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Three dimensional characterization of a silica hollow sphere with an iron oxide core by annular dark field scanning confocal electron microscopy

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Scanning confocal electron microscopy (SCEM) was first implement by N.J.Zaluzec et al. [1] and has recently become established as a three dimensional (3D) characterization tool by operating in energy filtered (EF) [2] and annular dark field (ADF) modes [3]. Since the 3D information transfer in these modes is improved compared with that of ADF-STEM and bright field (BF)-SCEM, the depth of field of not only point like objects but also laterally extended ones can be dramatically reduced [4-6]. Incoherent EF-SCEM has almost ideal 3D transfer function but inherently its signal is very small. On the other hand, although ADF-SCEM cannot be incoherent imaging and therefore its 3D transfer function is not ideal when low angle ADF electrons are collected, the depth of field of ADF-SCEM is capable of producing interpretable 3D images with sufficient signal intensity for various types of objects [6].

In the present work, we characterized 3D structure of silica hollow spheres with iron oxide cores by ADF-SCEM using an Oxford-JEOL 2200MCO equipped with aberration correctors for probe-forming and imaging lenses, pinhole, 3D stage scanning system and ADF aperture [3,7]. The sample was a rattle-type silica hollow sphere including an iron oxide (Fe_2O_3) core prepared by the template method described by Zhu et al. [8].

Elemental maps taken by energy filtering are shown in Fig.1. It is obvious that iron covers the silica surface, but internal iron distribution cannot be clearly observed. Figure 2 shows (a) HAADF-STEM image and (b,c) XY and XZ sliced images extracted from the 3D reconstruction through a computer tomography (CT) method. These were produced from a tilt-series of HAADF-STEM images from -60° to +60° at 2° decrements. The acquisition time of the tilt-series was about 2 hours. The internal structure is clearly seen in the XY sliced image, but it is noted that the XZ sliced image exhibits missing-angle induced artifacts.

Figure 3 shows (a) HAADF-STEM image and (b,c) XY and XZ sliced images taken by ADF-SCEM. It was found that the core was also hollow, that is consistent with the CT observation results. Thus, it is demonstrated that ADF-SCEM enables us to observe sliced images of objects at desired positions directly.

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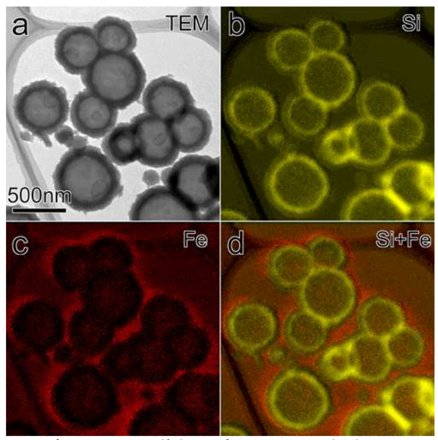

FIG.1 (a) HAADF-STEM image, (b) Si map, (C) Fe map, and (d) Si and Fe overlaid map of silica hollow spheres with iron oxide cores.

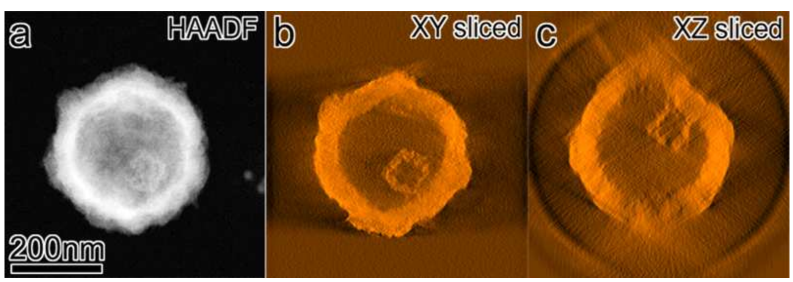

FIG.2 (a) HAADF-STEM image, and (b,c) XY and XZ sliced images extracted from TEM-CT 3D reconstruction of silica hollow spheres with iron oxide cores.

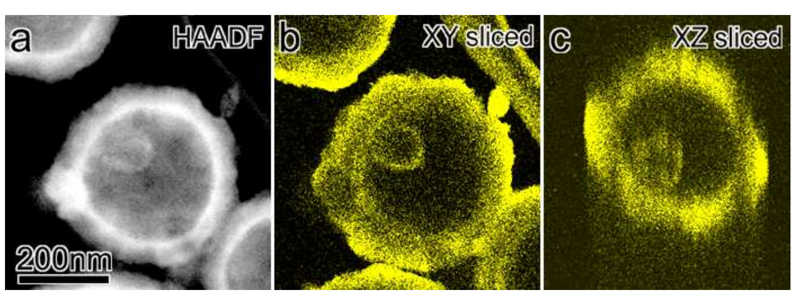

FIG.3 (a) HAADF-STEM image, (b,c) XY and XZ ADF-SCEM sliced images of silica hollow spheres with iron oxide cores.

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