Carbon Nanotubes and Microwaves: Interactions, Responses, and Applications

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Since their discovery, carbon nanotubes (CNTs) have generated great expectations for many diverse potential applications. In particular, the response of CNTs to electromagnetic radiation has been the subject of many recent investigations. Some basic studies have focused on understanding the nature of the interactions between radiation and CNTs, while the observed behavior has been used for many exciting purposes. For instance, photoinduced charge separation in CNTs has been studied for designing transistors and photovoltaic devices.[1] Hyperthermia can be produced as a consequence of radio frequency irradiation of cells cultured with CNTs and has been applied toward cancer therapy.[2]

Microwaves and Carbon Nanotubes. Among the many possible frequencies that can be employed, microwaves represent one of the more interesting possibilities. In the electromagnetic spectrum, microwave radiation is located between infrared and radio waves. When CNTs are exposed to microwaves, strong absorptions are observed,[3] producing intense heating, outgassing, and light emission. Although the mechanism of CNT−microwave interaction remains incompletely understood, microwaves have been used for the purification of raw CNTs for their chemical functionalization or to provide a reactive environment to promote the modification of other products in the presence of CNTs. Applications not only include chemical modification but also technological developments, such as CNT-forest lighting systems. In this Perspective, we attempt to summarize the latest developments in the use of microwaves with CNTs, mainly focusing on potential applications of these innovative methodologies.

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Mechanism of the Carbon Nanotube−Microwave Interaction. Any commercial sample of CNTs contains several different impurities such as amorphous carbon and metallic nanoparticles; thus, there are different factors to consider in order to explain the strong microwave absorptions observed. Microwave irradiation may cause heating by two main mechanisms: (1) dipolar polarization and (2) conduction (Joule heating). The presence of impurities that are electrically conductive, such as metals or carbon, could support a mechanism based on conduction heating. According to this mechanism, the microwaves do not heat the material immediately; rather, motion of the electrons is induced by the electric field, causing sample heating. In this way, Joule heating induced by the metal catalyst particles should generate localized superheating.[4] However, other studies indicate that nanometer-sized magnetic particles should be impacted minimally by microwave irradiation,[5] while the removal of catalyst has little effect on the microwave absorption of CNTs.[6]

The behavior of amorphous carbon and graphite impurities under microwave irradiation is less controversial; the extended π-system permits conductivity (and thus Joule heating) to enable localized heating.[7] However, another potential source of localized superheating should be considered, namely, the generation of gas plasma from absorbed gases (particularly H_2) in CNTs under microwave radiation, which has been reported in some cases.[3]

ABSTRACT The interaction of microwaves with carbon nanotubes (CNTs) is an interesting topic for a variety of potential applications. Microwaves have been used for the purification of CNTs and for their chemical functionalization, providing a technique for simple, green, and large-scale protocols. In addition, the selective destruction of metallic CNTs under microwave irradiation could potentially result in a batch of semiconducting-only nanotubes. As an innovative application, the combination of microwaves with well-aligned CNTs could produce a new illumination technology. Moreover, the microwave absorbing properties of CNTs and their different behavior from typical organic compounds may open the door to the preparation of a wide range of new materials useful in many fields. A few examples of practical applications include electromagnetic interference for protecting the environment from radiation and microwave hyperthermia for cancer treatment as well as other medical therapies requiring precise heating of biological tissues.

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Published online December 22, 2009.
10.1021/nn901604j

The dipolar polarization seems to have little contribution to CNT microwave absorptions, as no electric dipoles exist in nanotubes. On the other hand, a "perfect" CNT, due to its unique one-dimensional (1D) structure, has been shown to be a ballistic conductor,[8] meaning that its resistance is quantized and is independent of length and that no energy is dissipated due to electron movement. Consequently, the current induced during microwave irradiation is not converted to heat. However, as-synthesized CNTs usually have structural imperfections, which result in the decay of ballistic transport, allowing Joule heating to occur in CNTs and resulting in superheating. A simple model to explain microwave-induced heating of CNTs has been proposed, through transformation of electromagnetic energy into mechanical vibrations.[9] Within this model, CNTs subjected to microwave radiation undergo ultraheating due to a transverse parametric resonance, which arises from the polarization of CNTs in the microwave field. For nonpurified CNTs, Joule heating occurs because imperfections and impurities damp the transverse vibration mode. The same authors claim that, in a dense and viscous local environment, CNTs will not vibrate. Therefore, there is neither transverse resonance nor Joule effect and thus no heating, which would explain the different results of absorption, in the presence of solvents or under dry conditions, observed by other groups.

Purification of Carbon Nanotubes (CNTs) Using Microwaves. Although the mechanism behind CNT−microwave interactions remains unclear, there has been a wide array of potential applications involving microwave radiation with CNTs, including purification processes. CNTs are usually generated by arc discharge, laser ablation, or catalytic gas-phase growth, starting with carbon monoxide or other carbon sources. The raw material contains CNTs contaminated with different amounts of amorphous carbon and/or catalytic metal particles. One of the main problems in practical applications is that impurities are always present in the as-synthesized products, even for CNTs produced on a large scale. While there have been many CNT purification techniques published in the literature,[10] which include acid reflux, oxidation, and filtration, most of them involve long processing times or multiple stages, the use of large acid volumes, and, more importantly, may lead to damaged CNTs.

One of the first demonstrations of microwave-assisted single-walled carbon nanotube (SWNT) purification was published by our group in 2002, based on the treatment of raw nanotubes in a microwave domestic oven, under air and with no solvent, in which the selective burning of metal particles led to a strong depletion of the iron content in the soot.[11] A similar technique was implemented by Harutyunyan *et al.*,[12] while subsequent research has demonstrated that microwave-assisted purification of SWNTs is a promising technique for large-scale purification, inflicting minimal damage to the SWNTs and reducing significantly the processing times and the use of harmful reactants.[13−18] Moreover, the reported efficiency of SWNT heating under microwave radiation could enhance the quality of SWNTs. Mackenzie *et al.*[18] have suggested an interesting method to improve the crystallinity of large volumes of SWNTs in a quick and inexpensive annealing protocol that would be efficient on a large scale. In this approach, under microwave radiation, the defective and damaged SWNTs may be supplied with sufficient energy to reorient any "defective" sp^3 carbon bonds into sp^2 hybridization.

Another exciting possibility is the selective destruction of one type of nanotubes based on microwave treatment. As-synthesized SWNTs are usually produced as a mixture of both metallic and semiconducting tubes; however, for many applications, the employment of "only" semiconducting tubes is essential. Some authors have proposed a straightforward technique for the preferential destruction of metallic SWNTs using microwaves, based on the more efficient microwave absorption of this type of tubes.[19,20] This possibility is attractive, and the experimental results confirm the preferential conversion of metallic nanotubes, but this destruction is only partial. The degradation rate varies, depending on the diameters of the metallic tubes, and problems related to the microwave absorption of metallic catalytic residues and the progressive destruction of semiconducting tubes still need to be addressed, though, recently, Akasaka and coworkers have combined microwaves with acid dispersions improving this approach.[21]

Microwave-Assisted Functionalization of CNTs. Although a great deal of work has been carried out enabling methodologies for functionalization of carbon nanostructures, we are still far from facile integration of CNTs into organic, inorganic, or biological systems. Here, the challenge is to achieve sufficient functionalization of the CNT surface to ensure easy processing, while avoiding significant degradation of the structure which could compromise the main properties of the material. Nevertheless, most of the described protocols require long periods of time and use aggressive treatments that cause shortening and damage of the CNTs.[22,23]

Microwave-assisted organic synthesis (MAOS) is an enabling technology that has been extensively used in organic synthesis.[24−27] Usually, the use of microwaves facilitates and accelerates reactions, often improving the relative yields. Taking advantage of the strong microwave absorption displayed by CNTs, recent work has explored the use of this radiation to enable CNT functionalization.[28] Different approaches have been described toward covalent attachment of vari-

ous organic groups or chemical modification of these nanostructures. For example, a 3 min microwave treatment of a suspension of SWNTs in a mixture of nitric acid and sulfuric acid was sufficient to produce carboxylated and sulfonated SWNTs with relatively high dispersivity in water and ethanol.[29] Amidation or esterification of the carboxyl groups were also pursued under microwaves.[30−32] In a different procedure, MWNTs under microwaves have also been functionalized with carboxyl, carbonyl, hydroxyl, and allyl terminal groups, without the use of aggressive oxidants. The technique is also useful to derivatize CNTs with Au nanoparticles synthesized *in situ* by metal-ion reduction during functionalization.[33] Microwave treatment of oxidized MWNTs has also proven to enhance the loading of Pt nanoparticles.[34] On the other hand, microwave activation has been used to enable cycloadditions in prefunctionalized CNTs[35,36] or in pristine CNTs using ionic liquids,[37] to perform electrophilic additions using alkyl halides and alcohols,[38−40] to carry out radical additions,[41] to apply the Bingel reaction to side wall functionalization of CNTs,[42] and to assist bromination of double-walled nanotubes.[43]

In all cases, microwave irradiation of CNTs reduces the reaction times and gives rise to products with higher degrees of functionalization than those obtained by conventional thermal methods.

In all cases, microwave irradiation of CNTs reduces the reaction times and gives rise to products with higher degrees of functionalization than those obtained by conventional thermal methods. Interestingly, some authors[41] suggest a competitive effect of microwave irradiation that both promotes functionalization and removes some functional groups that are initially present. This observation supports the idea of microwave-assisted annealing commented above, with microwaves submitting enough energy to transform sp^3 carbon to sp^2 hybridization.

The reaction conditions applied under microwaves have been similar to the ones used with classical heating. Our groups have described a solvent-free technique combined with microwave irradiation,[44] which provides a fundamentally different approach when considering the chemistry of CNTs. In the absence of solvents, CNTs absorb the radiation directly and it is possible to take full advantage of the strong microwave absorption typical of pristine nanotubes. Moreover, solvent-free conditions pave the way to green protocols and large-scale functionalization. The methodology has been applied to produce multifunctionalized carbon nanotubes using a combination of two different addition reactions: the 1,3-dipolar cycloaddition of azomethine ylides and the addition of diazonium salts, both *via* a simple, fast, and environmentally friendly method (Figure 1).[45] The reactions can be followed by Raman spectros-

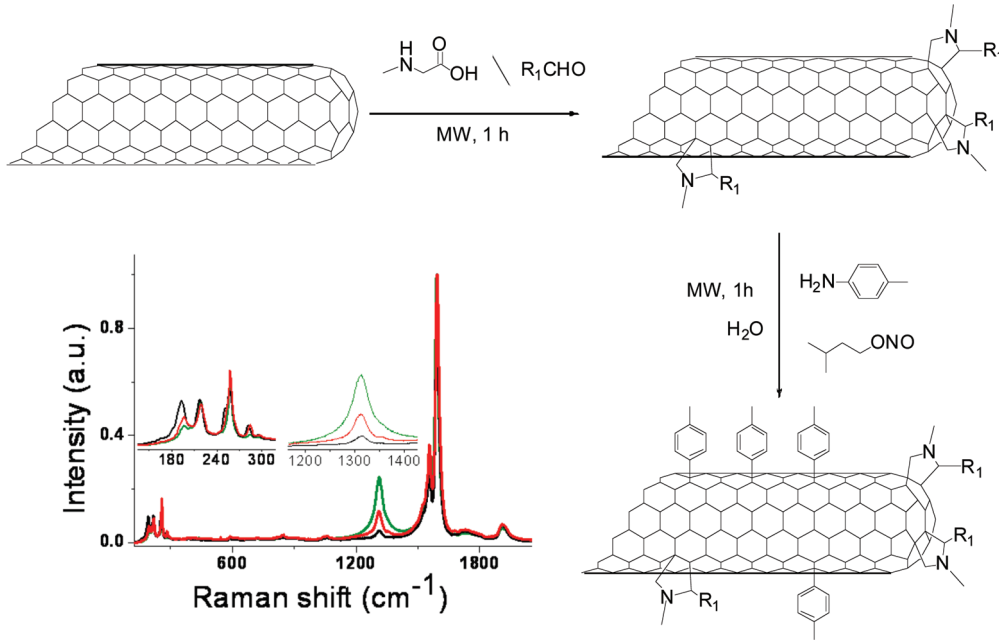

Figure 1. Microwave-assisted double functionalization of SWNTs. Raman spectra (λ_{exc} 633 nm) of pristine SWNTs (black line), SWNTs functionalized by 1,3-dipolar cycloaddition (red line), and doubly functionalized SWNTs (green line).

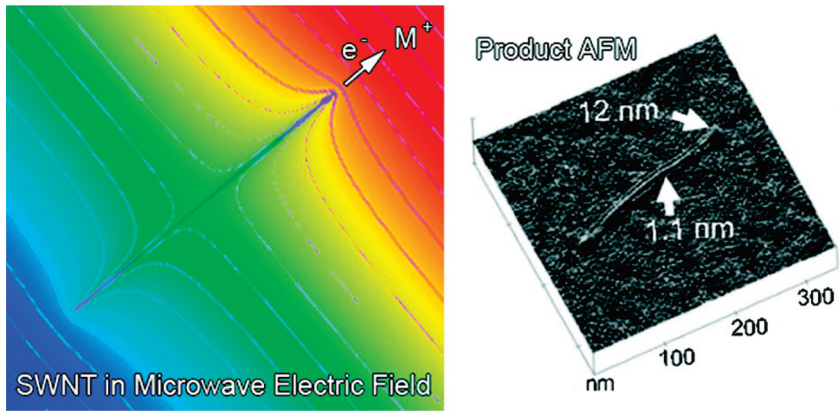

Figure 2. (a) Tip-selective reduction of metal salts onto SWNTs by microwaves. (b) Representative atomic force microscope image of tip-selective reduction of Au metal salts onto SWNTs by microwave reduction. Reproduced from ref 58. Copyright 2008 American Chemical Society.

copy, which shows the increase of the D-band (~1300 cm^{-1}, sp^3 carbons) at the expense of the G-band (~1600 cm^{-1}, sp^2 carbons). As a final remark, the extremely high temperatures observed in the absence of solvents might permit the functionalization of CNTs using new types of reactions that do not work under classical reflux heating.

Using a similar approach, a solvent-free microwave-assisted methodology has also been employed for the covalent functionalization of carbon nanohorns (CNHs) based on the Bingel reaction[46] and the 1,3-dipolar cycloaddition of azomethine ylides.[47] CNHs are similar in structure to star-aggregated SWNTs, but they are produced without the use of any metal catalyst, such that they are completely metal-free. That makes these materials very attractive, not only because of the easy functionalization process, which could open the door to new applications for CNHs, but also because it could help to understand the behavior of CNTs under microwaves.

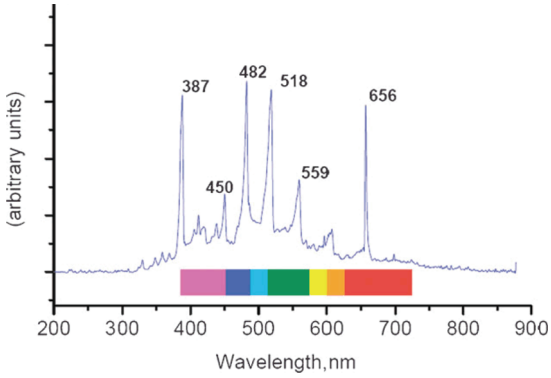

Figure 3. Typical emission spectrum of CNT lamp ignited by microwave irradiation, showing intrinsic polychromatic emission wavelengths involving significant red, green, and blue wavelengths. Reproduced with permission from ref 59. Copyright 2009 Royal Society of Chemistry.

From Telecommunications to Biomedicine. In recent years, a significant amount of research has focused successfully on electromagnetic interference (EMI) shielding or microwave absorption for the purpose of protecting the workspace and the environment from the radiation emitted by telecommunication systems.[48] Recently, it has been suggested that the microwave absorbing properties of CNTs make these structures potential candidates for the preparation of a wide range of EMI shielding materials, involving CNT−polymer[49,50] and CNT−ceramic composites.[51−54]

Moreover, CNTs can be used to provide a reactive environment in the presence of microwave irradiation. They absorb the energy that leads to fast and direct heating and thus can be used to rapidly cure ceramic composites[55] for the microwave bonding of plastics[56] and in the formation of strong CNT−polymer bonds.[57] In addition, it has been shown recently that, when subjected to microwave fields, highly dispersed SWNT−surfactant suspensions generate polarization potentials at their extremities that drive electrochemical reactions.[58] In the presence of transition metal salts, SWNTs drive reductive condensation to metallic nanoparticles, which deposit regioselectively at the SWNT tips, producing novel structures (Figure 2). This interesting electron-transfer process under microwave irradiation could open the way to new applications of CNTs in different fields such as chemical synthesis or nanomedicine.

The distinct behavior of CNTs when subjected to different microwave fields could be used to produce noninvasive and selective thermal cytotoxicity.

Recently, some authors have shown that under microwave irradiation well-aligned CNTs exhibit a bright white-light emission with narrow-band RGB (red, green, blue) colors (Figure 3).[59] This could lead to a new low-cost illumination technology with bulbs composed of CNT forests containing no mercury and having no electrode to wear out, with the corresponding saving in energy.

In the field of biomedical applications, the combination of microwaves and CNTs has not been exploited to full potential. The distinct behavior of CNTs when subjected to different microwave fields could be used to produce noninvasive and selective thermal cytotoxicity. The microwave treatment of malignant tumors should be enhanced by introducing CNTs inside malignant cells, which should convert microwave energy into lethal heat, faster and more efficiently than in healthy cells, serving directly as an antican-

cer therapeutic agent. Furthermore, the microwave power has also been shown to have great therapeutic benefits in medical treatments requiring precise heating of biological tissue, for example, in microwave cardiac ablation. CNTs could also help to improve this technique, permitting heating in other biological tissues with lower conductivity.

CONCLUSIONS AND OUTLOOK

The use of microwaves for the activation of CNTs has not been fully explored. While there is still a need to investigate the mechanism of CNT−microwave interactions, the strong absorptions observed suggest a broad spectrum of applications of which we are only beginning to catch a glimpse. So far, microwaves have been used for the purification of raw CNTs and for their chemical functionalization, providing a technique for simple, green, and large-scale protocols, but this is only a first step. By controlling the preferential microwave absorption of different types of tubes, it is possible to obtain samples consisting primarily of semiconducting nanotubes, which is critical for many applications. In addition, the extremely high temperatures observed, in the absence of solvents, might activate the reactivity of CNTs, permitting their modification in new kinds of reactions that do not work under classical reflux heating. In the presence of microwave irradiation, CNTs can also be used to provide a reactive environment to promote the modification of other products, for example, for driving electrochemical reactions. Moreover, the awareness of the particular microwave absorbing properties of CNTs and their different behavior with respect to typical organic compounds should open the door to the preparation of a wide range of new materials useful in assorted fields, including telecommunications, biomedical applications, and illumination technologies. From the examples discussed here, it is clear that the potential related to the interactions of CNTs with microwaves is worth further exploration, with many possible applications not only in basic research but also in everyday life.

Acknowledgment. Part of the work described in this Perspective has been financed by DGICYT of Spain through project CTQ2007-60037/BQU and by Consejería de Educación y Ciencia through projects PBI-06-0020 and PCI08-0040, the University of Trieste and MUR (PRIN 2006, prot. 2006034372 and Firb RBIN04HC3S).

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