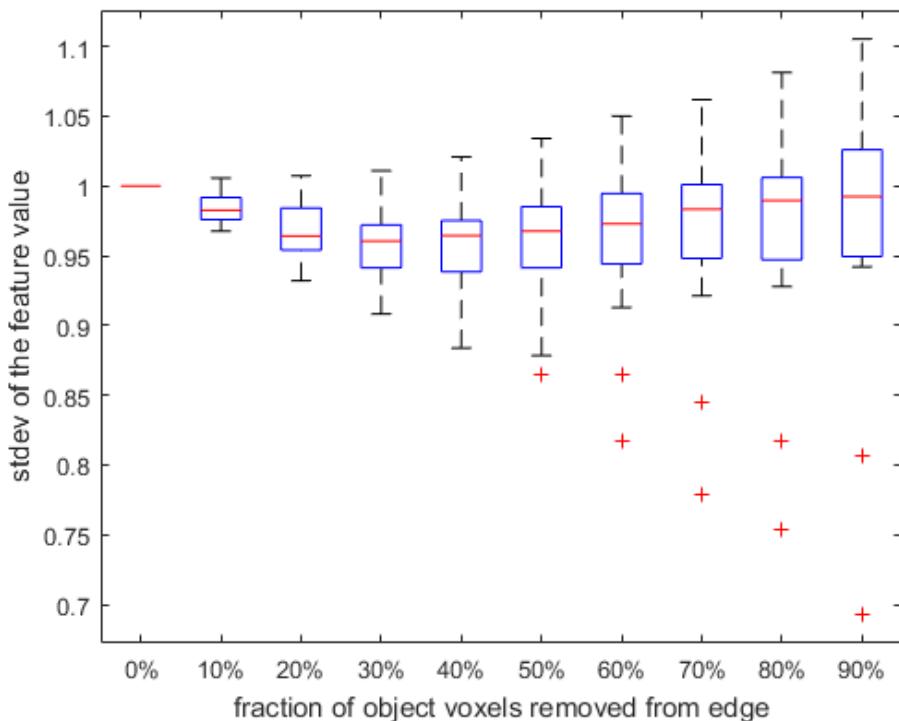
14



15 *Figure S2 AFS evaluated per voxel (rather than per object) for the voxels in the disk samples and aggregated into box plots,*
16 *the red crosses denote outliers.*

17 This limited low noise robustness is the motivation for the median aggregation of segmented structure
18 voxels in the proposed procedure. Additionally, as object edges are disproportionately affected by

19 artifacts, our procedure includes a method of mitigating edge artifacts specifically. This is accomplished
20 by use of mathematical morphology (erosion): specifically, the segmentation masks are iteratively
21 eroded, so that increasingly wider layers of the edge area are ignored before the AFS value is
22 calculated. Out of the series of AFS values thus generated, one for each erosion iteration, the lowest-
23 variance AFS value is retained to represent the object. This is found to improve precision: Figure S3
24 presents boxplots of the relative increase/decrease in variance of the AFS values for the objects in the
25 mixed sample (Fig. 13) as the amount of edge voxels that were eroded prior to the signal analysis
26 changes, compared to the situation when no edge voxels are removed. Notice how removing 30-40%
27 of the edge typically results in a lower-variance estimation.

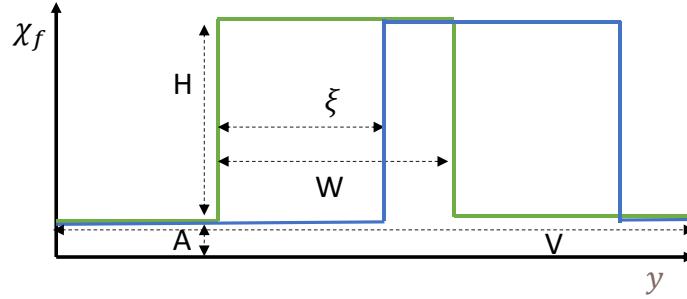


28

29 *Figure S3 Relative scaling of AFS variance, as a function of the fraction of the edge voxels that are eroded prior to AFS*
30 *estimation.*

31 Supplementary Information S2: Pore space model

32 We model sub-resolution pores as idealized pulses of identical contrast H , cfr. Figure S4. The width W
33 of these pulses, characteristic for the material's porosity, is our feature of interest.



34

35 *Figure S4 Idealized model for a single pore green in terms of the fine part of the materials' complex refractive index (green),*
 36 *as well as a shifted version (blue) by some correlation length ξ .*

37 Evaluating equations (1) and (2) in terms of this model to obtain its DFEC reveals a piecewise linear
 38 function in terms of the correlation length ξ . For correlation lengths below the smallest pore size $\xi <$
 39 W , we find that the DFEC is proportional to:

40
$$DFEC \sim \frac{(V - W)A^2}{V} + \frac{W(A + H)^2}{V} - \frac{(V - W)A^2}{V} + \frac{\xi A^2}{V} - \frac{W(A + H)^2}{V} + \frac{\xi(A + H)^2}{V} - \frac{2\xi(A + H)A}{V},$$

41 which simplifies as:

42
$$DFEC \sim \xi \frac{H^2}{V}$$

43 On the other hand, for correlation lengths that exceed the largest pore size, i.e. $\xi \geq W$, we get:

44
$$DFEC \sim \frac{(V - W)A^2}{V} + \frac{W(A + H)^2}{V} - \frac{(V - 2W)A^2}{V} - \frac{2WA(A + H)}{V}$$

45 Which simplifies to:

46
$$DFEC \sim \frac{WH^2}{V}$$

47 In other words, the *DFEC* values form, by approximation, a piecewise linear function of ξ that starts
 48 with a sloped segment and transitions into a constant segment, with the segment transition located
 49 around $\xi = W$.

50 A real-world sample consists of a variety of pores sizes with non-spherical shapes, which result in the
 51 autocorrelation function being a weighted average of apparent cross-sectional pore sizes. If the

52 correlation length ξ is below the smallest pore size W_{min} , each contribution to the *DFEC* will be in the
53 sloped regime of the function, and thus the total *DFEC* slope will be largest in magnitude. With
54 increasing correlation length values ξ between W_{min} and the largest pore size W_{max} the slope would
55 decrease until reaching 0 for $\xi = W_{max}$. Due to the quantized nature of such measurements in finite-
56 sized voxels, the measured slope would therefore characterize a weighted average of typically
57 encountered pore sizes and therefore be a characteristic for the pore size distribution. Furthermore,
58 the slope may be robustly extracted from many sample points, i.e. dark field acquisitions at different
59 correlation lengths ξ . This consideration is the motivation for extracting the slope as the characteristic
60 feature.