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Pseudometallization of single wall carbon nanotube bundles with intercalation of naphthalene

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Intensive change in electronic structure of single wall carbon nanotube (SWCNT) bundles is observed, arising from intercalation of naphthalene into the interstitial spaces of the bundles with adsorption from solution. Ultraviolet photoelectron spectroscopy shows a clear increase in the density of states reaching the Fermi level, explicitly indicating pseudometallization of SWCNT by this simple and scalable intercalation method. On the other hand if a nonvolatile pentacene is deposited on the external bundle surface in vacuum, SWCNT shows no similar change in the density of states.

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I. INTRODUCTION

Application of single wall carbon nanotubes (SWCNTs) in electronics, particularly as quantum wires for nanoconductors, nanotransistors, and gate electrodes in field emission transistors, has been extensively studied in recent years.[1–9] The practical problem in these applications comes from the lack of possibility to synthesize strictly one desired type of the SWCNT. Some of the first advances have been reported by Li[10] or Bachilo *et al.*[11] who synthesized the nanotubes bulk containing mostly tubes of certain chirality. Unfortunately, it is not yet possible to produce the nanotubes of perfectly uniform chirality, for example, only metallic, for which the (n,m) indices are $n=m$, or $(n\text{-}m)=3i$ (i is an integer). In most of the cases, the synthesis gives a mixture of chiralities, summarized nicely in Refs. 12–14 and therefore selective synthesis remains the ultimate goal for scientists in the field. The most recent success in this approach is reported in Ref. 15 where varying the noble gas ambient during thermal annealing of the catalyst, in combination with oxidative and reductive species, resulted in maximum of 91% of the obtained nanotubes being of metallic conductivity.

In contrary to above-described selective synthesis approach, finding a treatment which would design the electronic properties of the carbon nanotubes film (bundled structure popularly called "bucky paper") is also an exciting possibility. Ostojic *et al.*[16] and Finnie *et al.*[17] have reported that the electronic properties of SWCNT are affected by environments, where SWCNT still has large band gaps. We present here a simple concept of how it is possible to influence the electronic structure of the SWCNTs film by adsorption treatment from solution with small polyaromatic compound. In the extensive study we conducted on adsorption from solution of polyaromatic and aliphatic hydrocarbons on SWCNTs,[18–21] we found that adsorption of phenanthrene and tetracene depends on the nanocurvature, which is the feature defining the difference between the nanotubes and the plain graphite sheet. Very intriguing finding in these studies was also the adsorption of naphthalene, a two-ringed molecule.[19] This substance was not expected to be stably adsorbed by the curved graphitic surface of the nanotubes at room temperature because of the extreme low sublimation point and the high vapor pressure for a solid. After we studied deeper this phenomena, in present study we are able to show how naphthalene is adsorbed in the "sandwich" manner in the interstitial space between the nanotubes if the adsorption is performed from the solution. Moreover, it is influencing the overall electronic structure of the nanotubes film inducing a metallic behavior, as determined by ultraviolet photoelectron spectroscopy (UPS). The case is opposite for the adsorption coating of the tightly bundled nondispersed SWCNT film, as evidenced by vacuum deposition of pentacene film on the SWCNT film at room temperature. Since SWCNTs are not debundled, coating occurs only on the outer surface and no change in the electronic structure of the film was observed.

II. EXPERIMENTAL

The SWCNTs produced by laser-ablation synthesis, with Ni and Co as catalysts (Institute of Research and Innovation, Chiba, Japan) were purified by refluxing in 15% H_2O_2, at 373 K for 35 min; the metallic impurity content was only 4 wt %. Thus-purified SWCNTs have still closed caps according to the N_2 adsorption analysis which is shown later.

Adsorption from solution was performed as already reported in our previous work.[18–21] 3.28 mg of SWCNTs and 5.17 mg of naphthalene (total molecular surface ratio approximately SWCNT/naphthalene=1:1) were immersed in 100 ml of toluene and ultrasonicated in iced water for 4 h until dispersions were homogenized. Samples were then filtrated and obtained SWCNTs film cut into rectangular piece of 1.5 cm × 1 cm.

After the preparation of the film in the atmospheric conditions, the sample was inserted using an oil-free airlock system into the preparation chamber of the UPS apparatus with a hemispherical electron energy analyzer (SPECS-PHOIBOS 100) (energy resolution: 50 meV), in which a measurement chamber is connected to the preparation chamber using a UHV valve aiming to preserve a cleanness of the measurement chamber.[22] The base pressure of the preparation chamber was 4.9×10^{-8} Pa, while after the insertion the sample, it decreased to 1.2×10^{-6} Pa immediately. The natural degassing was continued for 15 h until the pressure reached 1.1×10^{-7} Pa. Then the He I UPS measurements were performed in the measurement chamber first. Consecutively, we annealed the film in the preparation chamber to remove the adsorbed molecules from the sample. The annealing temperature was sequentially increased with aim to remove slowly and partially the adsorbed molecules at lower temperatures and to gradually reach the complete desorption at higher temperatures. In each step, the film was cooled down naturally to 295 K and all UPS measurements were performed at this temperature.

A SWCNT film for pentacene deposition was newly prepared and was cleaned by annealing at 673 K of 24 h in the preparation chamber. Pentacene was purified by three-cycle sublimation in an Ar gas stream of 13 Pa before use and was deposited after sufficient degassing, as 0.5 nm layer at rate of 0.1 nm/min onto the cleaned SWCNT film at 158 K. The deposition amount and rate were measured with a quartz microbalance. Raman measurements were performed by 532 nm frequency-doubled Nd:YAG Laser (1.8 mW), on adsorption treated and dried samples by direct examination of the multiple spots on the films.

III. RESULTS AND DISCUSSION

Figure 1 shows the comparison of the heat-cleaning and annealing effects on UPS spectra of naphthalene-adsorbed SWCNT films. These spectra are selected from all heat-treatment processes and belong to (i), a nonheated sample, followed by spectra of the sample heated at (ii) 423 K for 1.0 h, at (iii) 523 K for 2.0 h, at (iv) 573 K for 10.0 h, and (v) 673 K for 17.5 h, respectively. In the (v) spectrum, the several band peaks A–D can be recognized and they are unchanged after heating at higher temperatures [see, for example, spectra (iv) and (v)]. The peaks A–D are thus ascribed to valence band features of a clean SWCNT film. To confirm this result, a photoemission spectrum ($h\nu=65$ eV) of SWCNT film (vi) measured by Ishii *et al.*[23] is compared. It was confirmed that the peaks in the (v) are well corresponding to A–D in the (vi) spectrum although a large background of the secondary electrons somewhat conceals the appearance of the spectral bands in the (v). From the previous study,[23] the band A around 3 eV and the band D around 8 eV can be related to the π band and σ band of graphene sheet structure, respectively. The confirmation of the bands indicates that the surface of the final-heated SWCNT film is rather clean.

On the other hand, only slight differences can be observed between the (i) and (v). In the (i) spectrum, there is a band located clearly at lower energy side from band A, and several broad bands between bands A and C, indicated by bars. Naphthalene-adsorbed SWCNT films should show a different electronic structure from that of the clean SWCNT films. Indeed, positions of the features in the (i), which are clearly different from those in the (v), correspond well with those of a (vii) gas-phase UPS spectrum of naphthalene molecules [see also the (i)′ spectrum, which is a negative of second derivative of the (i) spectrum].[24] Therefore, the features in the (i) spectrum originate from naphthalene molecule, and especially the shoulder at 2.7 eV can be assigned to the highest occupied molecular orbital (HOMO) of the naphthalene solid.[25] Nevertheless, the changes from the (i) to the (v) suggests that the surface of the nonannealed sample is in part influenced naphthalene molecules, which can be supported by metastable atom electron spectroscopy (MAES), the most surface-sensitive technique (supporting material Fig. S1).[26] It is reasonable to think that naphthalene was adsorbed in the inner surfaces of the SWCNT bundles, as the molecule does not stably adsorb on the outermost surface of SWCNT at 295 K.

From the UPS results in Fig. 1, one would expect no particular changes in the Fermi region since the changes in spectral features between spectra (i) and (v) is so small that there is no strong electronic interaction between naphthalene and SWCNT. Nevertheless, a remarkable and unusual phenomenon indeed appears in the Fermi region as an appearance of a new peak by the naphthalene adsorption. The enlarged spectra of the naphthalene band-gap region are given in Fig. 2(a), where their intensities are normalized at 1.5 eV to compare the relative intensity of the spectra without a contribution of their background, especially the expected HOMO band tailing of naphthalene molecules. Surprisingly, overall intensity in the Fermi region is strongest for the nonannealed sample (i) and decreases depending on the sample annealing. To clarify the detail, we present differential spectrum of the (i)–(v) in Fig. 2(b), which also shows that, the intensity of the spectrum is over 0 up to the Fermi level and a clearly observable peak appears at 0.15 eV. Two weak dips at 0.34 and 0.68 eV, which are marked as D1 and D2, should be spikes of the (v) sample caused by the one-dimensional van Hove singularities in the occupied density of state (DOS) of semiconductor SWCNT near the Fermi level,[23] which can be seen also for other SWCNT films as discussed later, indicating the cleanness of the surface of the final-annealing SWCNT films again.

In general, the adsorption of organic molecules would work as a contamination for semiconductor or metal surfaces, causing a decrease in DOS near Fermi level since organic molecules usually have wide HOMO and lowest unoccupied molecular orbital (LUMO) gap. In particular, HOMO-LUMO gap of naphthalene is ca. 5 eV (Ref. 27) and thereby no electronic charge transfer is expected between naphthalene and SWCNT. Also, even if a background of secondary electrons is different between (i) and (v), it would not influence the increasing intensity near the Fermi region. Therefore, the appearance of the new peak at 0.15 eV and the increase in DOS near the Fermi level should be indeed unusual.

So far we reported that polyaromatic molecules could be trapped not only at the surface of SWCNT but also at an interstitial space between the SWCNT in bundles within a film.[19] The UPS and MAES results in this paper indicate strongly that the surface of the naphthalene-adsorbed SWCNT film is relatively clean even if the film is not annealed under the ultrahigh vacuum. Therefore, one can expect that the change in electronic structure of SWCNT film is induced by intercalation of naphthalene between the

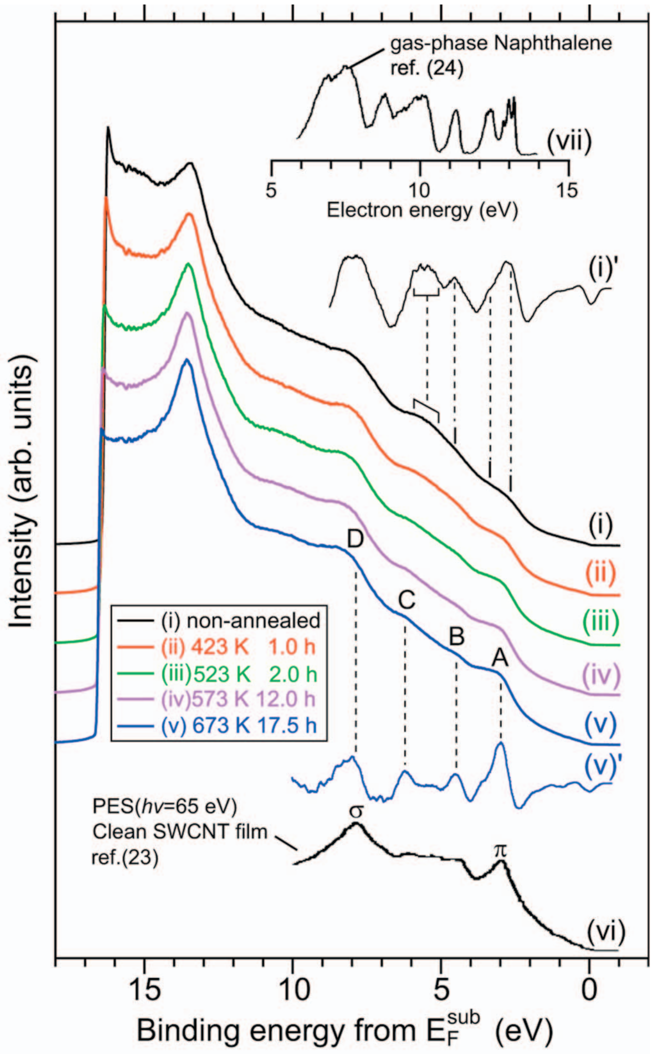

FIG. 1. (Color) He I UPS spectra of the naphthalene-adsorbed SWCNT films at different stages of sample annealing at increasing temperatures [see the insets (i)–(v)]. (i)′ and (v)′ spectra are negative of the second derivative of the (i) and (v) spectra ($-d^2I/dE^2$) to distinguish the naphthalene and SWCNT features. (vi) a reference spectrum of the clean SWCNT film measured by H. Ishii *et al.* (Ref. 23) using synchrotron light ($h\nu = 65$ eV). (vii) A gas-phase spectrum of naphthalene molecules (Ref. 24). Bars and marked region at the (i) indicate density of state different from that of the (v). The Fermi level (E_F) was determined from UPS of an evaporated Au film.

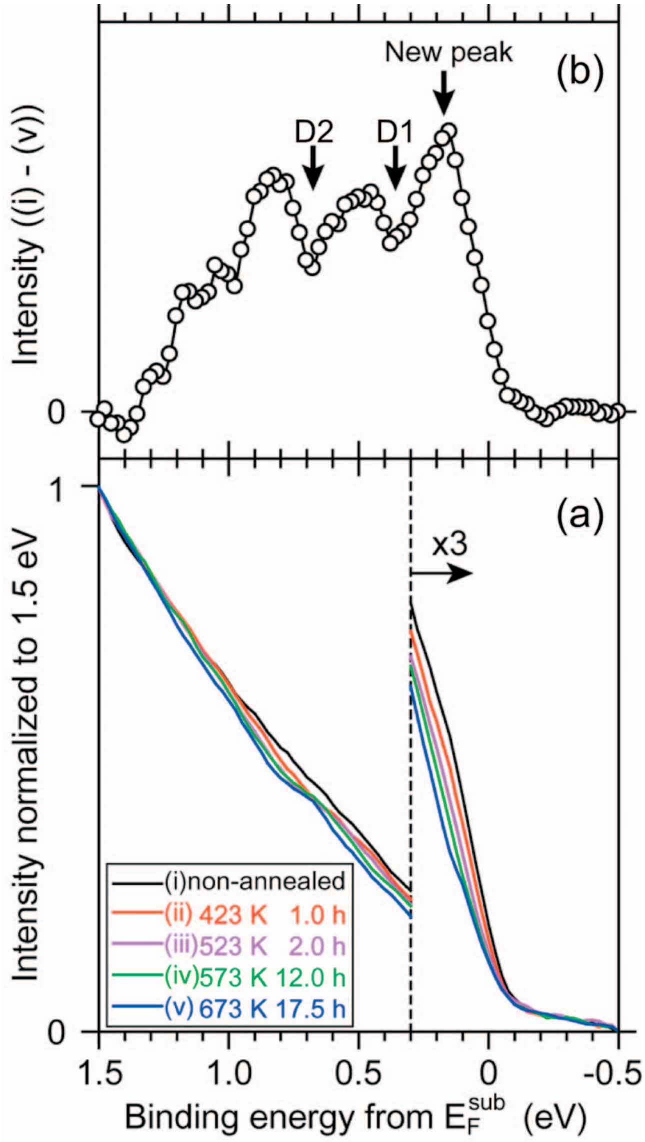

FIG. 2. (Color) (a) A comparison of relative intensity at the Fermi region of the He I UPS spectra of the naphthalene-adsorbed SWCNT film [(i)–(v)], where their intensities are normalized at 1.5 eV. (b) Differential spectrum of the (i)–(v).

SWCNTs in bundles and the naphthalene molecules adsorbed on the outer surfaces of the SWCNT bundles are not stable in vacuum, deducing no influence for the electronic structure of SWCNT film.

To additionally confirm the above mechanism, it would have been the best to adsorb the naphthalene molecules selectively on the outer surfaces of the SWCNT bundles at room temperature by sublimation technique. However, adsorption of naphthalene on the SWCNT bundles in vacuum is not possible because of its high vapor pressure. Pentacene is a five-rings aromatic molecule with a low-vapor pressure and the adsorption on graphite has been investigated successfully with photoelectron spectroscopy.[28,29] Therefore, we performed the adsorption of pentacene only on the exposed surface of the SWCNT by sublimation of pentacene in vacuum. However, there was no adsorption of pentacene on SWCNTs surface by sublimation method at 297 K, as confirmed by UPS. The adsorption by sublimation was then performed at 158 K. Figure 3 shows the UPS spectra of pentacene (mass thickness: 0.5 nm) on the SWCNT film at 158 K. In Fig. 3(a), before the pentacene adsorption, the several fine features D1 and D2 of SWCNT films, which are also seen in Fig. 2, are observed, evidencing that the surface of SWCNT films is clean at 158 K. After the deposition of pentacene, the D1 peak remains with an intense peak appearing at around 1.3 eV. The peak position of the new intense band corresponds well to that of HOMO of pentacene monolayer on highly oriented pyrolytic graphite (HOPG) [see the spectrum in Fig. 3(a)], where the pentacene molecule is lying flat to

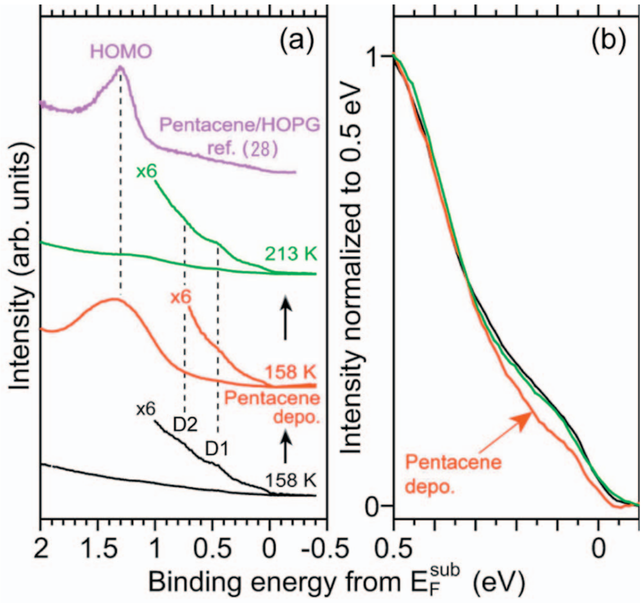

FIG. 3. (Color) (a) HOMO band and (b) Fermi level regions from He I UPS spectra of the SWCNT film, covered by a 0.5 nm purified pentacene layer, deposited at 158 K: clean SWCNT films at 158 K (black); pentacene deposited SWCNT films at 158 K (red); after annealing at 213 K (green). As a reference, UPS spectrum of HOPG, covered by monolayer of flat-lying pentacene (0.3 nm) at 297K, reported in Ref. 28 is also shown at the top in the (a). In the (b), the intensity is normalized at 0.5 eV to discuss the relative intensity near the Fermi region without a contribution of a back ground slope of pentacene HOMO band as can be seen at 1.3 eV.

the substrate surface at room temperature.[28,29] Also, considering about 1 nm of the mean-free path of electrons ejected from sample surface for UPS, the D1 peak in the 158 K spectrum indicates clearly that the coverage of pentacene should be below a few layers. As can be seen in Fig. 3(b), however, no increase in the intensity was observed near the Fermi level irrespective of adsorption of pentacene. On the other hand, the HOMO band became weak, when the sample temperature increased to 213 K [see Fig. 3(a)], indicating desorption of the pentacene molecules from the surface of the bundled SWCNT film, which supports no adsorption of pentacene at 295 K. These results show that the adsorption of aromatic molecules on the outer surface of SWCNT bundles is extremely weak and cannot affect the electronic structure of the SWCNT films, in other words, only the intercalation of the naphthalene molecules in the bundle structure can induce the metallic behavior of the SWCNT films probably by gluing SWCNTs electronically.

Strong confirmation of the UPS results came from the nitrogen adsorption isotherms of the sample films before adsorption (as-purified SWCNTs film) and after adsorption of naphthalene shown at Fig. 4. Namely, the Brunauer-Emmett-Teller surface area,[30] which is the measure of the "outer" surface in the nanoporous materials, of as-purified sample was 381 m^2/g, while the value drops as low as to 63 m^2/g after naphthalene adsorption. The first value corresponds to the usual value obtained for similar SWCNT films with closed caps[19] in which case, N_2 molecules are adsorbed on the external bundle surface in addition to the interstitial pores. As naphthalene molecules are adsorbed on the external surfaces in the solution, inducing the naphthalene-intercalated bundle and their aggregation, the N_2 adsorption in the interstitial spaces is blocked by the naphthalene molecules intercalated and the contribution from the external surface of the aggregated bundles becomes much less. The x-ray diffraction showed the presence of the peak corresponding to the intercalation structure (supporting material Fig. S2);[26] each SWCNT is coated monolayerly with naphthalene molecules and then the double layer of naphthalene is formed in the inter-SWCNT spaces. The results match the above conclusions represented by the UPS on naphthalene and pentacene adsorption. In the case of adsorption from solution, the interactions between the SWCNTs and the organic molecules are particularly improved by ultrasonication. At each single nanotube, the molecules are well adsorbed and after the drying, the tubes are bundled again. These interactions between the naphthalene molecules in the interstitial space between two NT surfaces remain strong. However, molecules exposed to the outside, namely interacting only with one nanotubes surface, are desorbed, already at low annealing temperatures.

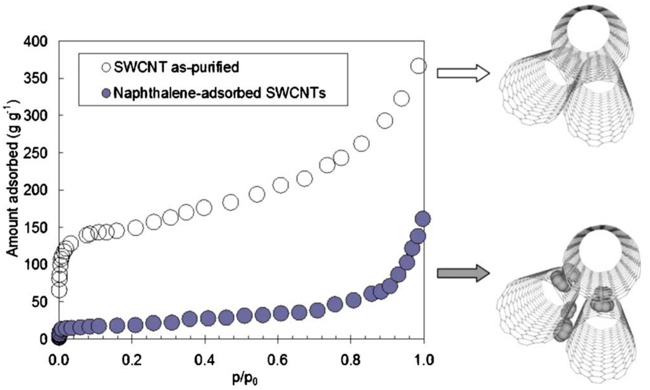

FIG. 4. (Color online) Nitrogen adsorption isotherms of as-purified SWCNTs film and the naphthalene-adsorption treated sample with corresponding schemas showing idealized pictures of the bundles in both cases.

Along this study a Raman spectroscopic examination was also performed. It has shown the presence of a strong interaction between SWCNT and naphthalene molecules. The Raman intensity for the radial breathing mode (so-called RBM), which can be observed below 300 cm^{-1} region, is strongly enhanced when the incident or scattered light is in resonance with an excitonic transition.[31] Therefore, the intensity change in the RBM reflects the electronic structure change in the SWCNT. Also, each RBM peak position can be assigned to the SWCNT of the inherent tube diameter through the chirality using indexes (n,m) which describe the electronic state. It is interesting to show the Raman spectroscopic data on HiPco (high-pressure CO conversion) SWCNT sample which contains a considerably large amount of metallic SWCNTs. The tube diameter distribution of the HiPco SWCNT sample is very wide and thereby many RBM peaks can be observed. Figure 5 shows the RBM spectral change in the HiPco sample with adsorption treatment of naphthalene

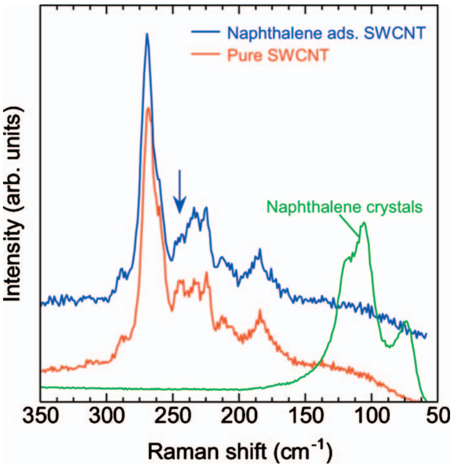

FIG. 5. (Color) Raman spectra of the RBM region in pure and naphthalene-adsorption treated Hipco SWCNTs, where naphthalene crystal spectrum is also shown. The decrease in the Raman intensity at 244 cm^{-1} (as arrowed) indicates the change in electronic structure of semiconducting SWCNTs by the naphthalene adsorption.

together with the spectrum of crystalline naphthalene. This figure clearly indicates the decrease in the RBM peak at 244 cm^{-1}, being assigned to a semiconducting peak (n, m $=11$ and 3), upon adsorption of naphthalene, although we cannot observe explicit changes in other wave number region. Here the peak at 244 cm^{-1} is indicated by an arrow in the figure. Accordingly this RBM data support the strong interaction between SWCNT and naphthalene molecules, which is evidenced with UPS in this study.

Recent experiments by other groups done with the combination of normal incidence x-ray standing waves and photoemission spectroscopy about π-conjugated molecules on novel metal substrates have shown that the adsorption distance between the molecules and the substrate impacts the charge transfer between the molecules and substrate.[32] A small distance less than van der Waals radius of the atomic species in the molecules and the substrate can happen the charge transfer, even though this phenomena cannot be expected from an energy level diagram between the molecular films and the substrate, i.e., ionization potential and electron affinity of the molecules versus work function of the metals. From a point of view of energy level diagram, it is unlikely that the charge transfer between the adsorbed naphthalene and CNT films as mentioned in our manuscript. However, for the case of naphthalene adsorbed SWCNTs it is expected that the sandwiching effect of naphthalene between CNTs, which represent curved graphene sheets, gives rise to a strong interaction between them, and thus may result in a smaller distance than expected by van der Waals radius of carbon atoms. Although it is not easy to investigate the adsorption distance of the intercalated naphthalene to a CNT surface in the bundles, authors strongly believe this scenario is the origin of the increase in DOS near the Fermi level.

IV. CONCLUSION

In summary, the adsorption of SWCNTs with naphthalene molecules in solution under the ultrasonic treatment induces the intercalation of naphthalene molecules in the SWCNT bundles. The naphthalene-intercalated SWCNT bundles have a metal-like DOS structure near the Fermi level with UPS, although coating of the external surface of the SWCNT bundle with pentacene molecules by vacuum sublimation does not change the electronic structure of the SWCNT. The liquid phase adsorption treatment of the SWCNT bundles with naphthalene is expected to be a scalable and simple method to donate uniformly a metallic nature to the mixture of metallic and semiconductive SWCNTs.

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