Large Sheets of Carbon Nanotubes Made by CVD

The Pitch

Carbon nanotubes (CNTs) are capped hollow cylinders of graphene (a 1-atom thick film of C atoms bound together with double electron bonds—sp^2 bonds—arranged in a honeycomb-lattice pattern similar to graphite) with exceptional properties. These include breaking strengths of ~30 GPa at a density of about 1.2 g/cm^2 compared with 2.2 GPa for steel (piano wire) that has a density of 7.8 g/cm^2. The thermal conductivity of CNT wires (>2000 W/K at the nanoscale) is many times that of copper and their electrical conductivity ($>2 \times 10^6$ S/m) is less than copper under dc conditions but will increase with frequency and certain types of dopants.

The commercial form of carbon nanotubes has been an extremely short, loose, powdery material that presents both inhalation and processing issues. For example, the solubility in epoxy of loose tubes is very low and in the laboratory has been measured at over 8% but in practice is about 1%. A type of CNT paper called Bucky paper has also been synthesized by first exposing the powdery tubes to nitric acid to modify their surface chemistry, then adding a surfactant, and finally creating a concentrated suspension that can be used to make the paper. The properties of this material bear little relation to the constituent tubes mainly because these tubes are short, usually multiwalled, and covered with a hard-to-remove surfactant. Laboratory techniques to grow forests of CNTs on a variety of substrates are useful and have the potential for synthesizing high quality yarn.

The company, Nanocomp, uses a very high temperature floating (iron) catalyst method to produce relatively long CNTs that are fabricated into sheets or yarn prior to removal from the “harvesting chamber.” These sheets (shown in Figure 1) are presently as long as 2.4 m. In addition, the sheets can be joined to produce long panels that can be “prepregged” on commercial resin pre-impregnation systems (shown in Figure 2), thereby supplying CNTs in a form amenable for safe handling, compositing, and EMI shielding.

Applications for CNT sheets include EMI shielding from a few kHz to >1 GHz; composites (breaking strengths of more than 2 GPa have been reported); conductors (the sheet material in tape form is effective in shielding EMI emissions from cables and the tapes make effective high current conductors); and heaters that can be embedded into a composite or used externally.

Figure 1. A long roll of carbon nanotube (CNT) cloth, 1.2 m wide with a density of 15 g/m^2.

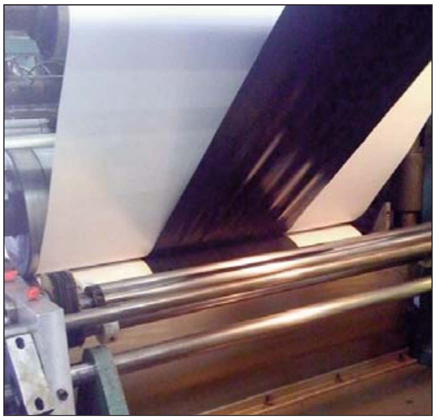

Figure 2. A roll of CNT cloth being prepregged with a commercial resin on industrial machinery.

It has been estimated that the present market for CNT materials is about $90.5 million. Currently, nanotubes represent a niche material market that has high revenue potential. New functionalized nanotubes applications are expected to enter the market in the next few years that are anticipated to greatly increase global revenues to ca. $1.4 billion by 2015, driven by the requirements of the electronics, data storage, defense, energy, aerospace, and automotive industries.

The Technology

The chemical vapor deposition (CVD) process used by Nanocomp to produce sheets of CNTs uses a carbon-based fuel such as ethylene or an alcohol. This fuel is caused to thermally decompose or “crack” before being exposed to an iron catalyst particle that has a diameter about the same or slightly larger than the diameter of the tube. It is postulated that the tube formation process involves a catalytic decomposition on the particle surface followed by complete saturation of the iron cluster with carbon. The next carbon atom “in” sits on the surface of the iron catalyst and combines with subsequent carbon atoms as sp^2-sp^3–bonded graphene that forms a cap or half a Buckyball sphere. Surface tension then pulls this cap off of the particle and subsequent graphene forms an sp2-bonded cylinder. During this process temperatures are sufficiently high and the particle size so small that it is believed that the catalyst particles are liquid during much of the growth. Large sheets of CNTs are formed by causing the cloud of CNTs to deposit on a moving cylinder or belt. The material builds up like thin layers of phyllo dough laid one on top of the other as the belt slowly translates back and forth. The nature of the CNTs can be tailored by means of the process parameters from single wall (SWCNT) to dual wall (DWCNT) or to multiwall (MWCNT).

Opportunities

Nanocomp Technologies is interested in joint opportunities for integration of their CNT sheet material into applications for structural composites, electrical properties, and EMI shielding.

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TECHNOLOGY ADVANCES seeks materials developments on the threshold of commercialization. Send suggestions to Renée G. Ford, Renford Communications, renford@comcast.net.

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Compact Field-Emission SEM Invented for Low Voltage Imaging

The Pitch

A low-cost, high-performance compact field-emission scanning electron microscope (SEM) has been designed and built by the company, Novelx. This represents a major innovation in how SEMs are built and operated. By leveraging silicon processing technologies, Novelx has miniaturized the core technology inside an SEM. This miniaturization enabled the design of an all-electrostatic electron beam column that when coupled with a thermal field emission electron source is optimized for low-voltage imaging at below 10 nm resolution. These imaging capabilities were previously only available in high-end, expensive, and much larger sized field-emission SEMs that are typically located at centralized core imaging facilities.

Advances in the field of nanotechnology have resulted in a drive toward smaller length scales that now spans all industries. Researchers and developers can no longer rely on optical microscopes exclusively for inspecting and evaluating materials. Optical microscopes are generally limited to the micron range by diffraction effects. Thus they cannot image the most promising and innovative technologies occurring in the nanoscale range. SEM imaging capabilities are typically located at centralized core imaging facilities, thus not conveniently available. Moreover, purchasing a SEM is often prohibitively costly. Even when funds are available, there is often inadequate space to house conventional SEMs, which are about the size of a refrigerator, which often require their own room with dedicated facilities and a dedicated operator, and consume a great deal of power.

The overall SEM market is about $500 million annually and the low-end benchtop SEM market was estimated in 2009 to be about $200 million. With the only research-grade compact field-emission SEM available called the *my*SEM (shown in Figure 1), the market for Novelx is estimated to be about $400 million and positioned for rapid growth.

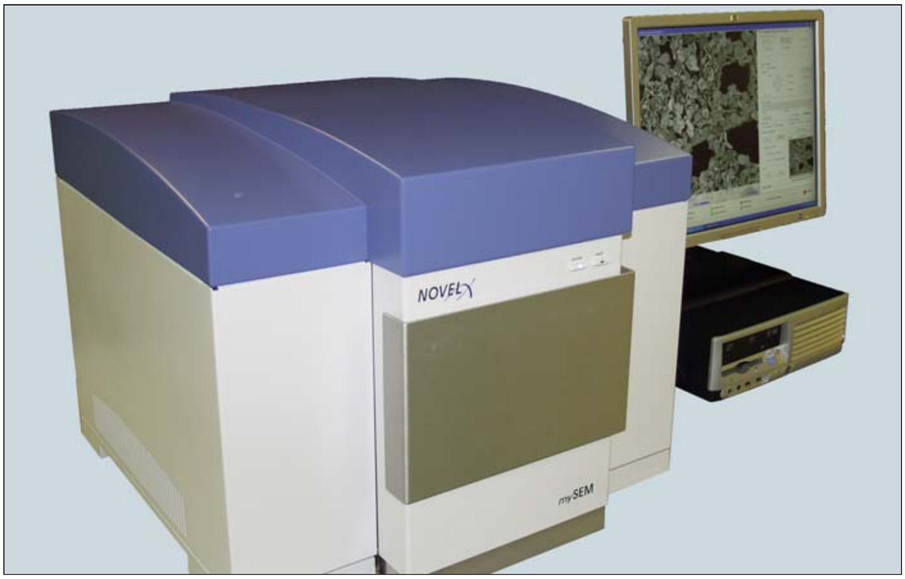

Figure 1: Novelx *my*SEM, a compact field-emission scanning electron microscope (SEM).

The Technology

The core technology inside a SEM is the electron beam column whose function is to extract, collimate, shape, scan, and focus the electron beam. The electron optics of a conventional electron beam column rely on precision-machined electromagnetic elements to control the electron beam. At the system level, closed-loop cooling and sophisticated vibration isolation is generally required to manage the high currents in the lenses and to deal with the enhanced sensitivity to external vibration due to the large size of the column. The result is a high-resolution electron beam column, but one that is expensive, large, and requires dedicated facilities. This same high-resolution performance can now be realized at a significantly lower cost and in a smaller size with the miniaturization of an all-electrostatic electron beam column.

Novelx uses stacks of silicon-on-insulator to form all the lens, apertures, and deflectors needed in the electron beam column. This patented Stacked Silicon Technology™ enables Novelx to build multiple lenses, deflectors, and apertures wafer-scale on 150 mm substrates. The company is leveraging silicon processing technologies to build SEMs in a unique way—wafer scale and orders of magnitude smaller than conventional SEM technology. The innovative design challenges large electron microscope suppliers by making compact but high-performance SEMs available to individual researchers and developers in their own laboratories at a price comparable to that of a high-end optical microscope.

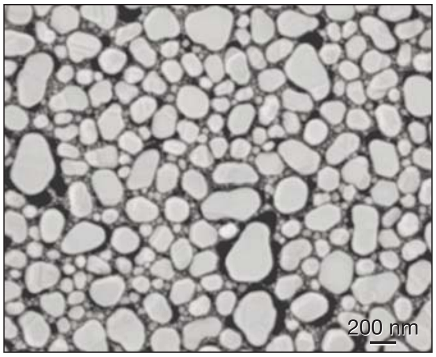

Figure 2: Novelx *my*SEM image of gold islands on carbon demonstrating 123,159 × magnification with a 3 μm field size.

The Novelx *my*SEM is now being used for imaging nanoscale objects and materials. As shown in Figure 2, the *my*SEM provides the same topographic and spatial information about surfaces as a conventional high-performance field-emission SEM. It is expected to find broad application wherever it is important to understand the surface features or the shape of objects and materials. With dimensions continuing to decrease according to Moore's Law, the semiconductor industry will increasingly need to rely on compact field-emission SEMs more often for quality control and defect analysis of semiconductor materials and electronics. The low-voltage imaging capabilities of the *my*SEM are particularly well-suited for the new clean tech industry and will be useful for imaging and inspecting thin film solar cells as well as fuel cell membranes.

Opportunities

Novelx, Inc. is interested in collaborating with researchers to evaluate new material analysis capabilities planned for the Novelx *my*SEM platform.

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Microwave Technology for Rapid Thermal Processing Reaches Ultrahigh Temperatures

The Pitch

Rapid thermal processing (RTP) is a common technology used in semiconductor manufacture and materials engineering to achieve desired properties for devices and materials. "Hotter and faster" are two market trends driving the growth in the RTP equipment industry. For example, the increasing demand for emerging compound semiconductors such as SiC and GaN creates a need for RTP of semiconductor at much higher temperatures than those for current Si technologies (e.g., 2000°C for SiC versus 1200°C for Si). In addition, continued miniaturization of silicon devices to nanometer length scales, such as <65 nm node metal-oxide-silicon (MOS) devices, requires significantly shorter thermal cycles (e.g., millisecond annealing).

The current halogen-lamp–based RTP technique developed for Si technology can no longer meet the challenge of the hotter and faster trends due to their incapability of reaching RTP temperatures well above 1200°C because the lamp quartz envelope softens at 1300°C. The current technique also had difficulty in achieving millisecond annealing because the lamps need to simultaneously heat both the wafer and the surrounding glassware.

To meet the critical needs of future RTP technology, LT Technologies (LTT) has invented a novel microwave RTP technology capable of reaching ultrahigh temperatures higher than 2000°C within a fraction of a second. The specific advantages of ultrahigh temperature and ultrafast heating make LTT's microwave RTP technology well-positioned to facilitate the transition of RTP equipment from current levels to much "hotter and faster" levels.

The overall semiconductor RTP equipment market, which was $450 million in 2006, is projected to double by 2011. The market can be divided into three segments: traditional silicon RTP, compound semiconductor RTP, and millisecond RTP. The two targeted markets for LTT's products, that is, compound semiconductor RTP equipment and millisecond RTP equipment, are relatively small today. However, they are expected to grow at a higher than 50% rate per year over the next five years and to replace much of the traditional silicon RTP equipment.

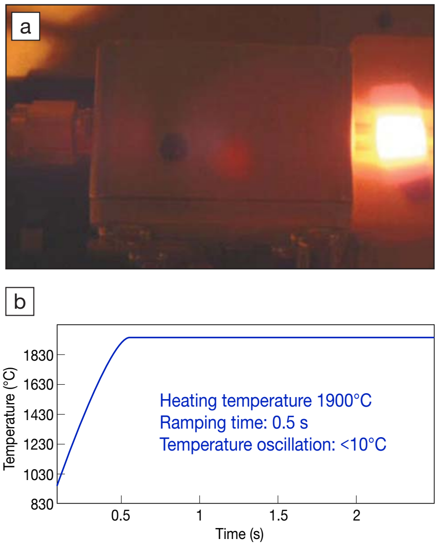

Figure 1: (a) Heating of a SiC sample with a microwave heating head; (b) plot of the temperature profile from the microwave heating of SiC.

The Technology

The microwave RTP apparatus developed by LTT consists of three main components: (1) a solid-state variable frequency microwave power source to generate high-power, short duration microwave pulses; (2) a microwave heating head to effectively couple microwave power from the source to the heating target; and (3) a PC-based control system to modulate the microwave pulses and to regulate the temperature uniformity and stability. Figure 1a illustrates the heating of a SiC sample with a microwave head where the sample is closely placed on the top of the microwave head and directly exposed to strong microwave radiation. A typical temperature profile of microwave heating is plotted in Figure 1b. It can be seen that the ultrahigh temperature of 1900°C was reached in 0.5 s and the temperature oscillation during heating was well-controlled within 10°C.

In addition to rapid and high-temperature heating, the use of microwaves has the advantage of selectively heating the material of the strong microwave absorber in a heterostructure. This selective heating allows the microwaves to heat only the high electrically conductive doping areas of the device layer while having little thermal effects on the other parts of the wafer volume such as lightly doped substrates, oxide layers, and nitride gates for which microwave absorption is poor. For microwave RTP of large wafers, an array consisting of multiple microwave heads and multiple temperature sensors is introduced. The number and size of the heating heads are flexible depending on the size of the wafer and the requirement of temperature uniformity. A multiple head control system is being developed to run and to control the RTP of larger wafers.

LTT has conducted extensive research on microwave RTP of SiC and GaN. The results demonstrate that microwave RTP technology may offer significant improvements in the electrical properties and quality of SiC and GaN semiconductors including: (1) unprecedented ultralow sheet resistance, (2) very high carrier mobility, and (3) near-perfect lattice damage recovery and defects elimination. In addition to the RTP of semiconductors, LTT's microwave technology also can be used in applications for developing advanced materials where ultrahigh temperatures and fast heating are needed such as the rapid sintering of ceramics, rapid oxidation and nitridation, high-yield growth of nanomaterials, and solderless bonding of microelectromechanical systems and microelectronics.

Opportunities

LT Technologies microwave RTP equipment and the technology (patent and pending patents) are available for purchasing, licensing, and/or collaborative research. LTT also offers the service of performing microwave RTP tests to meet the needs of its customers.

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