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Tube diameter estimation of single-walled carbon nanotubes by XRD simulation

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1 Introduction

For the proposal 2013G031, we investigate structural change of poly-iodine ions encapsulated in single-walled carbon nanotubes under high pressure. In the way of this investigation, it was found that it is very important to understand the relation between the structure of poly-iodine ions and tube-diameter. To clarify the relation, we should know reliable tube diameter value. For the estimation of the tube diameter, direct TEM observation is of course known and we can calculate tube-diameter with peak positions of Radial Breathing modes (RBMs) observed in the lower wavenumber region of Raman spectrum. However, TEM observation offers only local information and Raman method is effective for only a small number of tubes which are resonanced with excitation laser light. We reported in 2013 that we can estimate mean pore diameter of mesoporous materials which have ordered channel-like mesopores.[1] In the present study, we applied this method to the analysis of mean tube-diamater of single-walled carbon nanotubes. Furthermore, we performed in-situ XRD measurements of iodine encapsulated SWCNTs whose mean tube diameter was determined by our method.

2 Experiment

SWCNTs used in the present study were produced by arc-dishcarge method (Meijo-Nanocarbon: SO type). Iodine doping was performed by electrochemical oxidation method. High pressure experiments were done with diamond anvil cell. Synchrotron XRD measurements were performed at a beam line BL-18C of KEK. High energy X-ray of 20 keV was used as an incident beam and an imaging plate was used as a detector.

3 Results and Discussion

Figure 1 shows the comparison between the observed and simulated XRD patterns of the empty SWCNTs. As shown in the figure, the simulated pattern reproduce well the observed pattern. By this simulation, the mean tube diameter and the tube-tube distance were determined to be 1.48 and 0.31 nm, respectively. On the other hand, Fig. 2 shows the change in the XRD patterns of I@SWCNTs as a function of pressure. Even at low pressure we could not see 100 peak which is the strongest peak of SWCNT (Fig. 1) probably due to the encapsulation of iodine ions. The broad peaks are observed at around of 10 deg. and they can be assigned as higher order diffractions of I@SWCNTs. It was clearly observed that these peaks shifted to higher angle side with pressure. The detailed analysis of this pattern change is not yet finished.

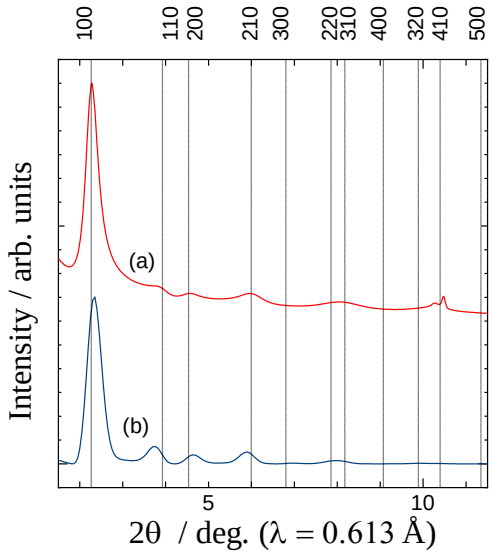

Fig. 1: The (a) observed and (b) simulated XRD patterns of the SWCNT sample.

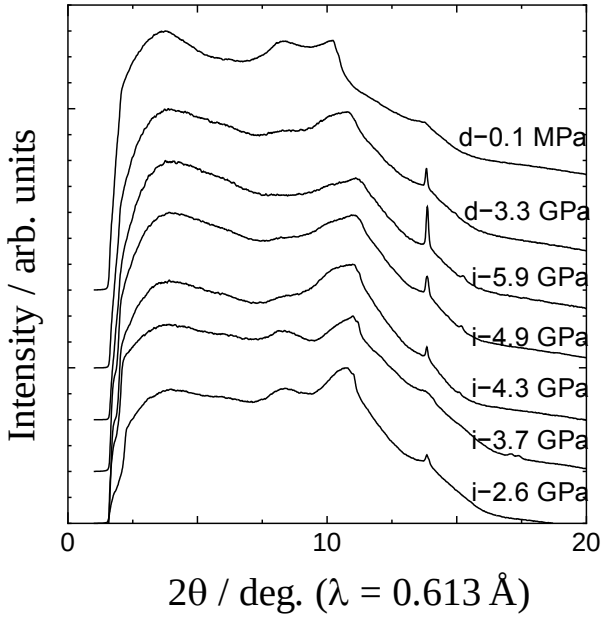

Fig. 2: Change in the XRD patterns of I@SWCNTs as a function of pressure. i-P and d-P indicate increasing and decreasing pressure values, respectively.

References

[1] Pore Size Determination in Ordered Mesoporous Materials Using Powder X-ray Diffraction, Y. Ishii, Y. Nishiwaki, A. Al-zubaidi, S. Kawasaki, J. Phys. Chem. C, 117, 18120-18130, (2013).

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