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Marquis, Fernand D.S.

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Carbon Nanotube Nanostructured Hybrid Materials Systems for Renewable Energy Applications

Fernand D.S. Marquis

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Global energy demand is growing at an alarming and unsustainable rate, drawing mainly on the use of fossil fuels. These reserves are decreasing rapidly and becoming increasingly expensive. The associated emissions of greenhouse gases and other toxic pollutants are becoming environmentally unacceptable. Energy security has become a major issue as fossil fuels are confined to few areas in the world and their availability is controlled by political, economic, and ecological factors. A global coherent energy strategy that encompasses the entire energy life cycle is required in order to address all the forms of energy harvesting, storage, conversion, transmission, and distribution. Hybrid nanomaterial systems hold the key to fundamental advances in direct renewable energy and energy storage and conversion which are needed to enable renewable energy and meet the general energy challenges and associated environmental effects. This paper presents new approaches and methodologies used to design and develop carbon nanotube nanostructured hybrid nanomaterial systems incorporating structural and light-absorbing electron donor polymers, inorganic semiconductors, metallic and ceramic nanoparticles as energy harvesting and storage systems.

INTRODUCTION

The global use of energy is expected to increase by more than 50% from

2010 to 2025. The use of the available three main fossil fuels—oil, natural gas, and coal—may not always be readily available to meet the global energy demands. The known available

reserves of the three fossil fuels are expected to be depleted well within the next two centuries. In addition the associated fossil fuel emissions will not be environmentally acceptable and thus alternative sources of energy are needed. Nuclear energy offers interesting advantages but issues associated with nuclear waste disposal and safety, or perceived safety issues, limit the full development of its potential. The major challenges associated with the development and harvesting of fusion energy are not expected to be resolved effectively in the next two decades. This leaves us with few choices and among those the development of key renewable energy resources appear as appropriate responses to the current energy challenge, at least in the short term.

In order to fully develop the potential of all forms of renewable energy, current challenges in energy storage and conversion have to be met. Carbon nanotubes (CNTs) are near-perfect one-dimensional molecules that have generated great interest since they were discovered in 1991. They have amazing electrical, electrochemical, electronic, thermal, and mechanical properties, very high surface area, and a large electrochemical stability which makes them ideal electrode materials for renewable energy applications.¹⁻⁴ In addition they can be combined with other functional nanomaterials in order to form CNT nanostructured hybrid materials, such as catalysts for applications in fuel cells and inorganic semiconductors for generation of electron-hole pairs in solar cells. The thermal conductivity of unroped single-wall carbon nanotubes (SWNTs) have been calculated to be within the range of 2,000–6,000 W/mK, although the thermal conductivity of SWNT ropes is considerably

How would you...

...describe the overall significance of this paper?

Globally we are currently experiencing considerable challenges in energy and environment. In the short term we need to achieve higher energy efficiency and increase supplies from all renewable energy sources. In the long term we need to develop an effective H₂ based economy. Carbon nanotube hybrid nanomaterial systems can effectively answer these challenges.

...describe this work to a materials science and engineering professional with no experience in your technical specialty?

Carbon nanotubes have amazing properties and consequently they have the capability to be integrated as a subsystem in more complex hybrid material systems for applications in direct energy harvesting: wind and solar, and in energy storage: supercapacitors, batteries, and fuel cells. In addition they can be used in the production and storage of H₂.

...describe this work to a layperson?

Conventional materials have all sorts of defects associated with their tridimensional nature and these defects limit the values of their properties. Nanomaterials with one and zero dimensional nature can be developed without such defects and thus with properties that are many orders of magnitude better than their conventional counterparts. Each of these types of nanomaterials can then be developed as a subsystem and then be integrated in an overall hybrid system in order to develop a wide range of amazing properties.

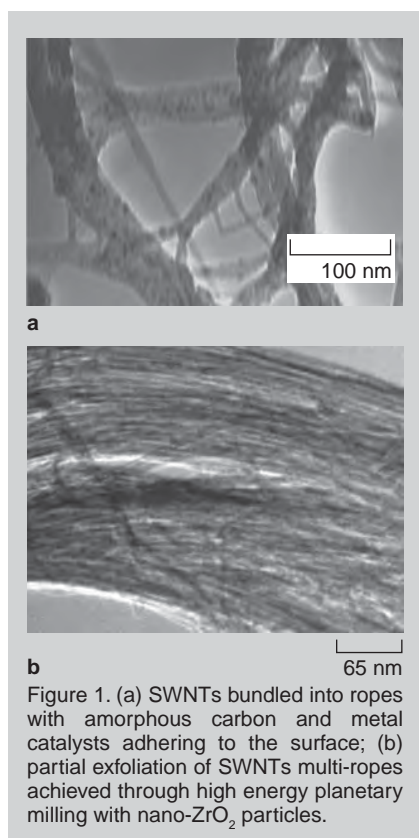


Figure 1. (a) SWNTs bundled into ropes with amorphous carbon and metal catalysts adhering to the surface; (b) partial exfoliation of SWNTs multi-ropes achieved through high energy planetary milling with nano-Zr₂O₂ particles.

lower and is observed to depend on the characteristics of the nanoropes.¹ The thermal conductivity of individual multiwall nanotubes (MWNTs) is within the range of 1,000–3,000 W/mK, and the thermal conductivity of double-wall nanotubes (DWNTs) is believed to be within the range of 1,000–4,000 W/mK.⁴

The electrical properties of carbon nanotubes vary from those typical of semiconducting to those typical of metallic conductors depending on the type and chirality of the CNT. The average electrical resistivity of carbon nanotubes is approximately 100 microhms, which is one order of magnitude lower than that of graphite.

The mechanical properties of carbon nanotubes are expected to equal or exceed the expected values of perfect materials. Since the C-C bond length in CNTs (0.142 nm) is shorter than that observed in diamond (0.152 nm), CNTs possess increased resistance against deformation. Theoretical predictions for SWNTs suggest the Young's modulus to be in the range 1.3–1.5 TPa and the strength to be approximately 200 GPa.^{1–4} For MWNTs the strength appears to be limited by the sliding of individual tubes in relation to each other

and thus the strength is expected to be significantly lower, while the Young's modulus is expected to be in the range of 1.0–1.1 TPa. This can be overcome by loading simultaneously all tubes at both ends of the MWNT assemblies.

Some of the major challenges that need to be overcome for the production of effective fully integrated carbon nanotube nanostructured hybrid functional nanomaterials are dispersion, functionalization, physical stability, integration into hybrid nanomaterials, and control of the tridimensional distribution or nano-architecture of the nanotubes and of the entire hybrid system.^{1–4} The focus of this research is the

development of carbon nanotube nanostructured hybrid materials incorporating additional functional nanoparticles, for renewable energy applications.

See the sidebar for a description of the nanomaterials used in this study.

PROCESSES, INTEGRATION, AND HYBRIDIZATION

Some of the major limitations in achieving the maximum active surface area of these nanomaterial systems are the dispersion of all types on CNTs, the inability to resolve the entanglements of multi-ropes of SWNTs into single ropes, and the inability to exfoliate the single ropes into single SWNTs. The

NANOMATERIALS AND PORE ARCHITECTURES

Nanomaterials Description

Typical nanomaterials used in this investigation have the following characteristics. The diameters were: single-walled nanotubes (SWNTs) unroped, ~1.4 nm in diameter; SWNTs (roped), 10–50 nm; multiwalled nanotubes (MWNTs), 10 to 50 nm, unroped; vapor grown carbon fibers, 30–200 nm and on average 100–150 nm. Lengths were more difficult to measure since it is hard to see ends: SWNTs: 0.3–10 s mm (before cutting), MWNTs: 1–100 mm. This means that MWNTs and SWNT ropes have three orders of magnitude more surface area and unroped SWNTs have four orders of magnitude more surface area than fibers. Thus MWNTs and roped SWNTs in a nanomaterial system may produce what is still considered a solid percolated network while the unroped SWNTs (and possibly DWNTs) have a chance to be fully integrated in the base material and thus induce conduction by other means.

With a nano-architecture consisting of surface treated individual SWNTs the typical active surface can be developed to be in the range of 6,000–7,000 m²/g. In addition SWNTs are molecules on a similar scale to nanoceramics, polymers, and fluid molecules which allow the development of several types of hybrid systems. This means that we have two distinct paths for engineering carbon nanotube nanostructured hybrid material systems: first, produce a mixture into which the nanotubes are added in order to change the strength, toughness, surface area, thermal, and electrical conductivity, via the rule of mixtures and, second, produce hybrid nanomaterials where the nanotubes are fully integrated into the matrix in order to mimic the properties of the carbon nanotubes into the final nano-hybrid system. The second approach leads often to the best performance with the highest degree of effectiveness and lowest carbon nanotube load. For this reason this is the preferred approach in this research and development work.

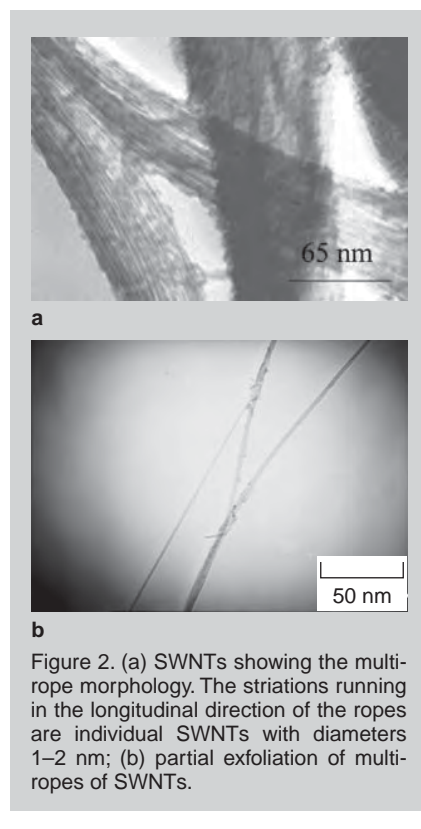
Pore Architectures

A unique advantage of carbon nanotube nanostructured hybrid systems is that they can be developed with extensive and effective pore architectures, tailored for the specific functional applications. The pore architectures in these systems can be classified as intrinsic and extrinsic. There are three types of intrinsic pore architectures. The first is intrinsic to the hollow tube cavities of SWNTs, DWNTs, and MWNTs. Their sizes can be computed taking into account the dimensions quantified in the previous section. This is another reason why CNTs need to be cut or decapped in order to activate all the pores and thus maximize the insertion of nanoparticles and ions inside the inner tubes. The second type comes from the interlayer spaces between the concentric and coaxial layers of DWNTs (typically 0.37 nm) and MWNTs (typically 0.34 nm). The third type is due to the interstitial pores associated with the bundling of CNTs in ropes, multiropes, and agglomerates, driven by van der Waals and electrostatic forces. The extrinsic pore structure comes from the nano-architecture or tridimensional packing and distribution of the CNTs in the hybrid system in association with the other nano system components. In other words the pore architectures need to be engineered and tailored for a specific functional application in order to harvest the full potential of the system.

magnitude of the entanglement of the multi-ropes is often magnified during purification of the produced SWNTs. One of the most promising methods toward the exfoliation of these ropes is through their cutting. This process can both contribute to the exfoliation of the ropes and help retard the process of re-agglomeration by shortening the nanotubes and contribute considerably to the increment of the active surface area of the system.

The SWNT ropes and the MWNTs were cut by physical and chemical processes, leaving them open on the ends for end cap functionalization and/or pore structure activation.

In addition chemical and physical processes were used to cut and exfoliate the multi-ropes by penetration with and intercalation of inorganic and organic nanomaterials between individual tubes in bundles. Exfoliation was achieved through multi and complex processes such as acid treatments used in conjunction with sonication and by high energy milling with nanoparticles. The sites at the end and along the nanotubes, once opened up, can be inhabited by groups such as $-\text{COOH}$ and inorganic nanoparticles until replaced with a final group used to re-functionalize the nanotubes, leading to a higher



original colloidal suspension ability in a particular matrix. These sites are also the first to be activated during the integration processes in specific matrices. Typical examples of the results of these procedures are shown in Figures 1 and 2.

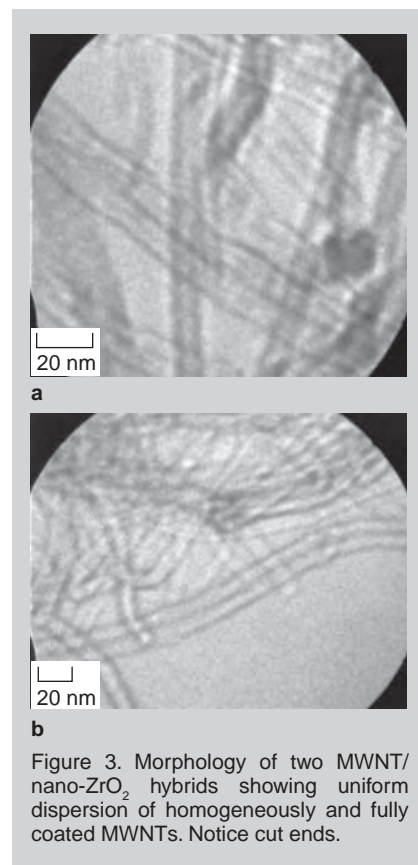
Hybrid nanomaterials incorporating SWNTs, DWNTs, and MWNTs and other inorganic nanomaterials were then developed using dispersion and integration techniques consisting of high energy planetary milling and hydrothermal reactions at elevated temperatures and pressures, followed by controlled densification. Some of these nano-matrices in the systems were ZrO_2 , SnO_2 , TiO_2 , MnO_2 , and LiO_2 . Typical results are shown in Figures 3 and 4.

The focus of this research is the full integration and hybridization of the unroped SWNTs, DWNTs, and MWNTs in these matrices and the control of the integrated nano-architecture, porosity and total surface area through advanced processing.

WIND ENERGY

The wind energy available on the Earth atmosphere is much larger than the current world power consumption. Potential on land and near shore exceeds 72 TW. This is equivalent to 54 million tons of oil per year, or over five times the total combined world power from all sources. Wind energy is clean and renewable without any form of emissions or toxic pollutants. It does not involve the depletion of any form of fuel. The growth in new capacity has exceeded 25% per year over the last five years. This is expected to continue or be exceeded for many years to come. This means that the wind energy industry is currently experiencing a very rapid development stage but it is far from reaching its mature stage. Currently it faces many important opportunities and challenges but its potential is extraordinary. The inconveniences of wind energy are few and they can easily be overcome.⁵

Some of the major challenges in wind energy are encountered in the blades and rotor subsystems. This is so because the wind power is proportional to the area of the blade, according to equation:

$$W_p = \frac{1}{2} \rho A C_p V^3$$


where W_p is the wind power, ρ is the density of air, A is the area perpendicular to the wind vector over which power is computed and C_p is the fraction of the power that might be captured by the wind turbine.

This means that in order to increase wind power, longer and wider blades are required, and thus the need for stronger (fatigue, tensile, and compressive strength), stiffer, and lighter materials with improved reliability and durability are also required. In order to harvest higher wind speed, turbines need to be installed off-shore. For that we need to reduce weight and use lighter materials. In addition, in order to function at low wind velocity and prevent damage at high wind velocity, blades need to be adaptable.

Carbon nanotubes can be introduced as critical enablers for new hybrid materials both as direct reinforcements and as reinforcements in a wide variety of the matrices consisting of vinyl esters, epoxies, and unsaturated polyesters. The focus of this research is the full integration of unroped SWNTs and MWNTs in these matrices using vacuum impregnation techniques (Figure 5) and other processes, followed by their

hybridization with multi-functional fibers, such as glass, basalt, carbon, and fabrics, for applications in wind turbine and other multifunctional applications. Typical results are shown in Figures 6–7. The advantages of these hybridizations are to confer strength, stiffness, toughness, and other CNT premium properties only where they are required, and thus enabling large blades to be manufactured at low cost. In addition other premium capabilities can be acquired such as fabrication into complex geometries, sensor capabilities for structural damage and failure and controlled radar cross section.

SOLAR ENERGY

Photovoltaic panels use solar cells packed in modules connected in multiple arrays to convert directly solar energy into electricity. In each module photons from the sunlight knock electrons into a higher state of energy and create electricity. Most devices are a type of a photodiode where the current comes from the transduced light energy. Until recently, photovoltaics were most commonly used in remote sites where there was no access to a commercial power grid or as a supplemental electricity source for individual homes and businesses. However recent advances in materials, technology, and manufacturing efficiency, combined with subsidies driven by environmental concerns, have dramatically accelerated the global deployment of solar panels. Installed capacity is growing by 40% per year led by increases in Germany, Japan, and the United States.

Even with all these advances solar

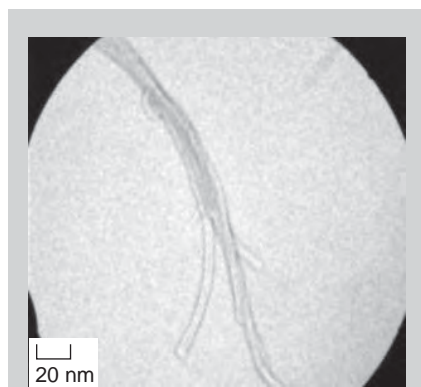


Figure 4. MWNT/Nano-ZrO₂ hybrid showing cut ends and intercalation of nano ZrO₂ particles between adjacent MWNTs.

electricity is still more expensive to produce than large-scale, mechanically generated power due primarily to the cost and efficiency of the panels. Low-efficiency silicon solar cells have been decreasing in cost though, and multi-junction cells with close to 30% conversion efficiency are now commercially available. Over 40% efficiency has been demonstrated in experimental laboratory systems. In recent years development efforts have been focused on increasing efficiency and decreasing the cost of the devices, or trading one for the other with overall benefits.

Carbon nanotube nanostructured materials with very high active surface area, high conductivity, high aspect ratio, and high electrochemical stability have the potential to increase the power conversion efficiency of solar cells by improving the excitation dissociation and charge carrier transport. In addition the capability of carbon nanotubes to receive and transfer electrons to an appropriate electron acceptor makes them ideal materials for applications in charge transfer processes.

Recently several approaches have been used to design photochemical solar cells incorporating assemblies of two or more nanostructured hybrid systems architectures. The use of CNTs to host light harvesting assemblies such as semiconductor particles provides effective mechanisms to capture photo-generated electrons and transport them to appropriate electron surfaces. In the CNT light-absorbing hybrid materials a direct charge transfer from the polymer to the CNTs has been observed.

Carbon nanotubes were used as effective photogeneration sites and charge carriers in collecting and transport layers.⁶ Carbon nanotube nanostructured hybrid nanomaterials incorporating TiO₂, CdSe, CdS, SnO₂, and Cu₂S have been used to increase the performance of solar cells.^{7–9} These hybrid systems were capable of generating photocurrents with higher efficiencies. Figure 8 shows some of the characteristics of the SWNT/CdS hybrid system.

In inorganic semiconductor hybrid systems free electron-hole pairs are formed. However in organic hybrid systems excitons or bound electron-hole pairs are formed. This drove the recent development of a new genera-

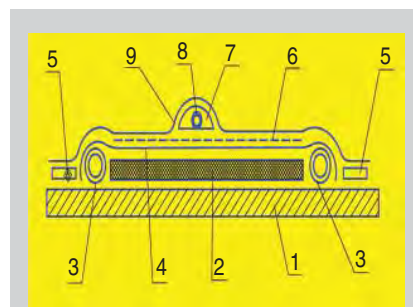


Figure 5. Vacuum hybridization process: 1–metal substrate, 2–stack of multifibers/fabrics, 3–vacuum tubes, 4–sucking glass fabric, 5–sealing cords, 6–polymer net, 7–connection mechanism, 8–polymer tube to feed reinforced resin, 9–polyimide vacuum film.

tion of devices called the third generation also known as organic photovoltaics. These devices incorporate CNT transparent electrodes and light-absorbing electron donor polymers and inorganic molecules trading inexpensive fabrication materials and processes for reduced efficiency. Several of these polymers have been used such as poly m-phenylenevinylene-co-diethylxy-p-phenylene-vinylene (i.e., PmPV) and poly-(3-hexylthiophene) (i.e., P3Ht) for short. The incorporation of CNTs in these hybrid systems provides an interface that drives away one of the charge carriers and develops a mechanism for collecting the charges at electrode surfaces. Double hybrid CNT systems incorporating both inorganic semiconductor nanoparticles and conjugated polymers have also been developed in order to increase the efficiency of charge harvesting and transfer.¹⁰

ENERGY STORAGE

Energy storage devices are characterized according to specific power (power density) and specific energy (energy density). Specific power represents the combination of current and voltage that can be supplied (wattage) and specific energy represents the longevity that the specific wattage can be supplied. There are four main types of energy storage devices. Classified by decreasing specific energy and increasing specific power they are: fuel cells, batteries, conventional electrochemical capacitors, and super capacitors (electrochemical double-layer capacitors).

Hybrid nanomaterial systems hold the key to fundamental advances in

energy storage and conversion which are needed in order to fully develop renewable energy and meet the general energy challenges and associated environmental effects.

SUPERCAPACITORS

The designation of supercapacitors comes from their very high specific energy compared with conventional capacitors. They are also called electrochemical double layer capacitors because the energy is stored in an electrical double layer formed at an interface between two polarizable electrodes and an electrolyte. There are three types of supercapacitors: electron conducting polymers, metal oxide, and carbon based. Supercapacitors based on carbon nanotubes nanostructure hybrid systems have the potential of high specific power, high specific energy, and longer life cycle. The potential for the application of carbon nanotubes (SWNTs, DWNTs, and MWNTs) in supercapacitors can be explained taking into account that the capacitance of a material is directly proportional to the surface area of the electrode, the permittivity of the free space, and the relative permittivity of the dielectric material and inversely proportional to the distance between the two electrodes, which in double layer capaci-



Figure 6. Nano-reinforced matrix resin consisting of a very stable colloidal suspension of SWNTs in Derakane 510 vinyl ester resin with controlled viscosity.

tors becomes the Helmholtz distance. Thus in order to maximize the capacitance a material must have the highest surface area and the smallest Helmholtz distance. This means the pore architecture and total active surface area of the CNT hybrid systems presented in the previous sections play a major role in achieving higher capacitance, as demonstrated by Arepalli and co-workers.¹¹

Yan and co-workers synthesized and characterized CNT/MnO₂ hybrid materials by reduction of potassium permanganate under microwave irradiation and showed that they were able to homogeneously coat the surface of the CNTs with MnO₂. They measured the capacitance of a CNT-15% MnO₂ composite with high cycle stability to be 85% of the theoretical capacitance, which they attributed to the high conductivity of the composite and the rapid transport of the electrolyte ions.¹² Hybrid SWNT/conjugated conducting polymer systems have also potential as candidate materials for supercapacitors.

BATTERIES

Among rechargeable batteries the Li ion technology has the greatest potential due to their combination of increased specific energy and specific power. One of the main challenges in the development of these batteries is to be able to sustain electrode integrity over a maximum number of discharge-recharge cycles. Most frequently the negative electrode has been made of graphite and the positive electrode incorporates a hybridization of lithium, cobalt, niobium and other oxides. These electrodes are separated by an electrolyte. During discharge the lithium ions stored in the negative electrode flow to the positive electrode where they generate an electric current by reacting with oxygen atoms. The process is reversed during recharging of the battery. The lifespan of these electrodes is limited by various factors such as lithium-alloying agglomeration and the growth of passivation layers which prevent the effective transfer of lithium ions. CNTs can be effectively used in both electrodes as a free-standing lithium ion storage and intercalation material and hybridization goals have been developed for both electrodes. For the nega-

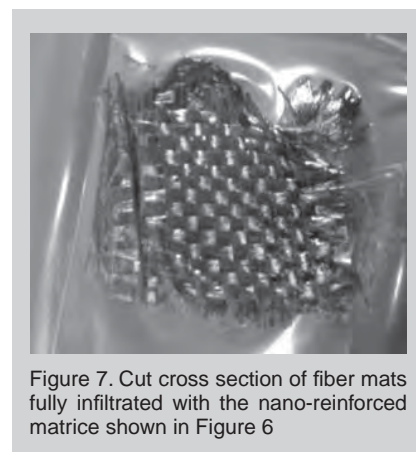


Figure 7. Cut cross section of fiber mats fully infiltrated with the nano-reinforced matrix shown in Figure 6

tive electrode the goals are to increase the total surface area by developing a highly porous material based on exfoliated and surface treated CNTs in order that they can store more lithium ions and thus increase the duration of the battery cycle. The goals for the positive electrode are to develop a highly porous hybridization of CNT with oxygen atoms attached to their surfaces and ready to bind with lithium.

Landi and co-workers developed CNT nanostructured hybrid anodes and showed that depending on processing factors their reversible lithium ion capability exceeded 1,000 mAhg⁻¹, equivalent to three times the capability of graphite anodes.¹³

Using the second approach Y. Shao-Horn at Massachusetts Institute of Technology showed that the CNT based batteries can have a specific energy five times higher than capacitors and deliver that power ten times faster than conventional lithium ion batteries. They used layer-by-layer assembly techniques to manufacture electrodes several micrometers thick consisting of additive-free, densely packed functionalized MWNTs. The electrodes could store lithium up to a reversible gravimetric capacity of ~200 mA h g⁻¹ and delivering 100 kW kg⁻¹ of power with lifetimes in excess of thousands of cycles.¹⁴

FUEL CELLS

Fuel cells are electrochemical devices that can convert directly the chemical energy of fuels such as methanol and hydrogen into electricity. They have high performance and are environmentally friendly since the byproducts of the overall reactions are usually water.

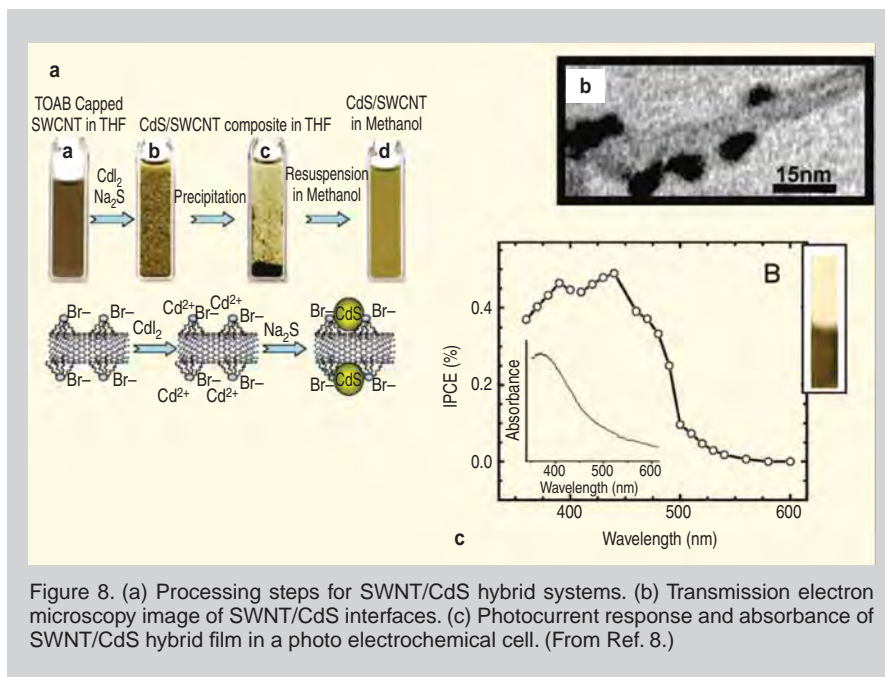


Figure 8. (a) Processing steps for SWNT/CdS hybrid systems. (b) Transmission electron microscopy image of SWNT/CdS interfaces. (c) Photocurrent response and absorbance of SWNT/CdS hybrid film in a photo electrochemical cell. (From Ref. 8.)

In addition they are reliable, have high efficiency and fuel adaptability, have no noise pollution, are vibration free, and can be deployed in remote locations.

One of the most critical issues in fuel cells is to simultaneously reduce the cost of electrodes and increase their catalytic activity. In the past carbon black has been used in proton exchange membrane fuel cells (PEMFCs) and direct methanol fuel cells (DMFCs). However since CNTs have better mechanical and thermal properties, much higher electronic conductivity and specific surface area, and a tubular structure they have the potential for development as hybrid nanomaterials containing fine controlled dispersions on nano-catalysts both on the inner and outer walls.

Carbon nanotube/platinum hybrid electrodes have been developed with more than one order of magnitude larger exchange current density than conventional carbon black/platinum electrodes even with lower active platinum load^{15,16} Hybrid CNT/Pt/Ru materials have been reported to have higher catalytic activity for PEMFCs and DMFCs due to the very high surface area and

the decrease of the overpotential for the appropriate reactions. In addition they exhibited a different catalytic behavior which could improve the mass transport within the layer of the catalysts and increase the mass activity.¹⁷ Other benefits of developing CNT hybrid materials are the formation of a larger number of smaller and more uniformly distributed nanocatalyst particles.¹⁸

CONCLUSIONS

Carbon nanotube nanostructured hybrid nanomaterial systems incorporating structural and light-absorbing electron donor polymers, inorganic semiconductors, and metallic and ceramic nanoparticles as energy harvesting and storage systems have been developed. Significant progress has been achieved in the design of their architectures, their preparation, and in the measurement of their performance. In designing and developing the appropriate system architecture controlled balances must be obtained in altering the original nanostructure and thus properties of the specific CNTs in order to prepare anchoring sites for the integration with the other nanosystem components. Although there are still many challenges

ahead in these areas it is expected that these systems will play a major role in harvesting the full potential of renewable energy resources and associated energy storage needs. We expect to see in the future the use of a system approach to the design, development, and manufacture of more complex and sophisticated functional nanosystems, and as a consequence we expect to see significant breakthroughs toward the resolution of the global energy challenge.

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Fernand D.S. Marquis is with the Wayne Meyer Institute of Systems Engineering, Naval Postgraduate School, Monterey, CA 93943; e-mail: fdmarqui@nps.edu.

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