PRODUCTION OF SINGLE WALLED CARBON NANOTUBES USING TUNABLE RADIATION FROM A FREE ELECTRON LASER (FEL)

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Introduction

Since the discovery of carbon nanotubes[1] different synthesis techniques, like Pulsed Laser Vaporization (PLV) [2], arc discharge [3], Chemical Vapor Deposition (CVD) [4], have been developed to improve both the quantity and the quality of this material. The free Electron Laser (FEL) provides a powerful, tunable and unique pulse train for PLV production of nano-filaments. In this article, we present the first results using an FEL to synthesize single walled carbon nanotubes (SWNTs).

Experiment

The details of the target preparation of the metallic catalytic targets have been reported elsewhere [5]. The targets were prepared by using different catalyts promoters: transition metals (e.g., Ni, Co, Fe) and rare earths (e.g., Y). These SWNTs samples were characterized by Raman spectroscopy and electron microscopy. A schematic diagram of the experimental set up at the FEL is shown in Fig. 1. The FEL beam at the Thomas Jefferson National Laboratory is controlled and aligned in the control room and directed onto a mirror wobbler, which scans it on the carbon target (Fig. 1). First the system with a mounted target is evacuated to $\sim10^{-3}$ Torr and heated up to 1000°C at ~3°C/min for the high temperature run. Argon gas at a flow rate of 100 sccm is admitted upstream into the system (as shown).

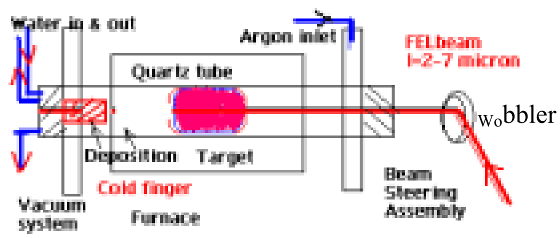

Fig. 1. Experimental set up for the SWNT growth by FEL.

The FEL beam was focused into a ~1 micron spot on the target. The experiments were performed at room temperature and 1000°C with laser parameters, 18MHz, 35MHz and 75MHz repetition rate; 200W, 250W and 300W laser power; 3, 5 and 6 micron wavelengths. The soot from each of the runs were analyzed by high-resolution transmission electron microscopy (HRTEM) [JEOL JEM1200, operating at 120kV] and Raman Spectroscopy [Nd:YAG laser, λ=1064 nm, T=300K].

Results and Discussion

Figure 2 shows the transmission electron micrographs for SWNTs prepared by FEL using Ni-Y catalyzed carbon target at two different temperatures. The SWNTs synthesized at RT have a larger diameter (~2.4 nm) in comparison to the soot synthesized (~1.5 nm) at 1000°C. The SWNTs synthesized at 1000°C have diameter similar to those prepared by table top PLV and arc-discharge methods.

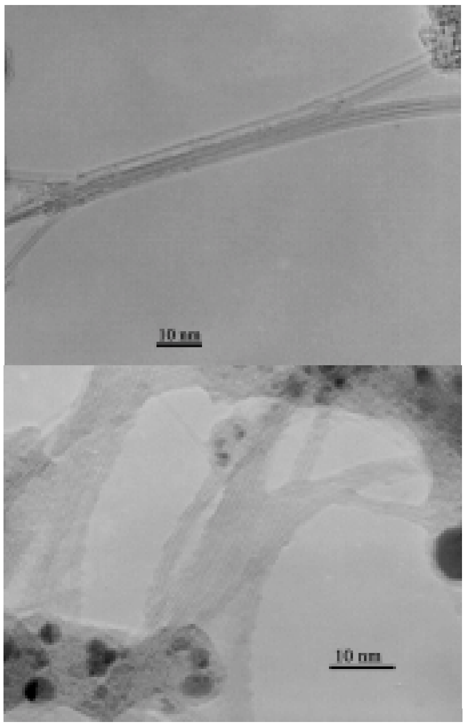

Fig. 2 TEM image of SWNTs, (top) room temperature, (bottom) 1000° C.

Raman spectra of the SWNTs grown with same experimental parameters from targets with different composition have been compared (Fig. 3). All SWNTs showed two distinct characteristic peaks; the higher (~ 1591 cm^{-1}) and the lower frequency (~ 160 cm^{-1}) peak correspond to the tangential and radial breathing mode, respectively. All samples prepared from three composites have same peak positions of these two modes. The calculated diameter of the tubes is found to be almost same (~1.5 nm) for all three targets.

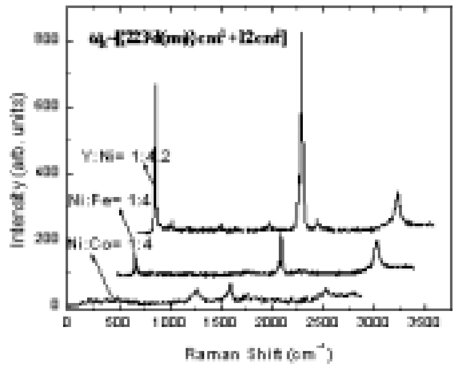

Fig. 3 Raman spectra of SWNTs prepared from different targets.

Samples prepared from Y:Ni and Ni:Co composites produced the strongest and weakest Raman nanotubesignal, respectively. The tubes from, Y:Ni targets also showed intermediate frequency Raman-peaks as observed in very good quality SWNTs with small diameters[6].

Conclusion

The FEL is found to be a promising source for the large production of SWNTs using pulsed laser vaporization. Among the three different targets SWNTs prepared from Y:Ni targets is found to be of very good quality. A current problem with the FEL set up is very high rate of production of soot. We are currently re-designing the chamber and optimizing the growth parameters to take advantage of the high soot production.

Acknowledgments

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