# Manufacturing Carbon Nanotubes Using Chemical Vapor Deposition

By: Charles Widmer

Submitted to:
Frank LaBanca, Ed.D.
Science Department Chair
Oxford High School
June 8, 2010

**Abstract**

In today's science world, Carbon Nanotubes have risen to the peak of fame, both the qualities of high tensile strength and high conductivity making them prime candidates for many different fields. The biggest issue with these tubes, though, is the fact that they cannot easily be manufactured to specific standards. Since their discovery in 1991, scientists worldwide have been searching for different ways of perfecting the creation of Carbon Nanotubes. One technique is called Chemical Vapor Deposition which involves extremely high temperatures to create the Carbon Nanotubes. Using different kinds of surfaces has led to a different growth pattern within the Carbon Nanotubes, some surfaces even creating Carbon Nanotube coils (springs). In this project, Carbon Nanotubes were created and, through the use of an Indium Tin Oxide coated surface, the Carbon Nanotubes formed traditional tubes and Springs.

**Introduction**

In 1991, a Japanese researcher by the name of Sumio Iijima discovered Carbon Nanotubes by sheer luck, his goal originally being to create fullorines. Since the discovery of these tubes, scientists worldwide have been researching intensively, trying to find all of the properties of the Carbon Nanotubes. The intensity at which Carbon Nanotubes are researched is at an extreme, close to seven research papers a day being published with Carbon Nanotubes as the main subject. The biggest issue with Carbon Nanotubes has been trying to make them to certain specifications. Companies have been desperately searching to find new, more affordable methods of creating Carbon Nanotubes, ones that would lead to a more simple separation of the tubes later on or just certain kinds of growth patterns of the Carbon Nanotubes. Different methods have been found to prove effective in the differentiating of the growth pattern of Carbon Nanotubes. The most common and effective method for creating nanotubes to certain specifications has been using different surfaces.

Chemical Vapor Deposition (CVD) is one of the many techniques used to make Carbon Nanotubes. It involves the heating up of a carbon source to extremely high temperatures in order to break up the carbon atoms. Then, the carbon rearranges itself on a surface with a heated up catalyst, causing the growth of Carbon Nanotubes. Researchers have begun worldwide to use different surfaces, some using Silicon Wafers, which resulted in a straight growth of Carbon Nanotubes rather than bundled up forests.

For Carbon Nanotube Springs to be created using CVD, there had to be a different kind of surface involved. Thanks to Astra Products, an Indium Tin Oxide coated plexiglass was now the surface to be used for Carbon Nanotube Springs.

The coiling of the Carbon Nanotubes occurs because of the pores within the plexiglass. The Indium Tin Oxide, when used to grow the Carbon Nanotubes, gives the nanotubes a twist as they grow outwards, creating Carbon Nanotube Springs. Since these tubes are at the nanoscale, the only possible way to view them is by using an SEM.

To view Carbon Nanotube Springs, a Scanning Electron Microscope (SEM) is needed. These special microscopes can view objects up to 1nm, some even closer than that. The way an SEM works is simple. A beam of electrons is sent out onto the sample. For this to work, the sample must be coated in a conductive substance, usually a thin layer of gold. When the electrons come into contact with the conductive surface, the electrons on the conductive surface become charged. The newly charged electrons send out a signal, one that is read by a special detector within the SEM. The detector converts the signals into an image that can be easily viewed.

**Materials**

| Chemicals/Consumables | Supplies | Equipment |
|---|---|---|
| Nitrogen<br>Iron Catalyst<br>Propane<br>Cortz Tubing | Indium Tin Oxide coated<br>Plexiglas<br>Bunsen Burner<br>Nickle Wire<br>Heating Tubing<br>Stoppers<br>Clamps<br>Striker | SEM (Scanning Electron Microscope) |

## Procedures

*Creating the Setup*

For the proper creation of Carbon Nanotubes, a few key factors must be utilized:

- No oxygen can be present within the chamber where the reaction occurs.
- The carbon source must be heated up to roughly 900˚ C to allow the decomposition of carbon.
- A crystallized metal catalyst must be present for the broken carbon to realign.

Each piece of the setup has a key role in maintaining the proper environment for Carbon Nanotube growth. The quartz tube is the component heated up as the carbon source (Propane) passes through the chamber. A metal wire was wrapped around the tube, which was hooked up to a regulator. The purpose of the regulator was simply to allow the amount of voltage being pumped into the wire to be regulated. Stoppers were used throughout the setup to allow no oxygen to come into the system while it ran. For the main chamber, another larger piece of quartz was used. This allowed viewing of the reaction in a safer and more effective manner. For the catalyst to be heated up during the reaction, a second regulator was hooked up to a pair of wires. These wires were then connected to the thin wire wrapped around a piece of glass. This is where the substrate would be placed during the reaction. The two gasses used were Propane as the Carbon source and Nitrogen as the source gas.

*Mixing the Catalyst*

The catalyst was mixed using pure Iron and Acetone. First, the Iron was weighed out using the ratio 1 to 10; for every 1 gram of iron, 10 ml of Acetone were mixed in. A total of 0.5 grams of Iron were weighed and mixed with 5 ml of Acetone. The surface on which the catalyst would dry and be used was then submerged in the mixture, removed from it, and placed in a tube to crystallize. A plexiglass surface that had a thin coating of Indium Tin Oxide was used throughout this project, being the only surface on which the Carbon Nanotubes were grown.

*Carbon Nanotube Construction*

Once the catalyst had finished crystallizing, the whole setup was ready to run. First, the catalyst-coated plexiglass was placed into the larger quartz tube, sitting in the glass bed wrapped in wires. The Nitrogen gas was run as the small quartz tube was heated up using 25 volts of electricity to heat the wire around it. Once the tube was hot, the electricity was turned off on one and set up for the other. When the electricity for the cataylst was turned on, only 8 volts were needed. The instant that the electricity was turned on, the propane would be shut on as well to run through the system. Once the broken up propane came into contact with the catalytic surface, nanotubes were created instantly. This could be seen as a soot-like substance on top of the surface used. Observation of whether or not the tubes had grown correctly, or if they had grown at all for that matter, was not possible until further examination of the tubes under an SEM.

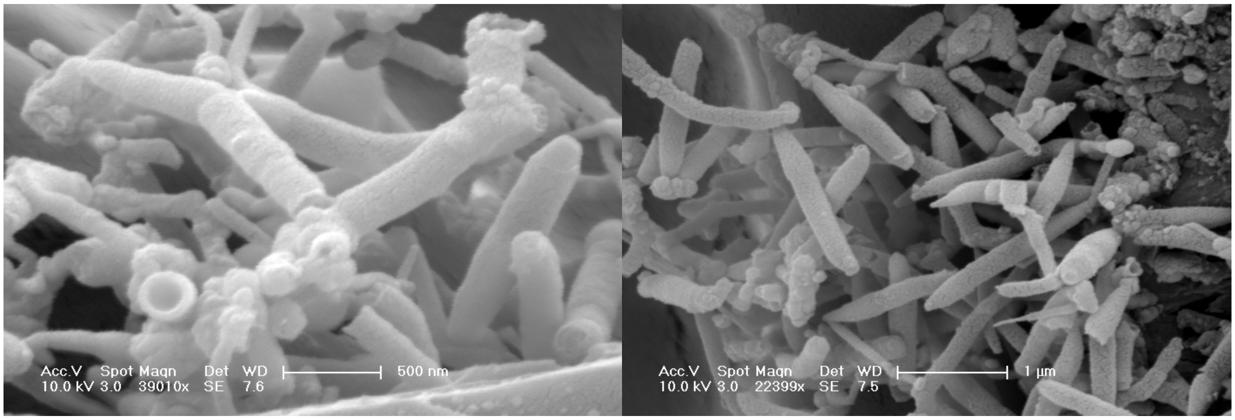
*Using the SEM*

A Scanning Electron Microscope (SEM) was personally used to view the results of making the Carbon Nanotubes. First, the sample had to be placed onto some conductive tape. The tape itself had a thin coating of conductive material along its surface. Due to the fact that the plexiglass nor the carbon were conductive surfaces, the whole sample had to be coated in a 3 nanometers of gold. This was done was by placing the sample in a cylindrical setup. All of the oxygen was removed with a vacuum and replaced with Argon gas. Once the gas had reached a certain pressure, tiny particles of gold were shot all over the sample for a total of 30 seconds. When the whole coating process was complete, the sample was removed and placed in the SEM. The coating of gold along with the tape now made the sample conductive, making it possible to view it on the SEM. For the SEM door to close and stay shut, a vacuum was turned on, sucking out most of the oxygen. Then the search for the Carbon Nanotube Springs began the very small sample now much larger when being viewed at 500 µm.

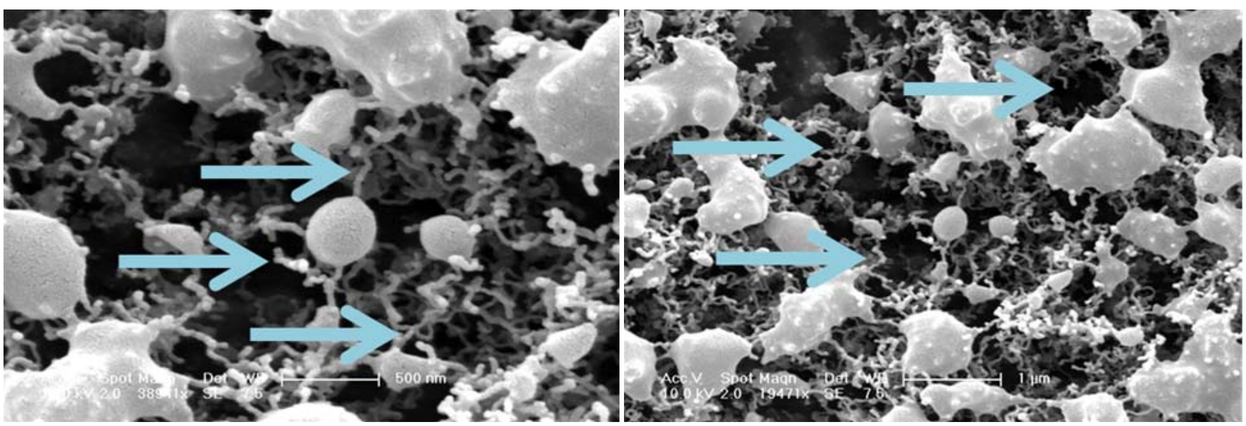
**Results**

(These are images of the Nanotubes that were created, taken with an SEM located in the Kline Geology Laboratory in New Haven, CT.)

Group of Carbon Nanotubes From One Sample




Carbon Nanotube springs growing off of Indium Tin Oxide Coated in an Iron Catalyst .



## Conclusion

From the images that were taken, it can be concluded that Carbon Nanotubes, when grown under specific conditions, will grow in a coiled alignment on an Indium Tin Oxide surface. The Indium Tin Oxide has proven to be efficient in coiling Carbon Nanotubes before and can now be validated through these results. Mainly due to expenses, a plexiglass surface was used and, even with extreme temperatures pounding down onto the plastic-like pieces, they surprising did not melt and were able to grow perfectly coiling Carbon Nanotube Springs. So, it can be concluded that the plexiglass, though not a completely efficient surface, will yield Carbon Nanotube Springs under the right conditions. Also, an SEM is an effective and accurate means of capturing the mere image of Carbon Nanotube Springs. Without it, the tubes could not be viewed, due mainly to the fact that the tubes are at the nano scale. There were some setbacks throughout the experimentation, one being the surface and how, at times, the melting was just inevitable. Also, there were no standardized procedures as well as a lack in the continuity of evaluation. Runs could not be done for more than a few seconds, the big fear being that the surface would melt due to the intense heat. A more efficient heating source for the surface which held the catalyst would have been effective but one was not used. CVD did prove to create Carbon Nanotube Springs consistently, though the intense heat caused problems for the surface used. A glass surface would also have proven more efficient and would probably have yielded more clear results as well as longer runs of the setup.

**References**

Chandler, D.L. (2009, September 21). Small Springs Could Provide Big Power. Retrieved from http://web.mit.edu/newsoffice/2009/super-springs-0902.html

Science News (2009, September 20). A recipe for controlling carbon nanotubes. Retrieved from http://esciencenews.com/articles/2009/09/20/a.recipe.controlling.carbon.nanotubes

Nednoor, Pramod, Gavalas, Vasilis G., Chopra, Nitin, Hinds, Bruce J., & Bachas, Leonidas G. (2007). Carbon nanotube based biomimetric membranes: mimicking protein channels regulated by phosphorylation. Journal of Materials Chemistry, Retrieved from http://www.rsc.org/publishing/publishing/journals/JM/article.asp?doi+b703365f

Yu, Jingxian, Losic, Dusan, Marshall, Matthew, Böcker, Till, Gooding, John Justin, & Shapter, Joseph George (2006). Preparation and characterization of an aligned carbon nanotube array on the silicon (100) surface. Soft Matter, Retrieved from http://www.rsc.org/delivery/_ArticleLinking/DisplayArticleForFree.cfm?doi=b611016a&JournalCode=SM

Liu, Cai-Hong, Liu, Yi-Yang, Zhang, Yong-Hui, Wei, Rui-Rui, & Zhang, Hao-Li (2009). Tandem extraction strategy for separation of metallic and semiconducting SWCNTs using condensed benzenoid molecules: effects of molecular morphology and solvent. Physical Chemistry Chemical Physics, Retrieved from http://www.rsc.org.publishing/journals/CP/article.asp?doi=b901517e