Laser Annealing Nanoscale Carbons: Material Transformations

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ABSTRACT

Laser processing of materials is not new, but using lasers to anneal, bond and otherwise transform carbons is. Fundamental understanding of the dependence upon carbon structure, morphology and chemistry is critical to implementing this technology into manufacturing and processing applications. Here initial results using pulsed laser annealing of nanocarbons are performed using a Nd:YAG laser operating at 1064 nm and laser fluence of 250 mJ/cm^2. A gated intensified CCD captured the spectra of the material incandescence, fitting the spectral profile yielded a temperature of 2800 K for this laser fluence. To resolve detailed morphological and nanostructural changes induced in the nanocarbons by the pulsed, high-intensity laser light, high-resolution transmission electron microscopy (HRTEM) is applied while electron energy loss spectroscopy (EELS) is applied to examine local sp^2/sp^3 chemistry. The annealing process accentuated the recognizable structural differences and led to graphitization of lamellae, but the spatial organization of lamellae was quite different across carbons tested. Initial nanostructure in conjunction with the chemistry of construction appears to govern the carbon transformation under pulsed laser annealing.

Keywords: laser, carbon, annealing, graphitization

1 INTRODUCTION

An investigation of the structural changes due to the different time scales of high temperature treatment on nanoscale carbons is presented, using pulsed laser annealing. To resolve detailed nanostructural changes, high-resolution transmission electron microscopy was used to examine the carbon before and after heating. Pulsed laser annealing process led to graphitization of lamellae and also accentuated the recognizable structural differences between the different carbons. Several structures are reported here, such as partial graphitization, formation of hollow particles, rosette structures, and multi-compartmented nanospheres.

2 MATERIALS AND METHODS

A pulsed laser induced annealing with high resolution microscopy coupled with post-image processing provided analysis.

2.1 Carbons

Nanoscale carbon materials were studied, including 0- 1- and 2-D allotropes in the form of fullerenes, nanotubes and graphene. Other materials included nanoscale carbon particles with fullerenic, amorphous and graphitic nanostructures.

2.2 Methods: Laser and HRTEM

Pulsed laser annealing was performed using a Nd:YAG laser operating at 1064 nm and a laser fluence of 250 mJ/cm^2. Multi-wavelength pyrometry was applied for temperature determination. A gated intensified CCD captured the spectra of the material incandescence, fitting the spectral profile to Planck's equation yielded a temperature of 2500 ^{0}C for this laser fluence. HRTEM images were taken using a 200 keV field emission TEM (JEOL® EM-2010F) instrument. The applied magnification was set at 40,000x for morphology investigation, and 500,000x for nanostructure analysis. Image analysis of the nascent carbons was performed using custom algorithms to quantify the nanostructure observed within the HRTEM images.[1,2]

3 RESULTS AND DISCUSSION

Figure 1 highlights by HRTEM the differences in the annealed carbons – quantified by lattice fringe image analysis as reported elsewhere. Under thermal equilibrium conditions carbon structure transforms towards a stable graphitic structure, this process is termed "graphitization[3-5]. In pulsed laser annealing, the materials transformations occurs within nanoseconds while the carbons are at elevated temperatures, namely above 2000 ^{0}C, a temperature commonly considered as a graphitization threshold based on traditional heating studies. Lamellae mobility is enabled and the soot nanostructure can relax into a more thermodynamically stable graphitic lattice structure. Formation of closed structures during the layer plane growth is energetically favorable since flat graphitic units of small size would still contain a large fraction of high-energy carbon atoms with dangling edge bonds. Small differences in nanostructure evolve to substantial differences upon fast annealing.

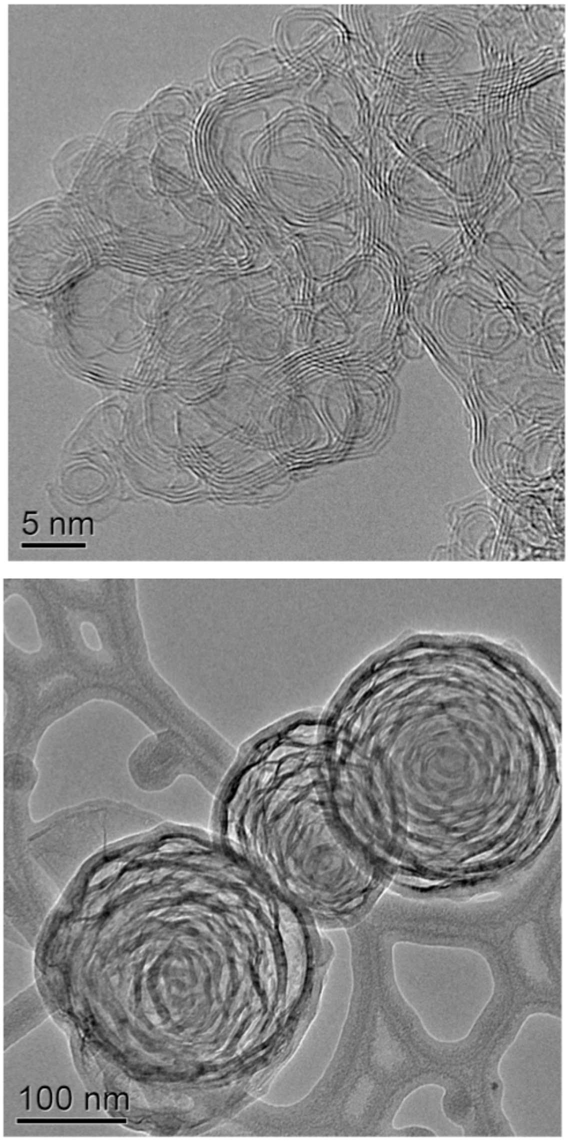

Figure 1. HRTEM images of the laser annealed carbons showing nanostructure of particles with the differences due to original starting material – accentuated by the transient heating action by the pulsed laser irradiation.

4 CONCLUSIONS

The action of pulsed high intensity laser light induced nanostructure changes. Visual inspection of the laser annealed HRTEM images indicates extensive graphitization with formation of shell-like, rosette and nested compartment structures depending on the nascent nanostructure and chemistry of the carbon sample.

REFERENCES

[1] Yehliu K., Vander Wal, R. L., Boehman, A. L. (2011). Development of an HRTEM image analysis method to quantify carbon nanostructure. *Combustion and Flame* 158:1837–51

[2] Yehliu, K., Vander Wal, R. L., Boehman, A. L. (2011). A comparison of soot nanostructure obtained using two high resolution transmission electron microscopy image analysis algorithms. *Carbon* 49:4256–68.

[3] Gadiou, R., Didion, A., Saadallah, S-E., Couzi, M., Rouzaud, J.-N., Delhaes, P., Vix-Guterl, C. (2006). Graphitization of carbons synthesized in a confined geometry. *Carbon* 44:3348–52.

[4] Oberlin, A. (1984). Carbonization and graphitization. *Carbon* 22:521–541.

[5] Franklin, R. E. (1951). Crystallite growth in graphitizing and non-graphitizing carbons. *Proc. Royal Soc. London, Ser. A* 209:196–218.