Information about the 3-D structure of polymer plugs in CNTs can be gleaned from sample tilt visualization experiments. Figure I below shows two high-resolution TEM images of the same PCL mass deposited at the end of a catalytic CNT. Each image corresponds to a different tilt angle of the TEM sample stage holding the grid on which this CNT is supported; (a) 0 deg, (b) 20 deg.

Figure I: PCL mass deposited at the end of a catalytic CNT. Each TEM micrograph corresponds to a different tilt angle of the sample stage holding the TEM grid on which this CNT is supported; (a) 0 deg, (b) 20 deg.

Figure IIa below shows catalytic, initially dry CNTs containing intercalated PCL. It is clear that all CNTs in this typical image contain polymer. On the other hand, Fig. IIb shows much less intercalated PCL in catalytic pre-wetted CNTs. Two of the five CNTs seen in Fig. IIb do not show any visible PCL inclusions. Two more CNTs of the five in Fig. IIb show small PCL masses in their channels, but also reveal large gaps, presumably caused by the earlier presence of water there, which prevented the polymer and its solvent toluene to enter. Only one of the five CNTs in Fig. IIb contains PCL along the entire channel with fill patterns similar to those seen in (a).
Figure II: Catalytic CNTs containing intercalated PCL (80 kDa) from a 1 wt % toluene solution. Image (a) corresponds to initially dry CNTs, while (b) shows pre-wetted CNTs which probably contained water (at least partially) before encountering the PCL/toluene solution. Note that in (a) the PCL masses are present throughout the entire visible CNT length, while in (b) the PCL masses are absent or show large gaps, presumably due to the earlier presence of water there, which prevented intercalation of the PCL/toluene solution (water and toluene do not mix and water is a non-solvent for PCL). Both images show CNTs supported on thin carbon webs (marked), which also reveal dried polymer patterns.