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Electron Tomography of Combustion Generated Nanoparticles

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The Problem

Transmission Electron Microscopy (TEM) of soot nanoparticles is limited to two dimensions. While it gives valuable information on the structure and reactivity of soot, it is not easy to correlate this to geometry of primary particles and agglomerates. Actual morphology is difficult to be interpreted while the surface area and volume are not simple to measure.

Proposed Solution

Apply 3D-TEM to characterise soot agglomerates to yield information on the volumetric character of fractal nanoparticles.

Aims: Create volume renders of 3D visualizations of soot nanoparticles and measure their surface area and volume.

Methodology

Image Acquisition

Electron Tomography: 3D structure produced from aligned tilt-series 2D-TEM images.

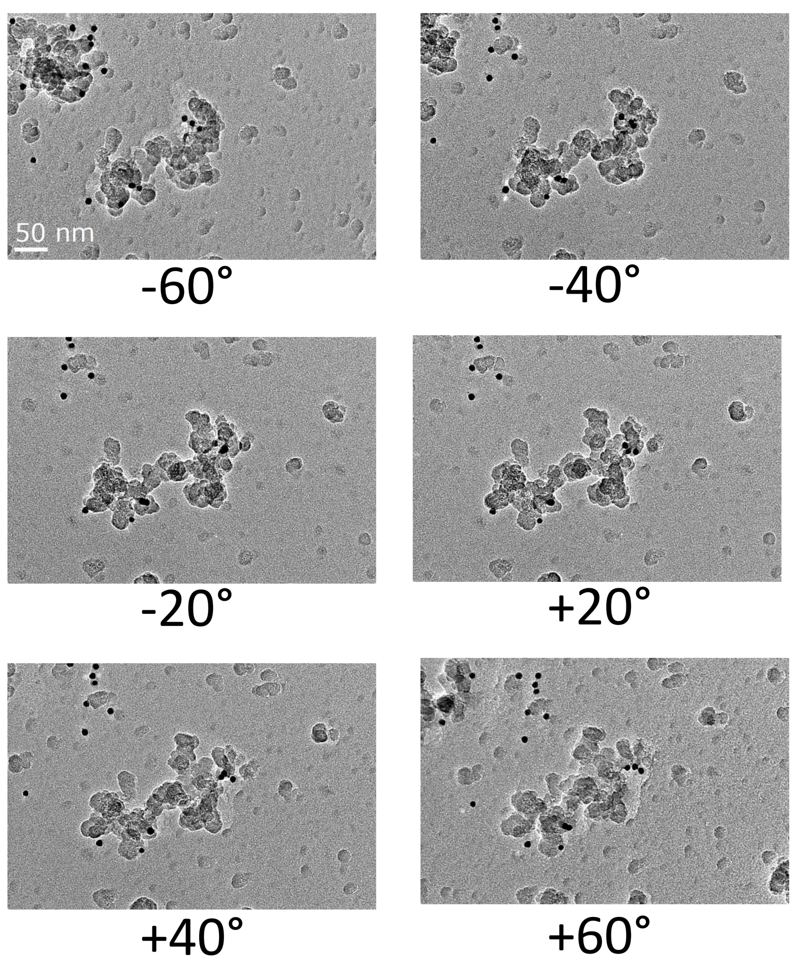

Figure 1. Six frames from the tilt series (aligned, unfiltered), with scale bar of 50nm, showing fiducial markers as dark dots.

121 TEM images, from -60 to +60 degree tilt with no sign of carbon build up were acquired.

Tomographic Reconstruction

Tomographic reconstruction using IMOD package. Weighted Back Projection (WBP) showed to produce clearest tomogram. Anisotropic Nonlinear Diffusion (AND) filter was employed to enhance the noise reduction.

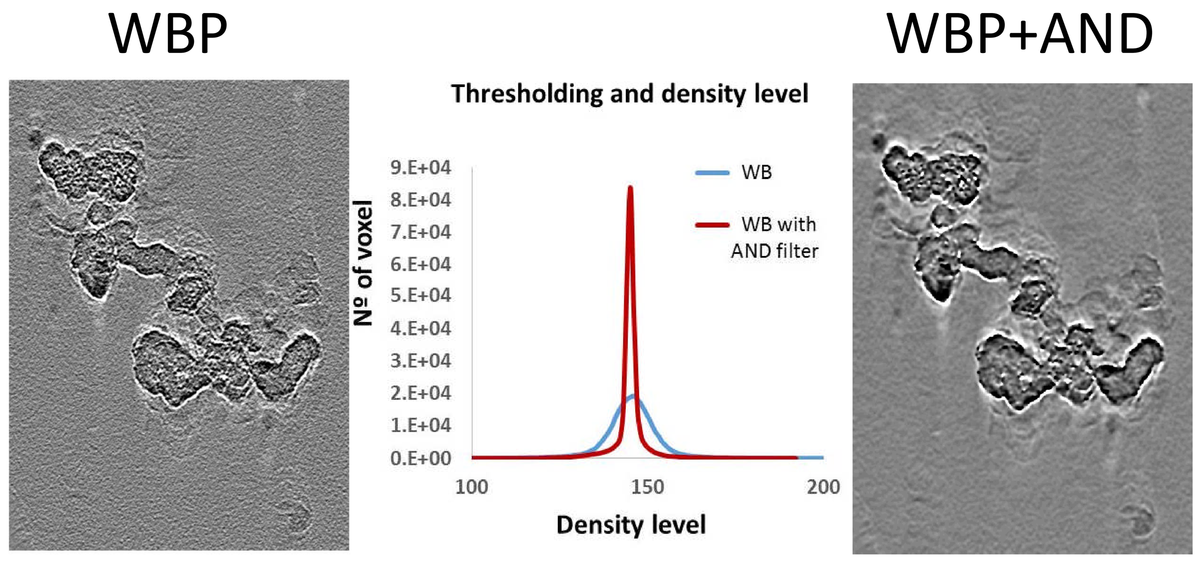

Figure 2. Comparison of tomographic reconstruction methods, and the effect of a AND filter on the tomogram. Tomograms produced using WBP algorithm.

AND achieves smooth voxels but preserving the edges as much as possible.

Segmentation then focuses on distinguishing the set of voxels constituting the structural components of the tomogram.

Model Construction and Analysis

Three-dimensional models were created using the IMOD package.

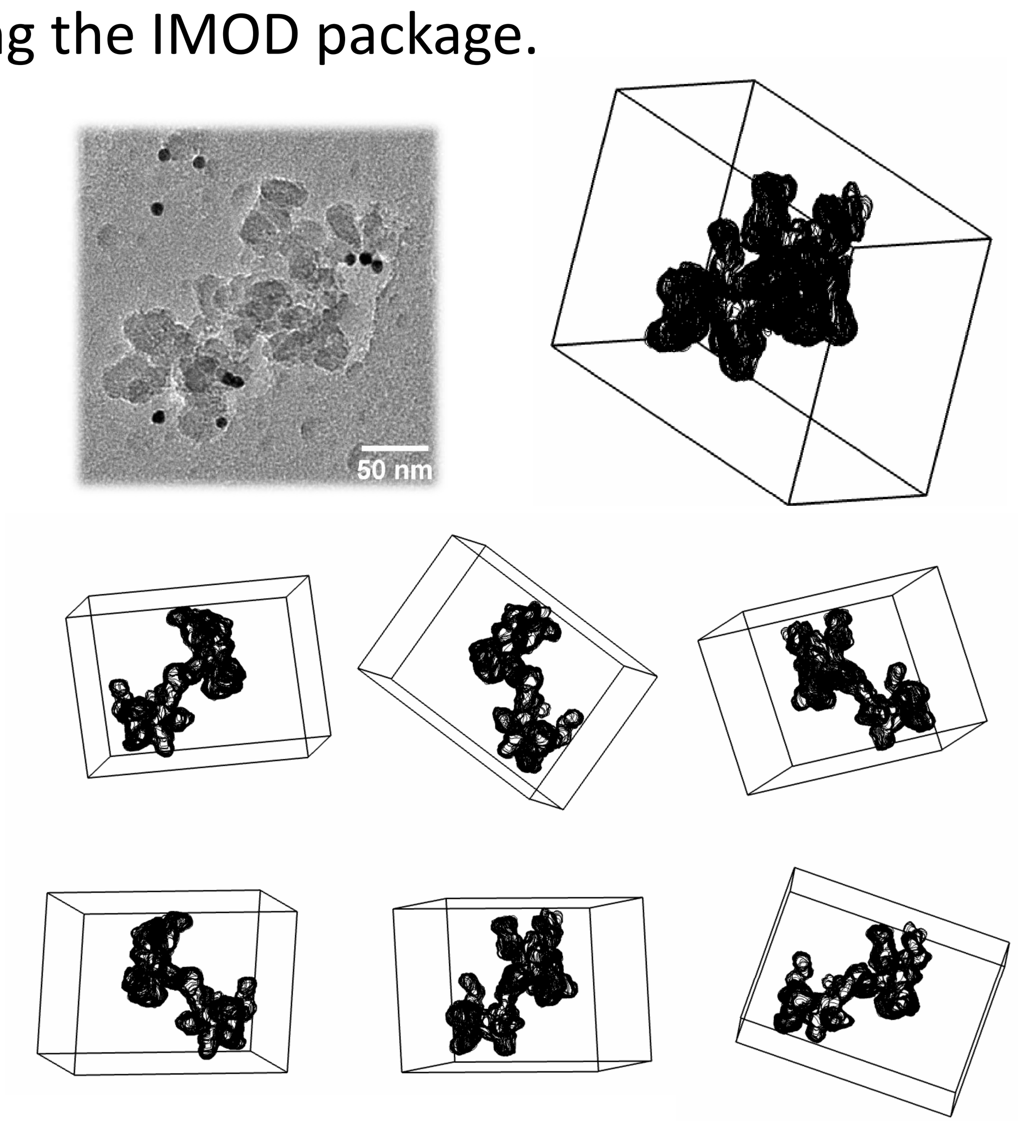

Figure 3. Volume renders of 3D visualizations of soot nanoparticle.

Compared with standard 2D-TEM, electron tomography has obvious superiority in revealing the actual agglomerate structure.

Results

Models were used to find the surface area and volume of soot nanoparticles. Analysis completed within Matlab. The results were compared against the surface area and volume calculated for a sphere whose diameter is the skeleton length of the nanoparticles measured from 2D TEM projections, as shown in the table below.

	Actual Surface Area 3D (nm^2)	Actual Volume 3D (nm^3)	Surface area of sphere enclosing particle (nm^2)	Volume of sphere enclosing particle (nm^3)	Sphere/Actual Surface Area ratio	Sphere/Actual Volume ratio
WBP	$57x10^3$	$554x10^3$	$172x10^3$	$6771x10^3$	3.1	12.2
WBP & AND	$43x10^3$	$343x10^3$	$170x10^3$	$6683x10^3$	4.0	19.5

Conclusions

Low background contrast graphene oxide films are particularly advantageous for work of soot characterisation using TEM.

3D reconstruction of soot nanoparticles in the range of 50-600nm was demonstrated using the widely available WBP algorithm.

Using the anisotropic nonlinear diffusion filter, strength and direction of filtering can be adjusted to maximise results.

Three-dimensional models can be used to measure surface area and volume of soot-in-oil nanoparticles.

Surface area and volume calculated using geometrical estimates, such as spheres or spheroids, for the soot agglomerates can lead to overestimation of the real size, with actual volume being between 12 and 19 times smaller than of a sphere of equivalent size. Surface area is overestimated by a factor of 4.

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