

Investigation of the crystallization behaviors in a sub-micron space using carbon nanocones

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Supportive Information

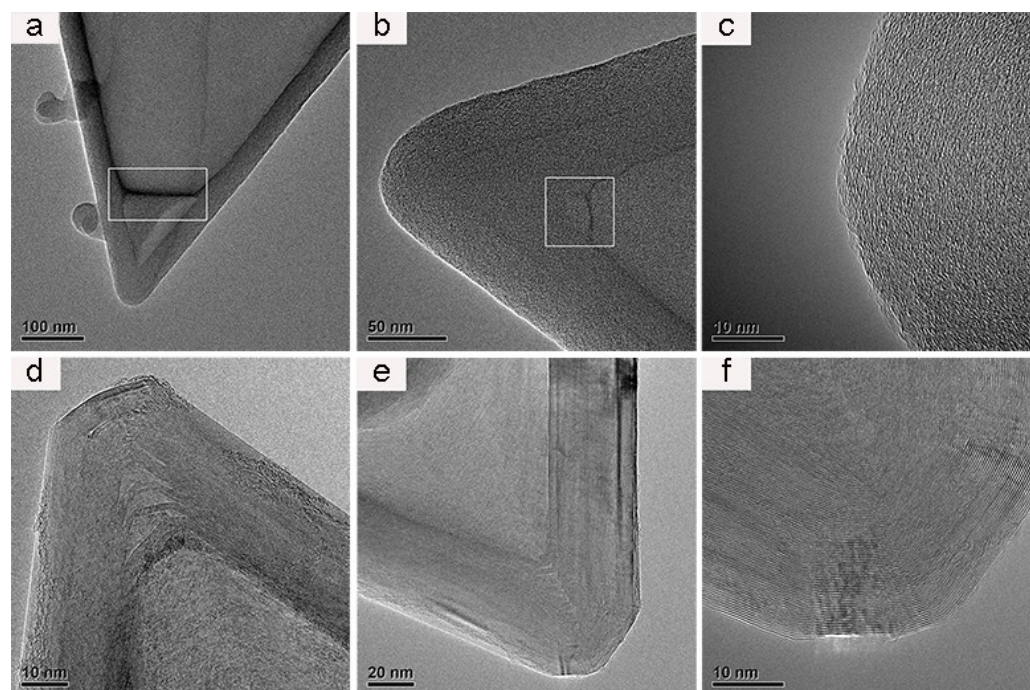


Figure S1. (a-c) TEM images of pristine CNCs-2 (which wasn't annealed at 2700°C) and (d-f) CNCs-1 (which was annealed at 2700°C) without incorporating Gd acetate.

As seen in Figure S1, a-c present pristine CNCs-2, d-f present pristine CNCs-1. From picture (a, b), we can see it clearly that there are some impurities aggregating near the apex of CNCs-2 and the graphite layers are not that straight from picture S1 c. While in picture S1 d-f, the apex of CNCs-1 appears very clean with nothing else enclosed and also the graphite layers straight. The degree of graphitization and the crystallization of CNCs-1 are higher than CNCs-2, making CNCs-1 more suitable for encapsulation experiments.

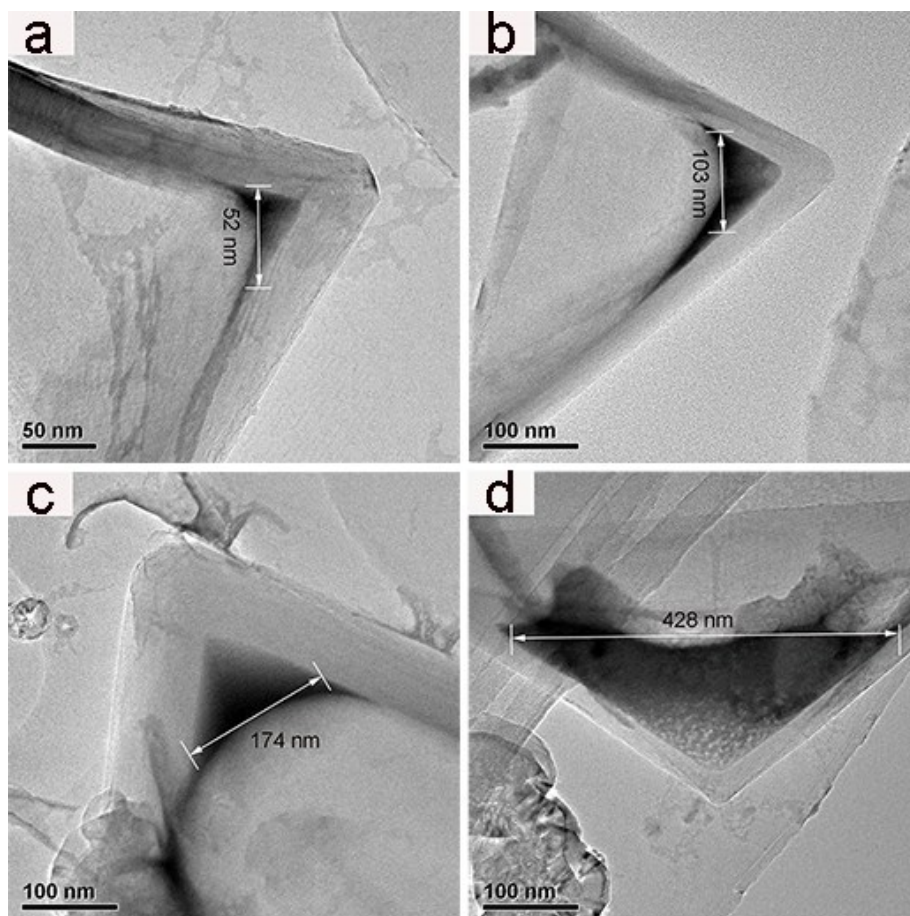


Figure S2 shows the various filling diameter of Gd compounds. The encapsulated cone-shaped Gd acetate have various diameters from 50 nm to 400 nm. Most of them fall in the range of 100-200 nm and the maximum can be up to 428 nm (Figure S2 d).

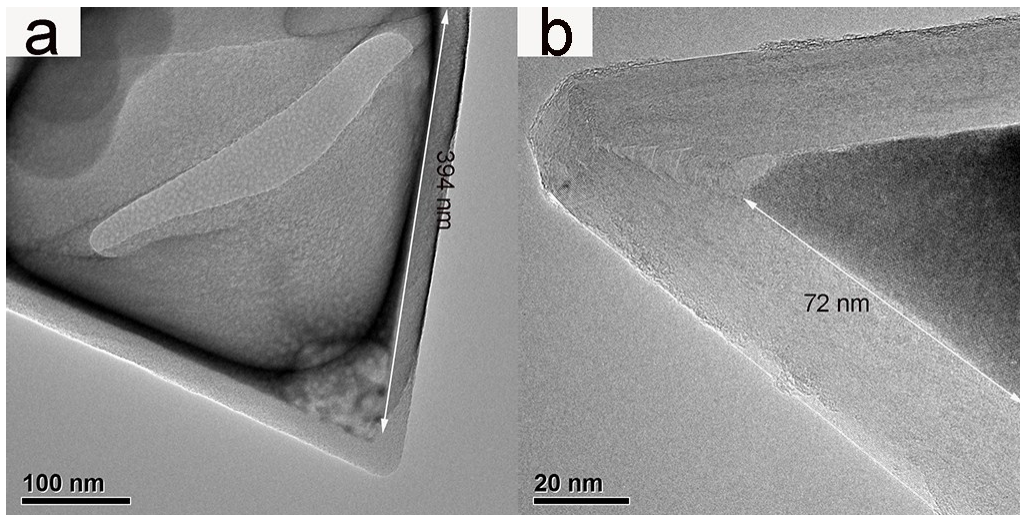


Figure S3 (a) Gd acetate adherent to the wall of CNCs-2 which reaches the tail of the cone with a length of 394 nm. (b) Gd₂O₃ single-crystal encapsulated in CNCs-1 with a size of more than 50 nm laterally and an axial length up to 72 nm.

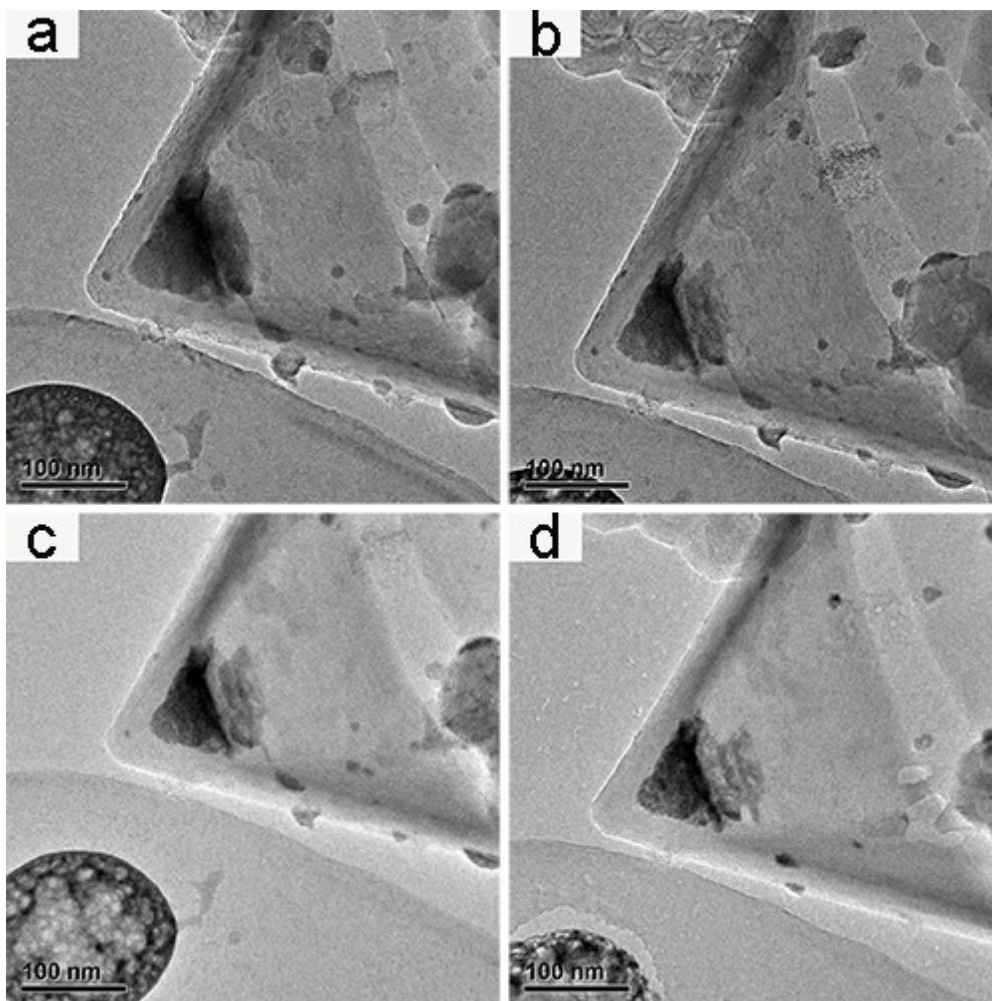


Figure S4 shows the serial images of the encapsulated Gd acetate transforming to Gd oxide when heated in-situ under TEM. The sample was dispersed on SiN film for in-situ heating and TEM

observations. A Pt circuit embedded in the SiN film was used for heating by flowing a certain current, and the temperature was calculated based on the thermal-resistance effect. In this experiment, we selected a GdAc@CNC-1 on the film under TEM and heat GdAc@CNC-1 via an external power supply. It's clear to see the process of Gd acetate converted to Gd oxide slowly. Fig S4 (a) is the pristine GdAc@CNC-1 before heating. When we raised the temperature up to 650 °C, Gd acetate began to decompose and the structure started to change obviously, as seen in Figure S4 (b,c). Then we kept raising the temperature to 1000 °C, inducing further structural changes, as shown in (d). But this heating process didn't last for a long time because the grid can't bear such a high temperature and we can see many broken holes of the film. However, the inside Gd oxide kept its cone-shaped form unchanged in the whole process.