Progress in Fine Structure Characterization of Carbon Materials with TEM

- 2D and 3D characterization of nanocarbons -

Noriko Yoshizawa
National Institute of Advanced Industrial Science and Technology, 16-1 Onogawa, Tsukuba
305-8569 JAPAN
e-mail: noriko-yoshizawa@aist.go.jp

Introduction

TEM (transmission electron microscope) has been used for a characterization of microtexture in carbon materials. Not only for traditional carbon materials such as graphite-based materials, carbon blacks and fibers, and activated carbons, observation with TEM plays a very important role also for a study of nano-sized carbon materials, including carbon nanotubes, nanofibers, and other types of nanoparticles.

TEM tomography, or 3D-TEM, is one of the recent progresses in the TEM technology. An image taken with TEM is generally a two-dimensional (2D) projection, and therefore it is difficult to characterize three-dimensional (3D) structure, including overlaps of texture or thickness of intended structure. Rotation of a specimen holder is one of the key techniques to obtain these types of 3D information in TEM. For this matter, the development of specimen holder rotated in a wide range, between +75° and -75° or wider, was achieved, which enables us to do the tomography analysis with incident electron beam in TEM. In this presentation, some of our related works will be discussed for a development of carbon materials to be used in energy storage devices.

Distribution of Pt particles on carbon nanoparticles

As a stable electrode in proton-exchange membrane fuel cell (PEFC), Pt/carbon catalyst attracts many attensions, and it is reported that carbon structure seemed to have potent influence upon its efficiency. We aimed to clarify the effect of carbon structure upon Pt dispersion by using pristine carbon nanosphere (CNS, 200 nm in average diameter) and its graphitized sample as supports [1]. As shown in Fig. 1, Pt particles on the pristine CNS were dispersed homogeneously on the surface of carbon, while the graphitized CNS had Pt particles aggregated along the polyhedron ridgelines or between CNS particles. It is also confirmed that all the Pt particles found in the slice image were present on the surface of CNSs, not in the texture of them.

For carbon nanoparticles, 3D connection of inner voids in carbon black particles was also clarified by using this technique [2]. Figure 2 shows some representative slice images reconstructed from 2D-projection images collected at different rotation angles. Here, we can estimate the inner texture as if we virtually cut the particles from the top to the bottom by the plane parallel to the grid. According to the sliced images, this bunch of carbon black particles presumably consists of four or more particles. The four particles obviously recognized are: two attaching ones on the left, large one on the right, and small one between the two particles above. Furthermore by viewing the series of slice image, connectivity of carbon texture in the outside region, as well as of open space in the center region, of aggregating particles were comprehensively illustrated. The fact was also confimed that the concentric texture in the center region is completely covered by the graphitic texture in the outside region, and that the open space in the center region is not connected to the surface of the particle. We believe that the specific understanding of the spatial arrangement of the particles shown above is one of the advantages of using the electron tomography technique.

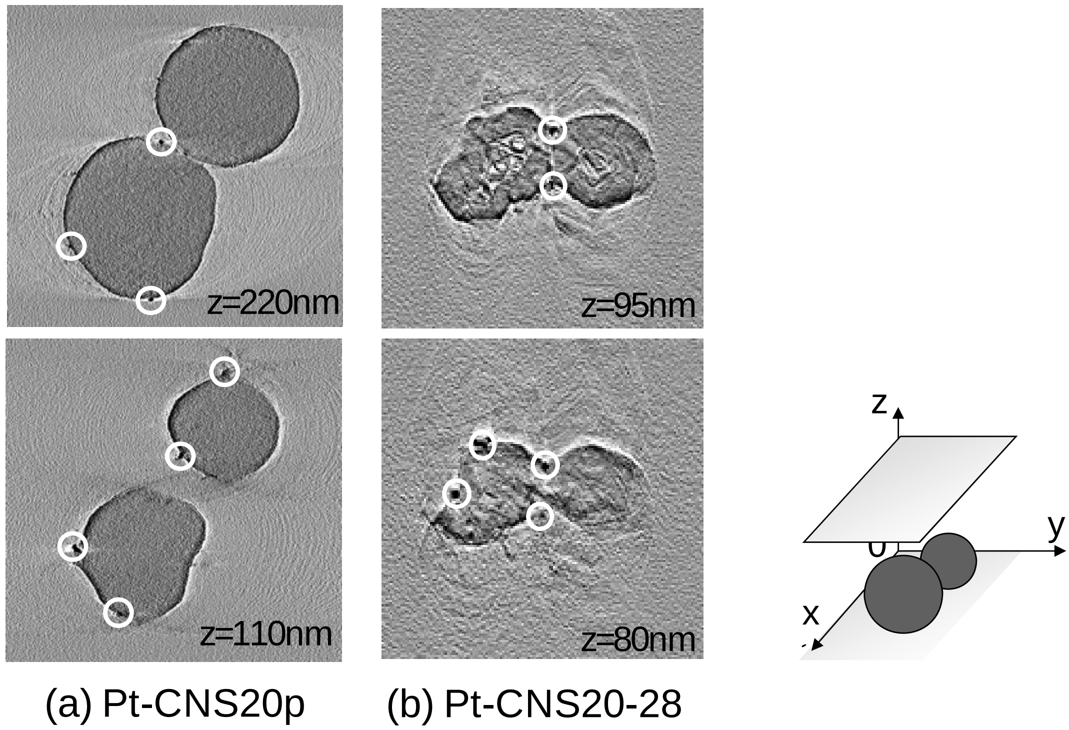

Fig. 1 Series of slice images for (a) Pt/CNS20p (Pt on pristine CNS) and (b) Pt/CNS20-28 (Pt on graphitized CNS). Pt particles detected are marked with white circles in each slice image.

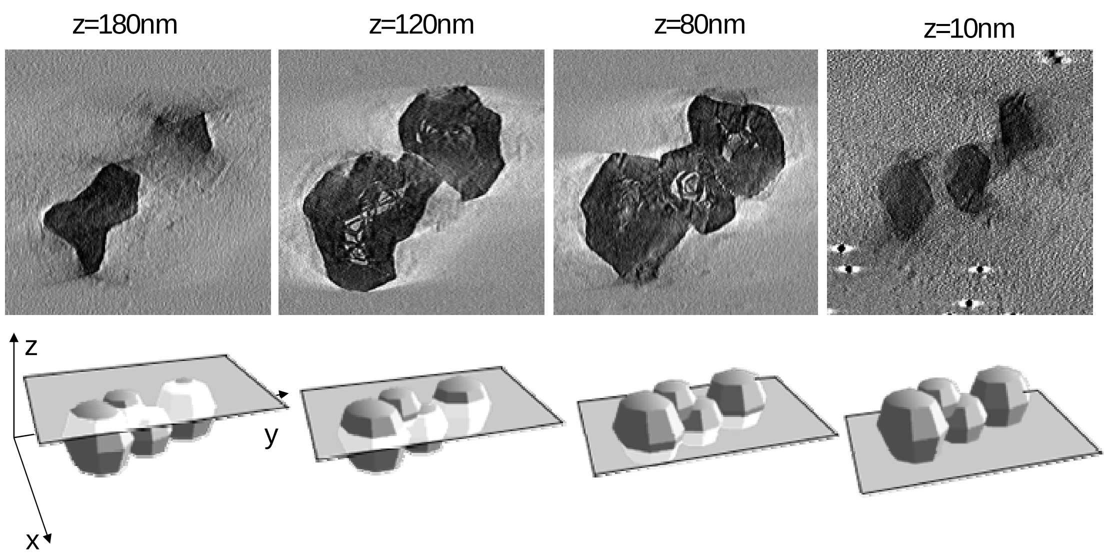

Fig. 2 Slice images of carbon black particles and their 3D-model with imaginary plane at different height.

References
[1] N. Yoshizawa, Y. Soneda, H. Hatori, H. Ue and T. Abe, *J. Nano Res.*, **9** (2010) in press.
[2] N.Yoshizawa, O. Tanaike, H. Hatori, K. Yoshikawa, A. Kondo and T. Abe, *Carbon*, **44** (2006) 2558.