Nano-Analysis of Mesoporous Carbon-Silica Nanocomposite by STEM-EELS

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Carbon incorporated silicon oxide nano-structures can emit white-light and such nanostructures have been often fabricated by chemical vapor deposition techniques or C^+ implantation into SiO_2 [1]. It is considered that the white-light emission is related to carbon incorporation in these nanostructures. We have synthesized a new white-light emitting mesoporous carbon-silica (MPCS) nanocomposite, using triconstituent co-assembly method [2] and systematic studies were done on the nanocomposites (specific surface area, the pore−size distributions, elemental composition and the FT-IR spectra [3]). It was reported that carbon in the MPCS nanocomposite samples plays a key role in producing the PL, though the mechanism of white photoluminescence (PL) in MPCS nanocomposites is still unclear..

In order to clarify the light emitting mechanism, the structure of MPCS nanocomposites, particularly the interface between carbon and SiO_2 was analyzed, using high-resolution scanning transmission electron microscopy (STEM) combined with electron energy loss spectroscopy (EELS). The MPSC nanocomposite sample was ground into fine powder, which was sprinkled on a carbon-coated micro-grid. The sample thus prepared was examined by a JEM-ARM200F double Cs-corrected STEM equipped with a GIF Quantum ER of High-Voltage TEM laboratory in Nagoya Unversity, operated at 80 kV to avoid the electron beam damages.

Figure 1 shows Si $L_{2,3}$ ELNES of a MPCS nanocomposite and those of SiC, SiO_2 and silicon-oxynitride for comparison. By comparing with the reference spectra, one can find that the Si $L_{2,3}$ ELNES of MPCS is mostly similar to that of SiO_2 in that the two feature peaks (108 eV and 115 eV) are easily observed. In addition, weak intensities are observed at the lower energy side of the main peak. This feature in Si $L_{2,3}$ of the MPCS nanovomposite is attributable to the presence of Si−C bonding, considering the feature of the reference spectra. The upper row of Figure 2 shows HAADF-STEM images observed along the directions parallel and perpendicular to the principal axis of the MPCS honeycomb structure. In the lower row of Figure 2 are shown the spatial distributions of SiO_2, amorphous carbon and Si−C bonding by reconstructing the STEM-EELS spectrum images, using the energy window ranging from 107-120 eV (main peaks of Si $L_{2,3}$ in SiO_2), 4-8 eV (carbon $\pi-\pi*$ plasmon) and 103-106 eV (lower energy tail of Si $L_{2,3}$), respectively for the two observation directions. It is clearly characterized from the observations that the MPCS nanocomposite consists of honeycomb silica framework with the inner walls of the nano-pores covered by amorphous carbon, and Si-C bonding is formed along the interfaces between the two phases. The relation between the nanostructures and the white PL is discussed.

References

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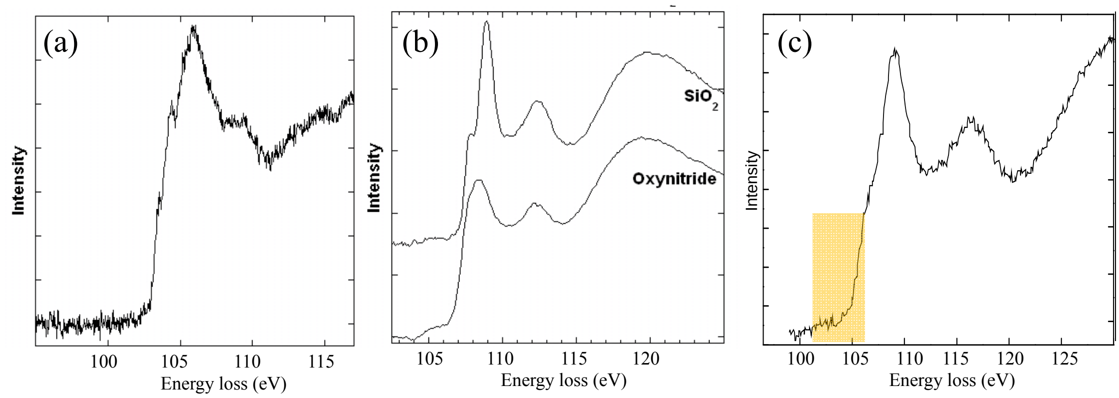

FIG. 1 Si $L_{2,3}$ ELNES from (a) amorphous SiC, (b) SiO_2 glass and silicon oxynitride and (c) the present MPCS nanocomposite. The weak intensities are highlighted in (c). See text for detail.

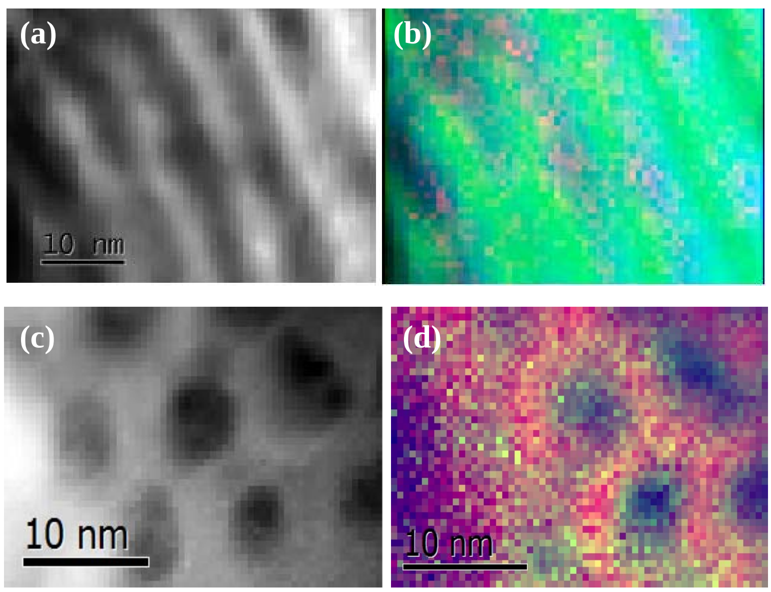

FIG. 2 (a, c) HAADF-STEM images observed along the directions perpendicular (a) and parallel (c) to the principal axis of the MPCS honeycomb structure. (b) Spatial distributions of SiO_2 (green), surface of silica wall (blue) and amorphous carbon (red) by reconstructing the STEM-EELS SI, using the energy window ranging from 20-25 eV (volume plasmon of silica), 15-18 eV (surface plasmon of silica) and 4-8 eV (carbon $\pi-\pi*$ plasmon), respectively. (d) Spatial distributions of Si-O_x (red) and Si−C (green) bonding states, using the energy window ranging from 107-120 eV (main peaks of Si $L_{2,3}$ in SiO_2), and 103-106 eV (lower energy tail of Si $L_{2,3}$), respectively.