DOI: 10.1017/S1431927605500175
Microsc Microanal 11(Suppl 2), 2005

Irradiation-Induced Structural Re-Organization of Carbon Aerogel and Its Derivatives in TEM

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Carbon aerogel (CA) is a relatively new type of carbonaceous materials that was first discovered in late 1980s [1]. The structural characteristic of CA is defined by a mesoporous network of carbon the pore size of which can be controlled according to the conditions of the polymeric reactions from which the material is synthesized. Normally, the type and properties of the pore structure of these materials have largely been the subject of physisorption method. Transmission electron microscopy (TEM) and scanning electron microscopy (SEM) have been used for direct imaging of the internal structures of the material. In this paper, we describe some novel behaviors of regular CAs and their derivatives when they are subject to enhanced electron irradiation in a JEOL JEM 2010 FasTEM operating at 200kV.

Fig 1 is a TEM image of a regular CA sample. The tortuous carbon network is clearly visible in addition to the presence of some pores. This typical microstructure is very stable at normal imaging conditions in the TEM used. However, significant structural re-organization has been routinely observed at enhanced current density (200-500pA/cm^2 as detected on the viewing screen, as contrasted to ~40pA/cm^2 for imaging). The result is onion-like structures with rather uniform sizes (fig 2). The radiolytic formation of carbon onions has been reported elsewhere [2-3] but this is the first time such phenomenon is observed on carbon aerogel. The mechanism for this structural change is widely believed to be that of the "knock-on" effect of the energetic electrons in a TEM [4]. In our experiment, regular CA has been modified into more graphitized type (fig 3a) to improve its heat resistance. Such graphitzation occurs at a very local scale so that the original pore structure is not severely perturbed. It is found that this derivative of CA also transforms into carbon onions at similar conditions in the TEM. Fig 3a-3f is a time series of such transformation. It is noted that such transformation is usually completed in less than 10 mins or so and areas not affected by the electron beam still sustain their regular structure. We further modify the graphitized CA by incorporating ruthenium particles and subsequently encapsulating them (fig 4a). We note that the carbon cage spontaneously shrinks under irradiation, evolving into a more compact carbon onion. The metal particles gradually egress outside until both are separated (fig 4b).

These findings help us understand the behavior of carbon at extremely small scale and off-equilibrium conditions. The formation of carbon onion is of particular interest since it may provide valuable information about the large-scale fabrication of this high-temperature, high-pressure solid lubricant.

References

[1] R.W. Pekala, J.Mater. Sci. 24 (1989) 3221.
[2] D. Ugarte, Nature 359 (1992) 707.
[3] F. Banhart, P. M. Ajayan, Nature 382 (1996) 433.
[4] F. Banhart, Rep. Prog. Phys. 62 (1999) 1181.

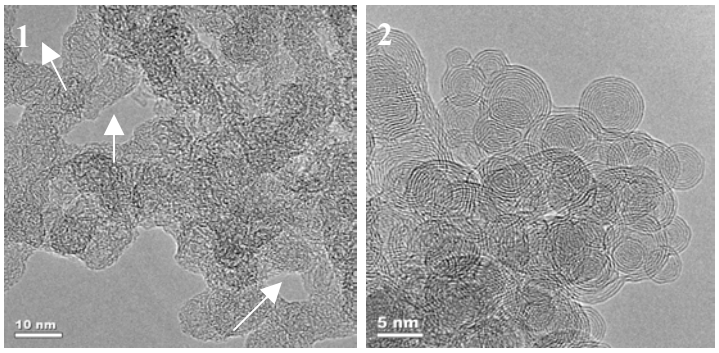

Fig. 1. TEM micrograph showing the typical microstructure of carbon aerogel. Note the presence of tortuous carbon network and mesopores (arrowed features).
Fig. 2. A cluster of carbon onions of uniform diameters formed from the TEM irradiation of regular carbon aergel similar to that shown in fig. 1.

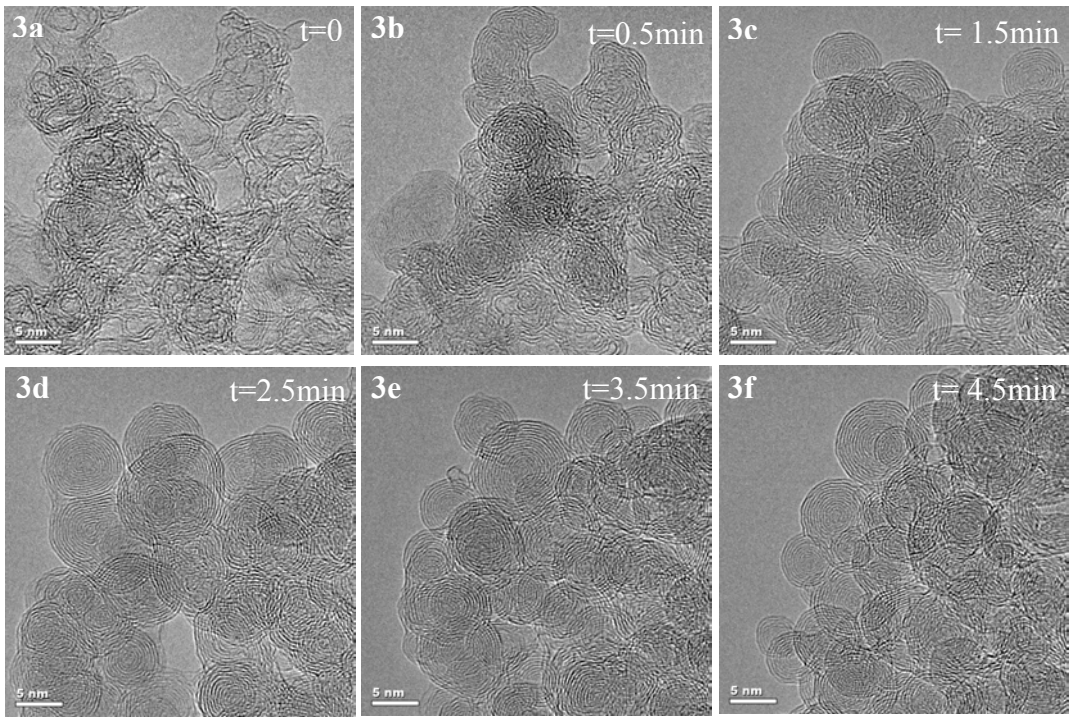

Fig. 3. A time series of TEM images showing the *in-situ* formation of carbon onions under enhanced electron irradiation. The sample was partially-graphitized carbon aerogel the initial structure of which is shown in 3a.

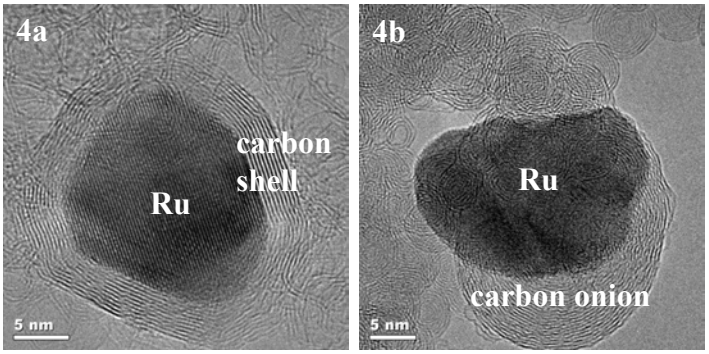

Fig. 4. Carbon-encapsulated ruthenium particle derived from the modification of regular carbon aerogel (a) dissociates into a free metal particle and a compacted carbon onion under the effect of elevated irradiation in TEM (b).

https://doi.org/10.1017/S1431927605500175 Published online by Cambridge University Press