

Photoemission and absorption spectroscopy of carbon nanotube interfacial interaction

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ARTICLE INFO

Available online 20 August 2008

Keywords:

Surface

Interface

Thin film

X-ray photoemission spectroscopy (XPS)

Extended X-ray absorption fine structure (EXAFS)

Near edge X-ray absorption fine structure (NEXAFS)

Single walled carbon nanotube (SWCNT)

Double walled carbon nanotube (DWCNT)

Multi walled carbon nanotube (MWCNT)

Chemical vapour deposition (CVD)

ABSTRACT

Element-specific techniques including near edge X-ray absorption fine structure, extended X-ray absorption fine structure and X-ray photoemission spectroscopy for the characterization of the carbon nanotube interfacial interactions are reviewed. These techniques involve soft and hard X-rays from the laboratory-based and synchrotron radiation facilities. The results provided information of how the nano-particles of catalysts are involved in the initial stage of nanotube growth, the nanotube chemical properties after purification, functionalization, doping and composite formation.

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1. Introduction

Carbon nanotubes consist of coiled graphene sheets. The nanotubes can be single walled (SWCNT), double walled (DWCNT) or multi walled (MWCNT) depending on the growth conditions [1]. In principle the walls of double or multi walled nanotubes may contain rows of arm-chair, zig-zag or chiral structures but usually mixtures of them. The outer surface wall properties may or may not be influenced by the underlying walls

since there will be interactions between the surfaces. These surfaces may also contain defects due to the formation of pairs of five–seven membered rings or addition of sp^3 bonds. The ends of nanotubes may be open or closed but are more commonly closed. Five membered rings must be present as in a fullerene structure for enclosure. It is possible that some defects contain seven membered rings or even dangling bonds, stabilised by delocalisation of electrons about the whole structure.

Non-covalent forces including π – π and van der Waals interactions between the surfaces result in the nanotube aggregation into bundles. The successful separation of the bundles into individual nanotubes controls the chemical–physical properties of materials formed from carbon nanotubes and other materials [2,3]. Hence it is not surprising

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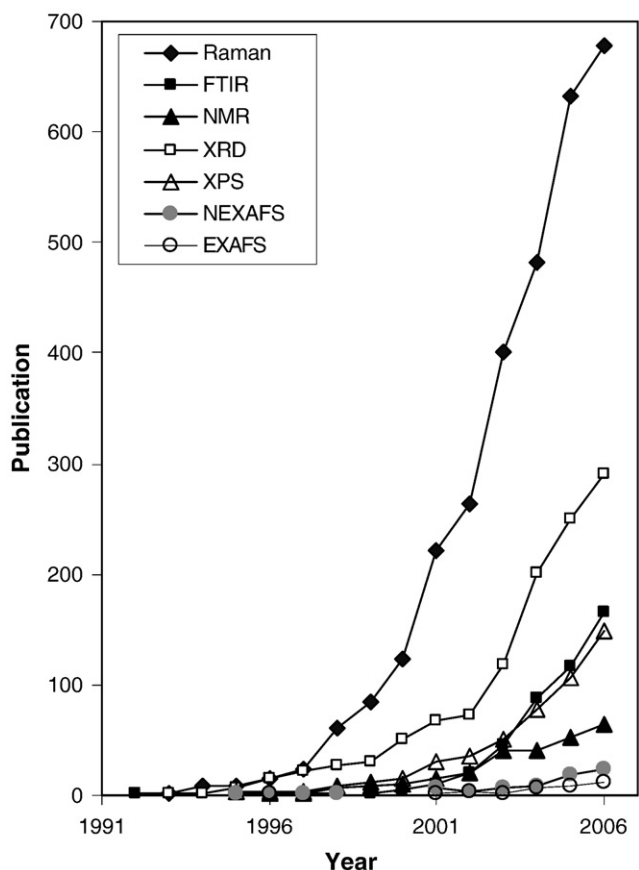


Fig. 1. Comparison of some characterization techniques reported in a number of research publications and patents of carbon nanotubes (from SciFinder Scholar 2006).

that there has been a rapid growth in use of techniques capable of characterising these surface forces, particularly Raman, Fourier transform infrared spectroscopy, Nuclear magnetic resonance and X-ray methods (Fig. 1) [4]. In this review we summarise the work by X-ray techniques. Elsewhere we will review other techniques and their applicability.

The techniques such as X-ray diffraction is normally used for the crystalline materials or materials with well-defined long-range orders and will not be further discussed here. However, it is useful for studying the short-range structures and chemistry of the substances using the element-specific techniques including X-ray photoemission spectroscopy and X-ray absorption spectroscopy. In these techniques, the energy of photoelectrons generated from the surface (and sub-surface) after interactions with X-rays can be analysed.

In general, X-ray photoemission spectroscopy (XPS) involves excitation of photoelectrons by X-rays to vacuum and the photoelectron kinetic energy is analysed, which results in information concerning chemical bindings [5]. Near edge X-ray absorption fine structure spectroscopy (NEXAFS) involves transition of electrons to the empty molecular orbitals (e.g., anti-bonding π^* or σ^* orbitals) and the electron relaxation process results in the emission of electrons or fluorescent signals [6]. The electron and fluorescence spectra provide information concerning local environments and oxidation states. Extended X-ray absorption fine structure spectroscopy (EXAFS) involves scattering of electrons by neighbouring atoms and provides quantitative information including co-ordination numbers and atomic distances [6].

XPS involves soft X-rays (energy usually lower than 3000eV) while NEXAFS and EXAFS involve soft or hard X-rays with the energy being up to several thousands eV. Synchrotron radiation, from which X-rays are generated by the electrons being accelerated to nearly the speed of light, allows the energy to be tuned into soft or hard X-ray regions. In

many synchrotron facilities it is possible to combine XPS, NEXAFS and EXAFS measurements together, so that it is evident that the same surface area is being monitored with the techniques hence creating greater reliability in interpretation.

There have been reviews of the interactions of soft X-rays with carbon nanotubes [7–9]. However a general discussion of surface properties in the light of information from other X-ray techniques has not been forthcoming. This review aims at covering both the soft and hard X-ray studies of the carbon nanotube interfacial reactions and integrates the results to demonstrate the properties of these surfaces, which are essential for understanding of material formation with carbon nanotubes.

2. Study of carbon nanotubes as single materials

Imamura et al. were amongst the first to report the soft X-ray NEXAFS study of carbon nanotubes [10,11]. In particular, a peak at 285.4eV of the C K-edge NEXAFS spectra of the MWCNTs produced by arc-discharge evaporation was attributed to the transitions of the 1s electrons to the anti-bonding π^* orbitals. Peaks above 290eV including those at 291.9, 292.9 and 297.5eV were due to the transitions of the 1s electrons to the anti-bonding σ^* orbitals. The 1s- π^* and 1s- σ^* peaks reflect the sp^2 and sp^3 -carbon bonds present, respectively. Additional peaks at 287.7 and 288.7eV were attributed to the interlayer interactions similar to those in graphite [12]. However, Tang et al. have performed the C K-edge NEXAFS of the nanotubes grown via annealing of carbon nanowires and suggested resonances in this region were due to the 1s- σ^* transitions of the other sp^3 , C-H bonds [13], clearly defects due to carbon bond impurities in the nanotubes.

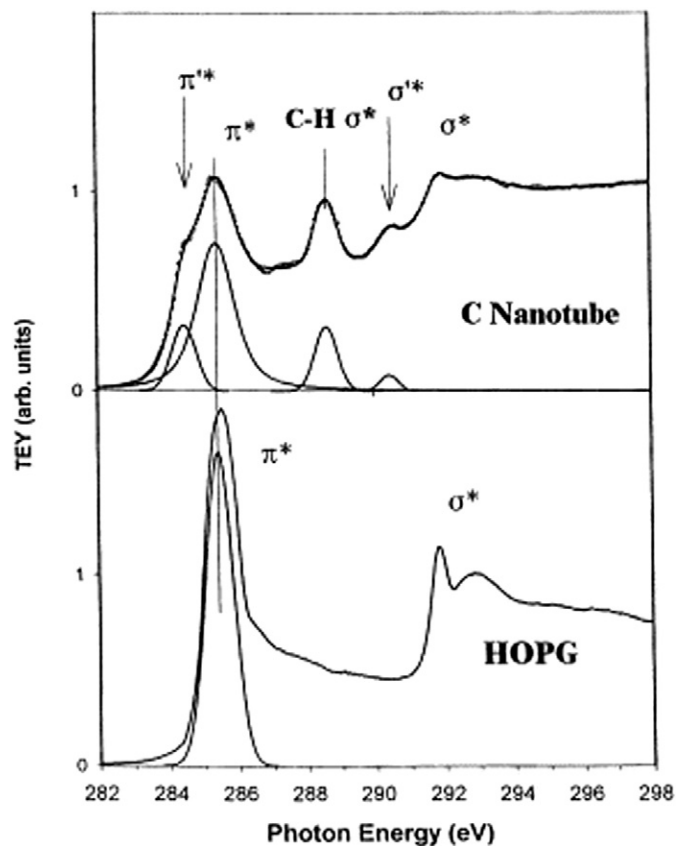


Fig. 2. Curve-fitted C K-edge NEXAFS of carbon nanotubes and highly oriented pyrolytic graphite. The shoulders of the π^* and σ^* peaks in the nanotube spectrum, not recorded in the graphite reference, are denoted as $\pi^{*'}$ and $\sigma^{*'}$ (Tang et al., Chem. Phys. Lett. 2002, 366, 636–641).

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