21st ETH – Conference on Combustion Generated Nanoparticles

Optimisation of 3D-TEM methods for high-throughput flame-generated soot nanoparticles analysis

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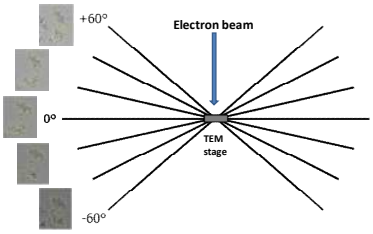
Characterisation of the fractal morphology of soot nanoparticles permits understanding of the behaviour and implications of soot in different environments. Typical characterisation via 2D electron microscopy suffers from a lack of depth perception meaning a host of approximations and correction factors are required to infer the values of real 3D properties. Prior research has also shown that 2D-derived results systematically under- & over-estimate the values of various morphological parameters. Reconstruction as 3D volumes via Electron Tomography allows direct measurement of morphological properties, improving the accuracy and effectiveness of the technique. However, 3D reconstruction of soot is in its infancy and significant improvements must be made to its speed in order for it to be a viable technique.

Aims: Improvements to the speed & quality of 3D reconstruction of soot nanoparticles was attempted by considering the choice of reconstruction algorithm, maximum tilt range & number of images comprising the tilt-series, and use of interpolation during segmentation.

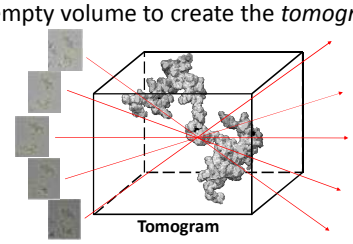
Tilt-Series Acquisition

By tilting the TEM specimen stage over a ±60° range, 121 images were produced of the same flame-generated soot particle from a range of different viewing angles



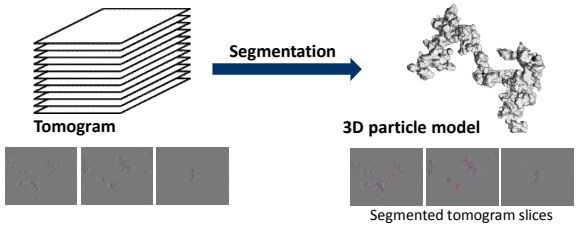
Tomographic Reconstruction

Computational algorithms are used to produce 3D information from the 2D information contained within the tilt series images. Back-projection methods thread pixel values through an empty volume to create the *tomogram*.



Tomogram Segmentation

Tomogram is arranged into slices, in which particle matter is distinguished from background areas manually using image processing software. Automated methods are not easily applicable due to high noise and low contrast.



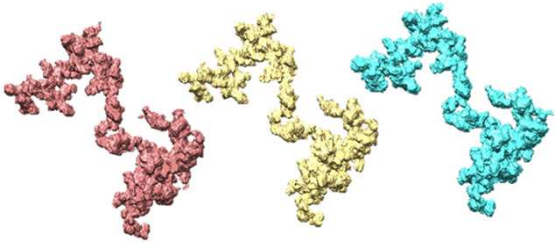
Segmented tomogram slices

Reconstruction Algorithm: Single-axis weighted back-projection (WBP), Dual-axis WBP, Simultaneous Iterative Reconstruction Technique (SIRT)
Tilt-Series Content: Tilt-series containing an image every 1°, 2°, 3°, 5° over a ±60° range, and an image every 1° over a ±50° and ±40° range (6 in total).
Interpolated Segmentation: Manual segmentation every n^{th} slice, with linear interpolation between (n = 5, 10, 15, 20).

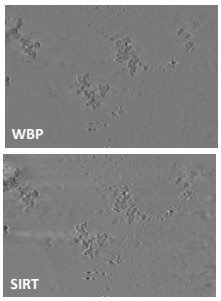
Results & Discussion

Reconstruction Algorithm (Speed *vs* Quality)

Single-axis WBP & SIRT (w/ 50 iterations) produce essentially identical tomograms, though WBP was much quicker (1 vs 135 minutes). Subjectivity in manual segmentation lead to deviations of 5.5 % in volume, 1.8 % in surface area, and <0.5% in fractal dimension and radius of gyration. Dual-axis reconstruction suffers from misalignment errors deriving from the tomogram combination process.



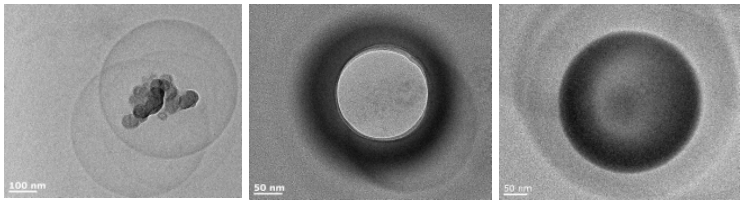
Single-axis SIRT | Single-axis WBP | Dual-axis WBP



Comparative tomogram slices

Tilt-Series Content (Quality & Beam exposure)

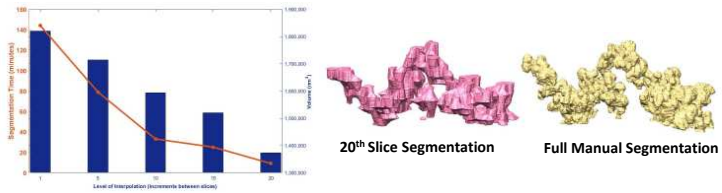
Most effective reduction of images results from larger increments between images, rather than reducing overall tilt range. As few as 25 images over a ±60° range are required to produce 3D volumes that do not differ significantly from those produced from 'full' tilt series. Samples are exposed to much smaller beam dosage and are therefore less prone to damage and contamination, which can be particularly problematic for soot extracted from engine lubricant oils.



Beam contamination in form of characteristic dark rings. Arises due to degradation of oil adsorbed onto surface of soot nanoparticles that have been extracted from lubricant oil phase of a GDI engine.

Interpolated Segmentation (Speed *vs* Quality)

Interpolated segmentation systematically reduced volume and surface areas, though had a negligible effect on the values of shape-descripting factors fractal dimension and radius of gyration. Interpolating between every 5th slice dramatically improved the speed of the process (79 vs 144 minutes) whilst deviations in volume, surface area, etc. were below the limits of human subjectivity in manual tomogram segmentation.



Conclusions

Optimal method for rapid 3D-reconstruction of soot nanoparticles:

- Single-axis WBP reconstruction
- 41 image, ±60° tilt-series (image every 3°)
- Interpolation between every 5th slice of tomogram

In comparison to typical procedure of ±70° tilt-series, SIRT w/ 30 iterations, full manual segmentation:

- Reduced reconstruction time by over 2 hours
- Reduced beam exposure by 71%
- Reduced segmentation time by 45%

with no significant deviation to the measured morphological parameters

This process represents a significant step towards high-throughput 3D characterisation of soot nanoparticles

Acknowledgements

The financial support through the scholarship provided by EPSRC DTG Centre in Complex Systems and Processes (EP/M506588/1) for one of the authors, E. Haffner-Staton, is also acknowledged. The authors would also like to acknowledge the support received from EPSRC Impact Acceleration Account.

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