

## **Assembly, analysis, and application of nanocarbons in composites and electrodes**

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Individual perfect nanocarbon structures have exceptional properties; the challenge is often how to exploit their potential in real macroscopic systems. Chemical functionalisation is critical to a wide range of nanocarbon technologies but needs to be versatile and applicable at scale. One particularly promising approach, relies on reductive charging to form pure charged nanocarbon anions which can be redissolved, purified, or optionally functionalised, whilst avoiding the damage typically associated with sonication and oxidation based processing [1]. This simple system is effective for a host of nanocarbon materials including MWCNTs, ultralong SWCNTs, carbon blacks, graphenes, and related materials. The resulting nanocarbon ions can be readily chemically grafted for a variety of applications. The chemistry of these discrete nano-ions raises interesting fundamental questions but is also practically useful. Solvated nanocarbon related materials can be assembled by electrophoresis, cryogel formation, or direct cross-linking to form Joule heatable networks, protein nucleants, supercapacitor electrodes, and catalyst supports [2][3]<sup>Error! Reference source not found.</sup>. Comparative studies allow the response of nanocarbons with different dimensionalities to be assessed to identify fundamental trends and the most appropriate form for specific situations. Combinations with existing commercial carbon fibres can provide opportunities to enhance state of the art performance or introduce new function [3]. Combinations of nanotubes and graphenes make excellent, durable fuel cell cathodes that resist corrosion. Networks of nanotubes can be formed into porous structures suitable for interacting with biological organisms for both fundamental studies and possible power extraction.

Although these types of macroscale assemblies and composite structures, built upon nanocarbon-related nanomaterials, are promising for a range of applications and devices, they can be difficult to characterise. The primary challenges revolve around understanding the morphology, dispersion, and spatial arrangement, as well as their interactions with other material phases. Multiscale and multidimensional characterisation methodologies can be used to investigate nanocarbon-based structures, employing a combination of optical and electron microscopy techniques, image processing and analytical methods. Confocal laser scanning microscopy (CLSM) is a valuable tool for microstructural investigation of graphene nanomaterials and nanocomposites, exploiting the technique's distinctive features, including high-contrast and non-invasive imaging, as well as depth discrimination. Using confocal reflection and total interference contrast imaging, the flake thickness distribution in graphene oxide (GO) films can be mapped rapidly and quantitatively. In addition to passive characterisation, CLSM can be used for simultaneous imaging and processing studies [1]: GO films can be selectively reduced in-situ to produce electrically conductive functional patterns at a range of lengthscales from millimetre to sub-micron. The versatility is demonstrated by direct writing "RC" circuits using the simple low power laser within the CLSM. Multi-modal tracking allows the conversion mechanism to be explored in situ while controlled ablation allows 3D architectures to be written directly.

Full characterisation in nanocomposites requires volumetric characterisation; a variety of complementary 3D imaging methodologies are therefore developed. Non-destructive CLSM stack imaging, using reflection and fluorescence modalities, is applied for large-scale examination of graphene nanocomposites. 3D characterisation methods based on destructive, serial array tomography enable correlative optical and electron microscopic imaging. Multiscale correlative characterisation is offers rich structural details of the local organisation, flake orientation, and morphology. The availability of 3D datasets provides exciting opportunities to quantify structural features. The methodologies developed will be widely applicable in the nanomaterials field.

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- 2) Govada, Shaffer, et al, Graphene-based nucleants for protein crystallization, *Advanced Functional Materials*, 32, 2022

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- 4) Li, Y., & Shaffer, M. S. (2023). Confocal Microscopy for In Situ Multi-Modal Characterization and Patterning of Laser-Reduced Graphene Oxide. *Advanced Functional Materials*, 2300479.