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# The Structure and Electronic States of Self-Assembled $C_{60}$ Crystals

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High-resolution transmission electron microscopy (HRTEM) can provide valuable insights into characterizing the underlying structure-property relations of various materials. It has been extensively applied in the detailed analysis of 2D and 3D carbon-based materials, e.g., fullerenes [1], nanotubes[2], and graphene[3, 4]. We are particularly interested in Buckminster fullerene, $C_{60}$, because of its recent detection in protoplanetary nebulae [5, 6], its potential to contain heteroatoms [7], and its ability to self-assemble into crystalline rods, tubes and islands in meso- and nano-scales [8]. However, nanoscale analysis of such C materials requires low-voltage techniques in order to mitigate electron-beam induced damage. Here we examine $C_{60}$, synthesized via aqueous self-assembly methods, as a means to investigate the low-voltage capabilities of the newly installed probe-aberration-corrected Hitachi HF5000 at the University of Arizona.

The $C_{60}$ nanocrystals were prepared by the wet chemistry method described in [4]. $C_{60}$ crystals, suspended in isopropyl alcohol (IPA), were dropcast onto a continuous carbon film supported on 400 mesh Cu grid and left in ambient air for three weeks so that the IPA could completely evaporate. The microanalysis was carried out using a Hitachi HF5000 TEM/scanning TEM (S/TEM) equipped with Oxford twin energy-dispersive spectrometers (EDS), providing a solid angle of ~1.7 sr. The HF5000 is also equipped with a Hitachi 3$^{rd}$-order probe-aberration corrector for atomic-resolution imaging in STEM mode as well as a secondary electron detector. We performed simultaneous ADF/BF/SE imaging at 60 kV, below the knock-on damage threshold for C, and which provided atomic-scale information of the $C_{60}$ crystals.

Low-magnification TEM images show the facets of the $C_{60}$ crystals (Fig. 1A). Bright-field STEM shows lattice fringes with a spacing of 0.81 nm, consistent with the $d_{111}$ in FCC fullerenes (Fig. 1B, Fig 2A). The low contrast in the ADF indicates that the particles have similar composition to the supporting matrix and also suggests that the sample is made up of a low-Z element (Fig. 2). EDS mapping, performed using a low background (Be) holder, reveals that the crystals consists purely of C (Fig. 3A). In addition to the carbon signal, a small amount of O was also detected on the carbon support matrix (Fig. 3B). These preliminary studies indicate that the $C_{60}$ crystals can be studied without noticeable beam damage at 60 kV acceleration voltage. It also paves the way to carry out EELS measurements of the plasmon excitation and fine structure of the $\pi^*$ and $\sigma^*$ bonding of the $C_{60}$ structures in various states. Furthermore, because crystal morphology is dependent on the growth substrate and solvent used [8,9], we plan on carrying out a systematic study of $C_{60}$ structures that can be synthesized based on the choice of the solvent(s) in conjunction with the substrate(s) [10].

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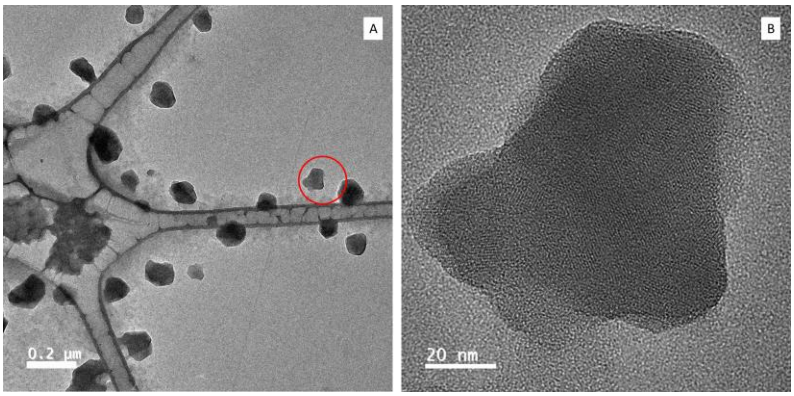


**Figure 1.** [A] Low magnification TEM image of $C_{60}$ particles on C film [B] HR image of the $C_{60}$ particle indicated in Fig 1[A]

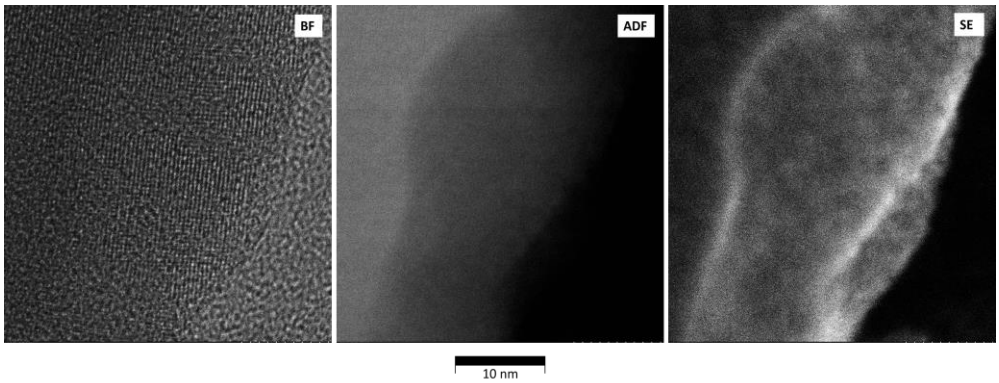


**Figure 2.** Simultaneous STEM BF/ADF/SE Imaging. The lattice fringes can be seen in the BF image.

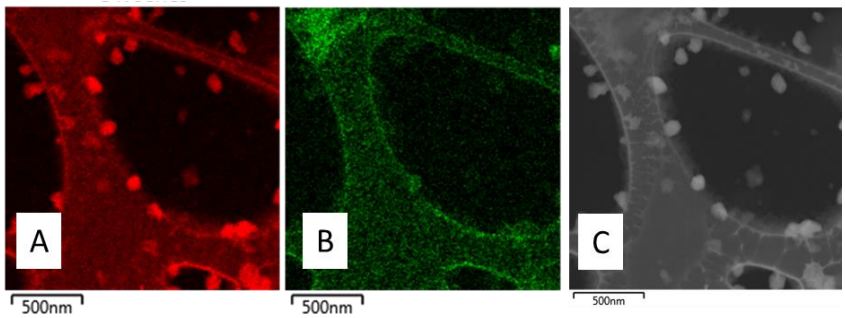


**Figure 3.** EDS Map [A] Carbon [B] Oxygen [C] Low magnification STEM Image.


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