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FIB Lift Out of Columnar Carbon Structures

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It is well known that graphite/carbon can undergo transformation when subjected to an energy source [1]. This transformation can either be destructive or constructive. Destructive transformation can be seen in the carbon/graphite used in nuclear reactors. As the graphite is exposed to radiation, defects are introduced to the point of disorder in the graphite structure [2]. Constructive transformation happens as an electron beam, incident to the sample, causes dislocations to move through the sample connecting discontinuous graphite planes [3].

Carbon aerogels and activated carbon have been shown to produce protruding over growths when heated to 2300 °C. Characterization of these columnar carbon structures (CCS) by conventional scanning and transmission microscopy, (SEM, TEM) showed the morphology of these assemblies very well. Data was gathered as to the shape, size, growth habit and populations utilizing these two methods, although most of the CCSs were too thick for the internal structure analysis by TEM. A representative carbon aerogel, dicyclopentadiene (DCPD), was chosen to make a focused ion beam (FIB) TEM sample. DCPD carbon aerogel was chosen because the CCSs were abundant as well as relatively short and a flat substrate was available.

The attempt to make a FIB sample, using typical ion beam settings, Table 1, for most CCS containing materials proved to be problematic. First, it was destructive, when the ion beam focused on CCSs, their integrity was compromised and they appeared to be melting (see Figure 1), and second when a cross-section was ion milled, there were copious amounts of re-deposited of carbon and Ga, as well as, extremely high Ga+ ion implantation. To protect the CCSs from ion beam damage during the initial cross-sectioning and lift-out, a protective coating of Al, 0.75 um thick, was deposited on the surface of DCPD carbon aerogel. Even after deposition of the Al and Pt, re-deposition remained an issue (see Figure 2).

After the initial cross-section successive thinning and cleaning steps were conducted at lower ion beam currents to lessen the re-deposition and Ga^+ implantation. Cleaning steps were performed between thinning to check for any damage to the CCS and carbon substrate. The closest cut face, after each cleaning step, was checked with EDS, Ga was only detected in the larger voids. Re-deposition continued to be a major issue throughout the thinning and cleaning steps. Using lower currents with multiple thinning and cleaning steps (four thinning and cleaning steps) the time for a cross-section and lift out was approximately 7 hours, Table 1. After the U-cut, the re-deposition was so thick, the carbon substrate and CCS features were totally covered. The sample was intentionally left thick to insure enough un-compromised material was accessible during the final thinning and cleaning steps.

The interior structure showed three distinct angles present in this area. A smaller apex, (127.16°) is on top of an extrapolated apex (138.36°), and a third, larger, angle (163.75°), is off center. These features indicate the interior has faceting and the smaller apex is due to the disclination angle of the helically wrapped graphene sheets. An apex angle of 143.96°, lower in the CCS, does not have multiple angles and is more in line with previously reported apex shapes [4,5,6].

The spacing of the graphene planes is 3.35 Å (see Figure 3). This interior lattice spacing is virtually the same as that of graphite, 3.349 Å. With the spacing of the graphene planes equal to the spacing of graphite indicates that the cleaning procedure with the Ga+ ion beam at lower accelerating voltages and beam currents, has not damaged the internal CCS carbon structure.

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Table 1. FIB procedure and parameters

procedure	ion beam, kV[a]	ion beam curr., nA[b]
Pt deposition	30(30)	0.92(2.8)
Initial Cross-section	30(30)	6.5(11)
Thinning Rectangle	30(30)	2.8(6.5)
Cleaning	30(30)	0.92(2.8)
Final Cleaning	16(30)	0.47(0.92)
U-Cut	30(30)	*6.5(11)
Rectangle cleaning 1	16(30)	43 pA(2.8)
Rectangle cleaning 2	16(30)	2.8 pA(2.8)
Cross-Section Cleaning 1	16(30)	2.8 pA(2.8)
Cross-Section Cleaning 2	5(30)	**0.45(43 pA)
Cross-Section Cleaning 3	16(30)	2.8 pA(43 pA)

[a]Values in parenthesis are typical lab ion beam values for lift outs of most materials, [b] Value in parenthesis are typical lab ion current values for lifts of most materials *caused major re-deposition on cleaned surface, **caused curtaining to occur

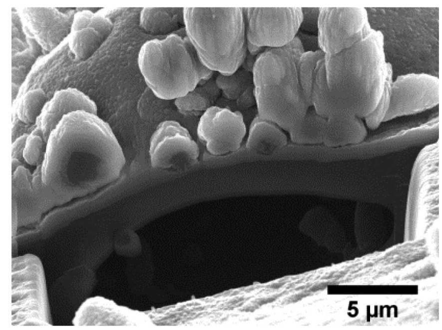

Figure 2. FIB cross section of Al deposited CCS.

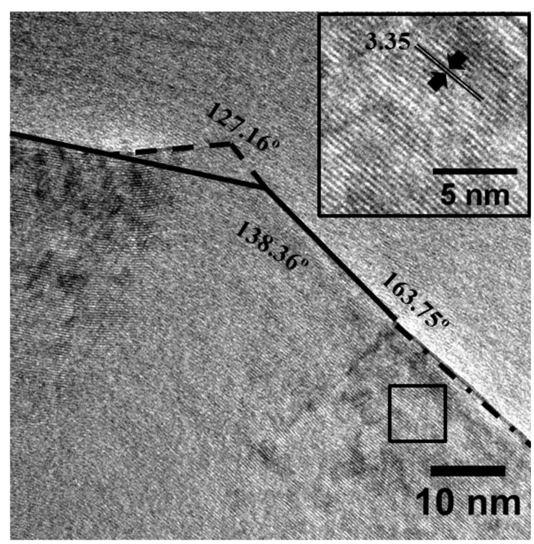

Figure 3. TEM image of CCS internal structure.

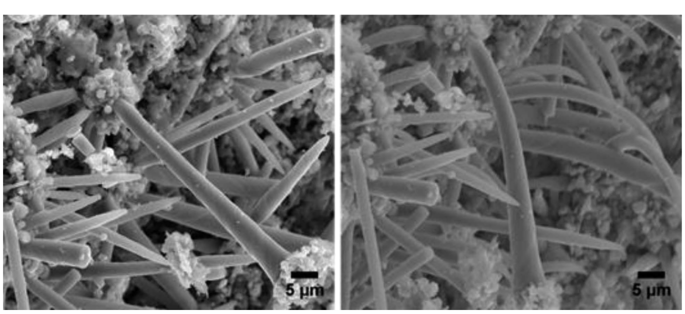

Figure 1. CCS before, left, and after, right, ion beam interaction.

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