



Effects of pressure transmitting media on Raman features of single-walled carbon nanotubes

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ARTICLE INFO

Article history:

Received 10 March 2008
Accepted 10 April 2008 by D.J. Lockwood
Available online 16 April 2008

PACS:

81.07.De
68.35.Ja
63.22.+m
78.30.Na
62.50.+p

Keywords:

A. Nanostructures
B. Surfaces and interfaces
E. Inelastic light scattering
E. High pressure

ABSTRACT

We have performed high pressure Raman studies of single-walled carbon nanotubes (SWCNTs) by using different organic solvents as the pressure transmitting medium (PTM). A linear dependence of pressure coefficient of Raman G band is found with the molecular weight of organic solvent. In addition, according to the experimental data and a comparative analysis of pressure measurement without the PTM, we explain the occurrence of plateau and hardening behavior of Raman G and D* bands upon increasing pressure.

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1. Introduction

Since their discovery in 1991, carbon nanotubes have attracted much attention of the scientific community. Their properties are considered as extremely valuable for the future development of nanotechnology [1]. The behavior of single-walled carbon nanotubes (SWCNTs) under hydrostatic pressure has been investigated by a number of theoretical [2–6] and experimental [7–22] works. Experimental investigations have employed powerful techniques to elucidate the structural, vibrational, mechanical and electronic properties of SWCNTs under high pressure. These include x-ray diffraction [8,10,13], neutron diffraction [9], optical absorption spectra [12] and Raman spectroscopy [7,11,14–22]. Diamond anvil cells (DACs) have been routinely used to perform high pressure Raman studies of different materials for the past few decades [23]. In such experiments, the sample is placed inside a microhole in a metal gasket that becomes a pressure microchamber pressurized between two diamond anvils. The microchamber

is filled with the pressure transmitting medium (PTM) to ensure a hydrostatic pressure on the sample as DACs are pressed together. In recent years, many Raman spectroscopy experiments have focused on solvent effects on structural and vibrational properties of nanotubes under pressure [20–22]. This study is extremely important because SWCNTs are very sensitive to their chemical environment due to their one dimensional character [16]. However, the detailed mechanism of interactions remains incomplete as well as the high pressure evolution of nanotubes and the interpretation of experimental data is still debated [24,25].

In order to explore the mechanism as to how PTM molecules interact with individual SWCNTs, we performed high-pressure Raman spectroscopy experiments by using five different organic solvents as the PTM in this work. We acquired important experimental observations and results not previously reported. Most importantly, we proposed explanations for the occurrence of plateau and hardening behavior of Raman G and D* bands upon increasing pressure, and found the linear dependence of pressure coefficient of Raman G band with molecule weight of the PTM. Our experimental data and analysis provide a necessary basis to further understand the high-pressure evolution of SWCNTs and effects of the PTM on vibrational properties of Raman bands of SWCNTs under pressure.

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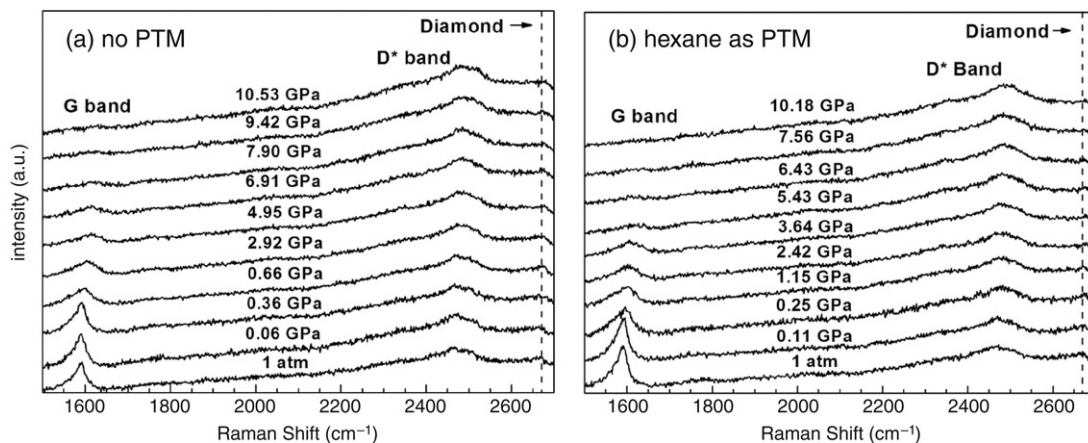


Fig. 1. Raman spectra of SWCNTs excited by the 514.5 nm line of an Ar-ion laser in pressure measurements (a) without the PTM and (b) with hexane as the PTM at room temperature.

2. Experimental procedures

A powder sample of SWCNTs with high purity, purchased from Chengdu Org. Chem. Co., was used. The average diameter of the sample was estimated to be ~ 1.3 nm by a high-resolution transmission electron microscope (HRTEM) (JEOL JEM-2010). Raman spectra were acquired by using a LABRAM-HR micro-Raman system equipped with a microscope attachment. No polarizer was used and a backscattering geometry was employed to collect the Raman signal. The 514.5 nm line of an Ar-ion laser with the power of 15.2 mW was used to excite the sample. Samples were loaded into a DAC for high-pressure measurement. Five different organic solvents, namely methanol, propanal, 1-butanol, hexane and octane, were taken as PTM. Pressure is measured via the standard ruby R1 line method [26], and we have confirmed from the linewidths and the separation of the ruby R1 and R2 lines that the hydrostatic or quasi-hydrostatic condition was satisfied in the applied pressure range (0–11 GPa). In this work, we also performed the measurement without using PTM for the purpose of a comparative analysis.

3. Results and discussion

Raman spectra of SWCNTs excited by the 514.5 nm line of an Ar-ion laser upon increasing pressure are shown in Fig. 1. The measurements with no PTM and hexane as PTM are shown in Fig. 1(a) and (b), respectively. It is well known that SWCNTs have four main Raman modes studied frequently, which are the RBM (the radial breathing mode in the range of $100\text{--}600\text{ cm}^{-1}$), D band (disorder-induced mode in the range of $1250\text{--}1450\text{ cm}^{-1}$), G band (tangential mode at about 1600 cm^{-1}) in the first-order and D* band (the overtone of D band) in the second-order Raman spectra [27,28]. In this work, we only focus on Raman features of G and D* bands due to weak Raman signals of the RBM and D band when exerting pressure in measurements. In Fig. 1, one can find that the intensity and frequency shift of second-order diamond Raman peak ($\sim 2668\text{ cm}^{-1}$) do not change with applied pressure. So we can take it as the reference to normalize Raman intensities for the G and D* bands. Upon increasing pressure, one can also find that the peak intensity of G band decreases gradually and disappears at 6–7 GPa, regardless of whether or not we took the PTM in pressure measurements. However, the peak intensity of D* band doesn't change with pressure. Raman peaks of both bands shift to higher frequency when being applied to higher pressure. As we refer above, the Raman peak of G band is mainly raised by the tangential stretching of C–C bonds and one of D* band is induced by defects/disorder of SWCNTs. We consider that applied pressure

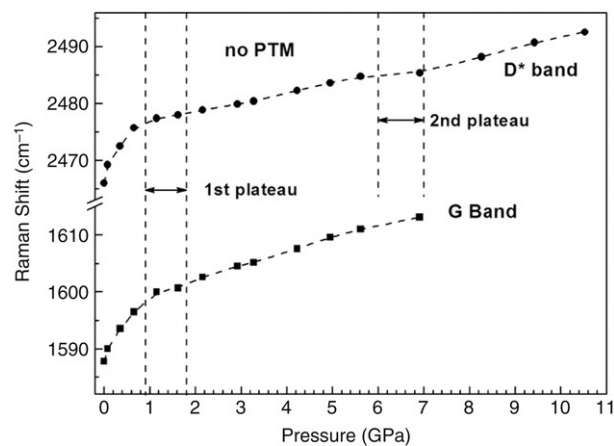


Fig. 2. Raman shifts of G band and D* band as a function of pressure under the non-hydrostatic condition.

can weaken the C–C tangential vibrations but cannot change the inherent quality of SWCNTs. Therefore, the peak intensities of two Raman bands exhibit their own features.

For Raman spectra measured under non-hydrostatic pressure condition in Fig. 1(a), we can acquire Raman shifts of G and D* band as a function of pressure, as shown in Fig. 2. There is a remarkable plateau in the range of 0.9–1.8 GPa for G band as well as there are two plateaus locating at 0.9–1.8 GPa and 6.0–7.0 GPa for D* band, respectively. Also, we exactly measured Raman shifts of G and D* band as a function of pressure with five different organic solvents as PTM. These relationships are shown in Fig. 3(a) and (b). One can find that there is still a plateau for G band as well as there are two plateaus for D* band. However, pressure ranges of plateaus in both Raman bands have not remarkable difference for different pressure transmitting media. In all cases, Raman shifts of both G and D* bands exhibit hardening behaviors upon increasing pressure. These phenomena are also observed in previous works [19,20,29,30]. For example, Peters et al. [19] found that the plateau for G band locates ~ 1.7 GPa under non-hydrostatic condition and Amer et al. [20] found these two plateaus for D* band locate at ~ 2 GPa and 6 GPa, respectively, under hydrostatic condition by using methanol as the PTM. Our experimental results are basically in accord with these observations.

As for the reasons of the hardening behavior and pressure coefficient transition of Raman band, Amer et al. [20] think they are attributed to the adsorption of molecules of the PTM around nanotubes. However, one can find that the plateau would

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