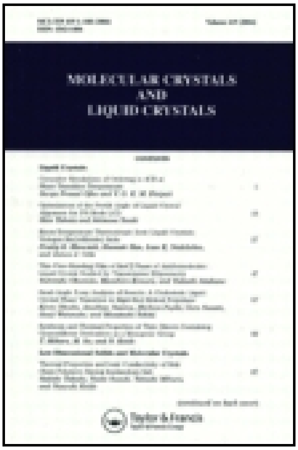


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# Hrtem observation of ball-milled lampshade carbon nanofiber

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# HRTEM OBSERVATION OF BALL-MILLED LAMPSHADE CARBON NANOFIBER

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*High-resolution transmission electron microscopy study of ball-milled lampshade carbon nanofibers is reported in the present paper. Milled fibers became shorter in length, preserving the lampshade structure with highly ordered graphene layers. We have also shown the simulated model for the shortening process of the fiber.*

*Keywords*: nanofiber; TEM; lampshade; milling

## INTRODUCTION

Recent production of cup stacked type pyrolytic carbon nanofiber obtained by floating reactant method opened a new field of application for the nanofibers [1,2]. Among them, the open-ended type cones will be referred to as lampshade in this paper. The lampshade carbon nanofiber differ from conventional catalytically grown carbon nano-fiber/tube in that the former has the stacked lampshade morphology, while the latter has the well-known tubular morphology at the sidewall of the fiber [3–6]. This structural difference leads to completely different physical, chemical, and electronic properties compared to the conventional fibers and tubes. Since the edge site of each graphene sheet is exposed both at the outer and inner side of the tube, lampshade fiber has a high reactivity with foreign species

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enabling the functionalization of the fiber. The striking characteristic of the lampshade nanofiber is the active site in the inner wall of the fiber. In order to maximize the accessibility to the inner wall, we have ball-milled the lampshade fiber to shorten the length of the lampshade nanofiber which is usually $10 \sim \mu m$. In the present study, we report the high-resolution transmission electron microscopy (HRTEM) study on the ball-milled lampshade nanofiber to reveal the microstructure of the milled nanofibers.

## EXPERIMENTAL

The lampshade nanofibers used in this experiment were prepared by floating reactant method using ferrocene as a catalyst. It was then ball-milled for 24 hours. We have observed the specimen using a JEOL JEM-2010FEF field emission TEM operated at 200 keV equipped with a $\Omega$-type energy filter.

## RESULTS AND DISCUSSIONS

Figure 1 shows the TEM images of the as produced and milled lampshade nanofiber. The magnification is adjusted so that the whole part of the nanofiber is included in the image. We can see that the length of the fiber is clearly shorter in the milled sample. Looking into the microstructure shown in Figure 2, it is clear that the lampshade structure with well-aligned layers

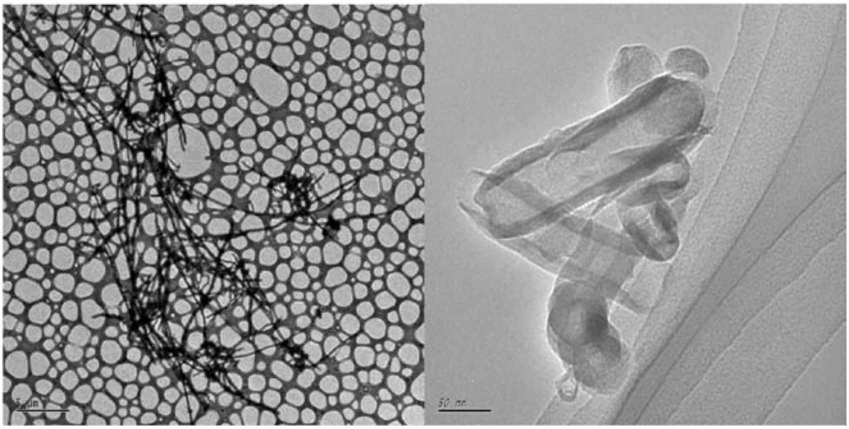

**FIGURE 1** TEM image of as prepared (left) and milled (right) lampshade carbon nanofiber. Intentionally changed the magnification of the images to show the difference in length.

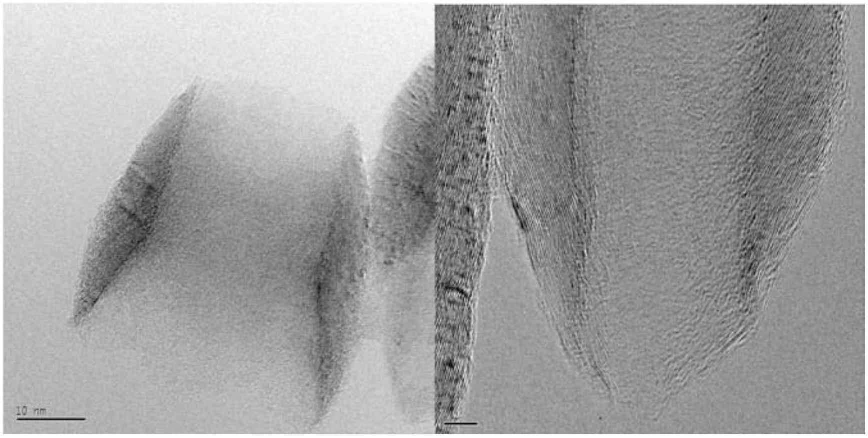


**FIGURE 2** High resolution TEM image of shortened lampshade carbon nanofiber. Well aligned layer structure is preserved after milling.

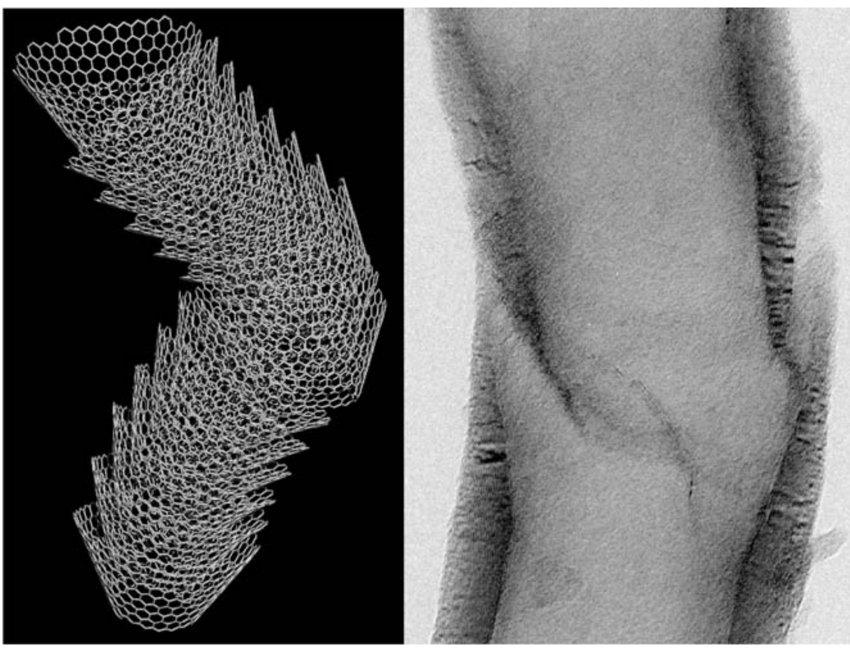

**FIGURE 3** Computer simulated lampshade nanofiber under bending stress (left) is compared with a TEM image of a milled lampshade nanofiber which has undergone similar bending stress (right).

is preserved even after the milling, and only the length of the fiber became shorter. The sharp spot appearing in the FFT image also proves the highly crystalline structure of the shortened nanofiber.

The result of the simulation of the deformation process of the lampshade nanofiber under bending stress is shown in Figure 3. We have changed the angle of the lampshade molecules at both ends to simulate the bending stress, and relaxed the structure using empirical potential MM+ to obtain the final structure. First, the fiber starts to bend according to the stress, and a severe deformation of the fiber starts to appear in several lampshades molecules where the stress is concentrated. When the stress concentration and the resulting deformation of the molecules exceed some critical point (estimation is underway), the lampshades start to slip out from the adjacent molecule, and shortened stacked lampshade unit appears. Since the milling process is virtually an application of continuous bending stress to the fibers, the fibers will become shorter as far as the bending stress can be applied. Thus we can conceive that there is a limit in shortening the fiber. It is proved in the experiment with longer milling time, which destroyed both the layer structure and the lampshade structure of the fiber.

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