Journal of Alloys and Compounds 643 (2015) S165–S171

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: www.elsevier.com/locate/jalcom

Interferometric laser measurement of multi-axial mechanical properties exhibited by carbon nanotubes

E. Feria-Reyes [a], C. Torres-Torres [a,*], H. Martínez-Gutiérrez [b], C. Mercado-Zúñiga [c], R. Torres-Martínez [d], G. Urriolagoitia-Calderón [a]

[a] Sección de Estudios de Posgrado e Investigación, ESIME ZAC, Instituto Politécnico Nacional, México, DF 07738, Mexico
[b] Centro de Nanociencias y Micro y Nanotecnologías del Instituto Politécnico Nacional, México, DF 07738, Mexico
[c] Subdirección C, Tecnológico de Estudios Superiores de Coacalco, Coacalco de Berriozábal, Edo. México 55700, Mexico
[d] Centro de Investigación en Ciencia Aplicada y Tecnología Avanzada Unidad Querétaro, Instituto Politécnico Nacional, Santiago de Querétaro, Querétaro 76090, Mexico

ARTICLE INFO

Article history:
Available online 26 March 2015

Keywords:
Mechano-optics
Nonlinear optics
Interferometry
Carbon nanotubes

ABSTRACT

Interferometric optical experiments for measuring changes in fringe irradiance patterns dependent on optical and mechanical responses associated to carbon nanotubes are reported. Thin solid films conformed by multiwall carbon nanotubes were prepared by an aerosol pyrolysis method. A Michelson optical interferometer with a highly stable 488 nm wavelength was employed for evaluating the studied sample. According to the interferometric results, the strain of the carbon nanotubes presented a nonlinear behavior resulting from applied multiaxial forces. On the other hand, for further investigating the effect of the variation of the mechanical properties and the optical characteristics of the tubes, nonlinear optical experiments were performed when the sample was exposed to a mechanical action. The third order nonlinear optical response was explored with the assistance of the second harmonic provided by a Nd:YAG laser system in a two-wave mixing configuration. Potential applications for mechano-optical modulation by nanosystems based on carbon nanotubes are contemplated.

1. Introduction

Nanostructuration of carbon materials is probably one the most fascinating process for developing superlative characteristics in mechanical experiments. Graphene, graphyne and their families present two-dimensional features that can be contemplated for outstanding optical and mechanical applications [1,2]. Besides, carbon nanotubes (CNTs) can be considered as the strongest low-dimensional fiber materials.

The mechanical behavior of carbon nanostructures depends on their morphology and purity; as a consequence, the processing route employed for their preparation generates important differences in their mechanical features [3,4]. It has been proved that isolated and grouped CNTs can be related to quite similar empirical force–deformation relations [5]. On the contrast, optical properties attributed to different CNTs frequently show remarkable distinctions [6]; and sometimes they are able to display opposite effects as it is the case of nonlinear optical absorption phenomena associated to decoration process [7].

In order to enhance optical and mechanical properties of CNTs diverse preparation methods have been proposed [8–11]. These preparation techniques provide particular optical, mechanical, and electrical properties that are related to potential applications [12–15]. Furthermore, zigzag-shaped CNTs [16], toroidal CNTs [17], coiled CNTs [18], or kinked CNTs [19] are alternative configurations that produce notable changes in their comparative optical and mechanical effects too.

In this regards, it has been pointed out that the alignment of CNTs originates superior mechanical properties in specific experimental geometries [20]; but this is precisely an indication that for instrumentation of mechanical signals, a sensitive measurement requires multiaxial explorations that ought to be considered to evaluate the effective mechanical response of mechanical sensors based on CNTs.

Different multiaxial experiments have been carried out to distinguish the tensorial mechanical properties of CNTs [21]. Considering the vectorial nature of light, it seems to be attractive to study the potential applications of the nonlinear optical effects exhibited by CNTs under mechano-optical actions. In this regards, we prepared a sample with multi-wall CNTs (MWCNTs) in a thin

* Corresponding author at: Sección de Estudios de Posgrado e Investigación, ESIME, Zacatenco, Instituto Politécnico Nacional, México, DF 07738, Mexico. Tel.: +52 (55)57 29 60 00x54686; fax: +52 (55)57 29 60 00x54587.
E-mail addresses: ctorrest@ipn.mx, crstorres@yahoo.com.mx (C. Torres-Torres).

http://dx.doi.org/10.1016/j.jallcom.2014.12.278
0925-8388/

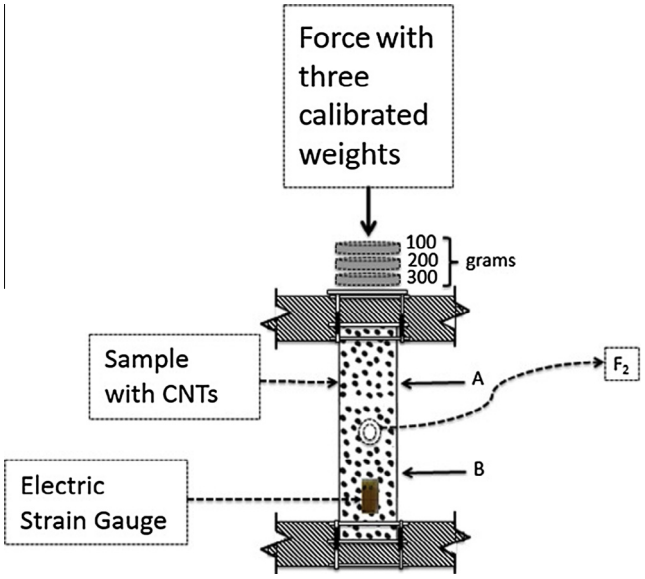

Fig. 1. Diagram of the mechanical experiment in the sample.

film form. Interferometric laser measurements were performed for identifying changes in the refractive index of the samples induced by multiaxial mechanical loads. A two-wave mixing (TWM) experiment allowed us to modulate the transmittance of non-resonant nanosecond optical pulses by considering the vectorial response exhibited by the tubes.

2. Experiment

2.1. Sample preparation

Thin films of MWCNTs were prepared following an aerosol pyrolysis processing route [22]. A solution containing hydrocarbon was employed as a solvent and an organometallic precursor as solute in the aerosol generated ultrasonically. The aerosol was carried by an argon flow (2.5 l/min) to be finally directed into a quartz tube located inside of a furnace operating at 800 °C during 30 min. Then, Ar flow was decreased to 0.3 l/min. until the stabilization of the furnace at room temperature. The samples were obtained using 0.5 wt% ferrocene and 2.5 wt% ethanol. Finally, the resulting MWCNTs were deposited as a thin film of about 50 μm width on a SiO_2 substrate. The studied thin film sample was obtained by a drip coating method with MWCNTs (1 mg) dispersed in an ethanol suspension (5 ml) on SiO_2 substrate. Transmission Electronic Microscopy (TEM) studies were carried out using a JEOL Transmission Electron Microscope JEM 2100 at an acceleration voltage of 200 kV equipped with an ultrascan CCD camera Model 994 TEM CCD. Besides, in order to corroborate the multiwall nature of the tubes, the samples were analyzed without previous purification in a micro-Raman Horiba Jobin Yvon LabRam HR with a He–Ne laser emitting at 632.8 nm wavelength.

2.2. Extensiometric measurements

For the preparation of the extensiometric measurements, uniformly degreasing the SiO_2 surface of the sample is the first steep that we made to remove oils, greases, organic contaminants, and other chemical residues. Surface must be abraded to remove any loosely bonded adherents and to develop a surface texture suitable for adherence. The desired location and orientation of the strain gage on test surface should be marked with a pair of crossed perpendicular reference lines. To provide optimum alkalinity for micro-measurements strain gage adhesives, the cleaned surfaces must be neutralized. After locating of the electric strain gage we prepare the MWCNTs thin film. We let the film in a drying process during 12 h. Once the electric strain gage was glued on the surface and the MWCNTs were over the cleaned surface, we performed the experiments.

Initially, we applied a controlled force in our MWCNTs sample by using calibrated weights from 100 to 300 g; Fig. 1 shows the experimental set up used in these tests. We performed many experiments that confirm that the maximum weight that can be supported by our matrix of SiO_2 was of 400 g before the yield point. A force F_2 was applied perpendicularly to the surface of the sample in a cyclic mode. This force was applied in order to induce a deformation in the sample. During this experiment we measured the contraction in length between points "A" and "B" by using electric strain gauges. These transducers are made of very fine wires that were glued to the sample being investigated. As the forces are applied to the sample, elongation or contraction of the wires takes place concurrently with a similar deformation as in the studied material. These changes in length alter the electrical resistance of the gauge, which can be measured and calibrated to indicate the strain that takes place [23].

Mathematically, the components of a force F acting on an area A is the stress σ and can be described by using the Hook's law [24].

$$\sigma = \frac{F}{A} \tag{1}$$

In general, the point of application of a force originates strong variations in the mechanical resistance and extensional strain; and it has to be referred to the plane of the section when we consider a two- or three-dimensional sample. If L_0 is the initial length of our reference and L is the observed length induced by a mechanical load, the elongation ε per unit of initial length will be given following the extensional strain relation [25]:

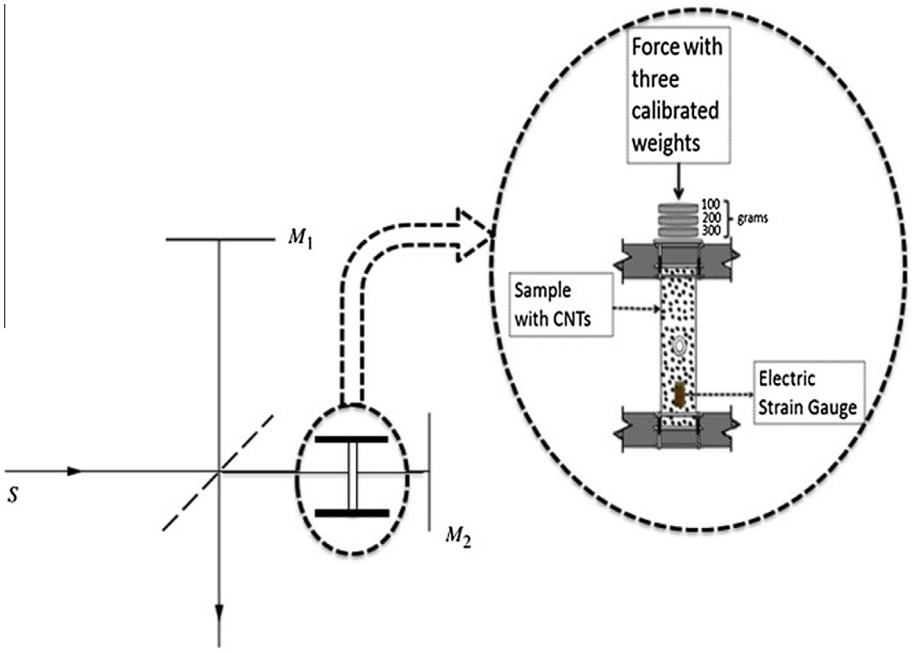

Fig. 2. Experimental set up to measure deformations.