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In-situ Heating of Functionalized Carbon Nanotubes

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Carbon nanotubes (CNTs) are promising materials as enforcements in polymer-, ceramic- and metal-matrix nanocomposites, due to their extraordinary mechanical, thermal and electrical properties. However, their hydrophobicity behavior and chemical inertness cause tangling or poor dispersion, limiting their commercial applications. To solve this technical issue, functionalization of CNTs by surface modifications using chemical reactants [1-6] have been introduced to improve the dispersion degrees [7]. This work is to evaluate the thermal stability of the functionalized CNTs by *in-situ* heating in a TEM.

The experimental material, CNT-COOH, was derived by reacting CNTs with mixed sulfuric and nitric acids. Sample for *in-situ* heating was deposited onto silicon nitride support film, with 50 nm thickness through a 0.25×0.25 mm size window (Ted Pella, Inc., Prod. No. 21502-10), and observed in a JEOL 2010 TEM at 200 kV equipped with a Gatan heating stage.

A typical appearance of a functionalized CNT is shown in Fig. 1(a). It is evident that reactants are coated on CNT surface. A magnified image is inserted, exhibiting the carbon layer lattice fringes. The EDS analysis in (b) reveals evident N, O and S signals.

The *in-situ* heating is performed from room temperature (RT, 16 °C) to 1000 °C. The RT image is shown in Fig. 2(a), with two enlargements from the framed areas shown in (b) and (c), respectively. Several individual CNTs are chosen for inspection during the *in-situ* heating, as labeled from 1-15 in (b) and (c). When it is heated to 250 °C, the image is shown in (d), which is enlarged in (e) and (f). It is noticed that the spacing between the top and bottom clumps is increased, indicating the evaporation of the clumps during the heating. Most of the CNTs remain stable, while some of them, such as the CNTs 8, 9, 10, 12, 13, 14, are coated with additional layers by the evaporation from the clumps. At higher temperatures, these CNTs remain quite stable, as shown in (g) taken at 1000 °C. Figs. 2(h) and (i) are two images after cooling down to RT, which are similar to the images at 250 °C in (e) and (f), respectively, indicating the considerably high thermal stability of the CNTs, which possess the potential for high temperature applications [8].

References

[1] K.A. Wepasnick, B.A. Smith, J.L. Bitter et *al. Analy. Bioanly. Chem.* **396** (2010) 1003.
[2] K.A. Wepasnicka, B.A. Smitha, K.E. Schroteb, *et. al. Carbon* **49** (2011) 24.
[3] L. Adams, A. Oki, T. Grady, *et al. Physica* **E41** (2009) 723.
[4] A. Oki, L. Adams, Z.P. Luo, *et al. J. Phys. Chem. Solids* **69** (2008) 1194.
[5] A. Oki, L. Adams, V. Khabashesku, *et al. Mater. Lett.* **62** (2008) 918.
[6] L. Carson, C. Kelly-Brown, M. Stewart, *et al. Mater. Lett.* **63** (2009) 617.

[7] Z.P. Luo *et al. J. Microsc.* **225** (2007) 118; *Polymer* **49** (2008) 1841; *Mater. Lett.* **62** (2008) 3493.
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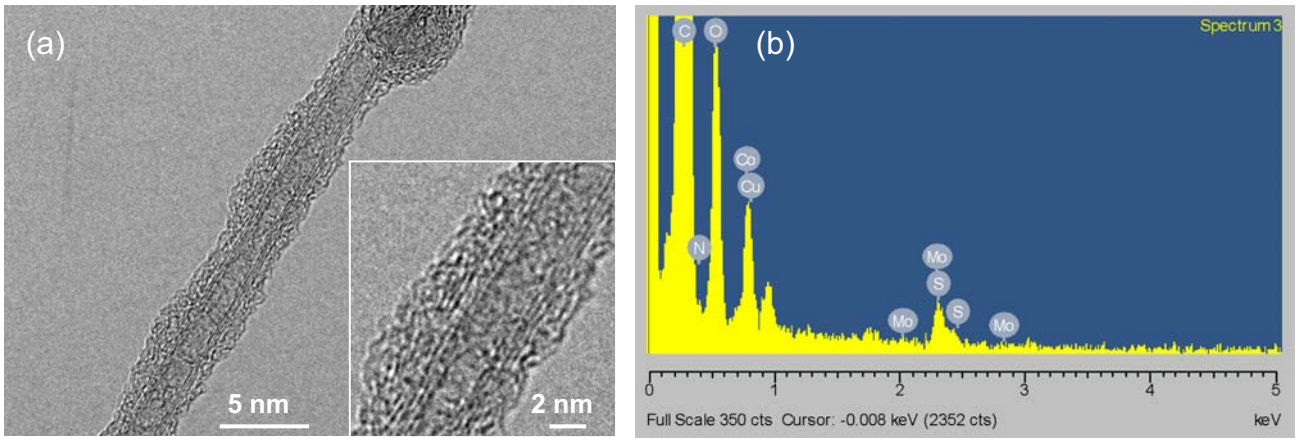

Fig. 1. (a) TEM image of a functionalized CNT; (b) EDS spectrum.

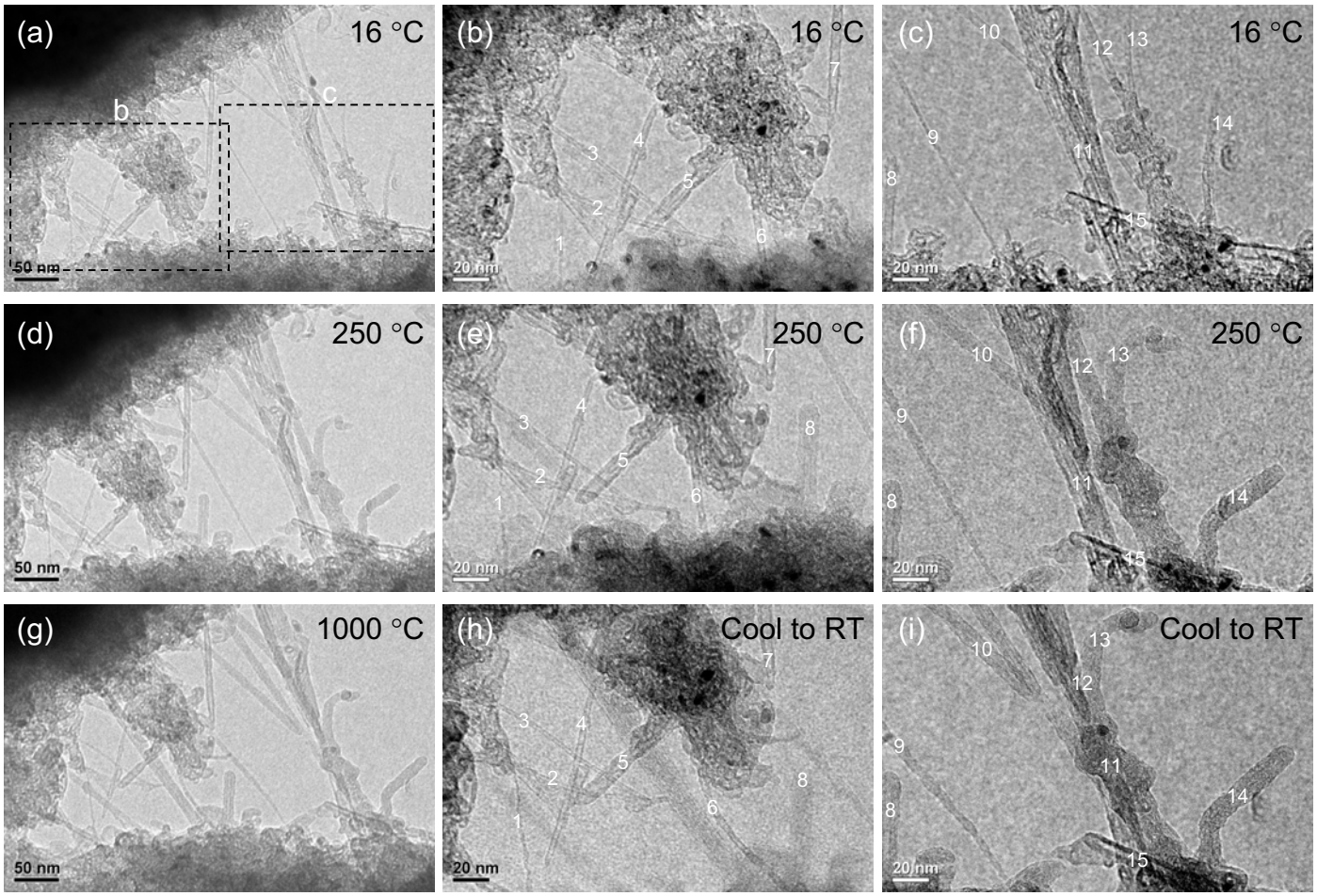

Fig. 2. TEM images of the same area during *in-situ* heating. (a-c) Room temperature; (d-f) 250 °C; (g) 1000 °C; (h, i) cooled down to room temperature.

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