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Mössbauer studies on core-shell Fe_{1.5}@Pt/C nanoparticles post-heated in NH₃ gas atmosphere

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Fe_{1.5}@Pt/C core-shell is used as an electrode catalyst for fuel cells, and research has been conducted to reduce costs and improve performance for application to fuel cells. In this study, the structural and magnetic properties of the Fe_{1.5}@Pt/C core-shell heat treated in an NH₃ gas atmosphere were investigated.

The Fe_{1.5}@Pt/C core-shell was prepared using the sonochemical method. Fe(acac)₃, Pt(acac)₂, and carbon support (Vulcan XC-72R) were dispersed in ethylene glycol. The mixture was placed in a girb0type sonicator and then it was irradiated over 3 h. To remove residual ethylene glycol, the obtained black slurry was filtered and washed with ethanol and DI water. The sample was dried in a vacuum oven at 70 °C for 12 h. Then, the post-heat treated Fe_{1.5}@Pt/C samples in an NH₃ gas atmosphere were annealed at 510 °C for 2 h. To remove iron oxides and other residues from the heat-treated sample, the sample was acid-treated in 0.1M HCIO₄ at 85 °C for 2 h. In order to obtain a clear core-shell structure, the sample was additionally annealed at a temperature of 300 °C in an H₂/N₂ atmosphere for 2 h. And finally, Fe_{1.5}@Pt/C with a core-shell structure was obtained. The sample was denoted as Fe_{1.5}@Pt/C NH₃.

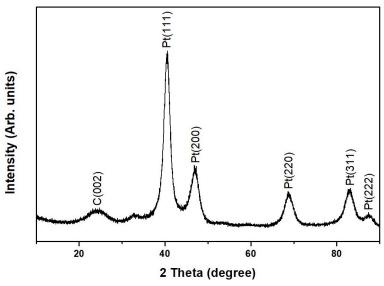


Fig. 1. XRD patterns of Fe_{1.5}@Pt/C NH₃

The crystallographic properties and core-shell structure of the Fe_{1.5}@Pt/C_NH₃ core-shell were confirmed through X-ray diffraction (XRD), transmission electron microscopy (TEM), and scanning transmission electron microscopy—energy dispersive spectroscopy (EDS). In addition, in order to investigate the magnetic properties of

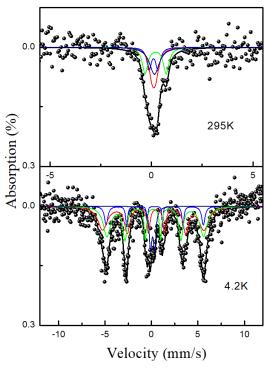


Fig. 2. Mössbauer spectra of Fe_{1.5}@Pt/C_NH₃ at 4.2 and 295K

the sample, the Mössbauer spectra were observed at 4.2 and 295 K and the M-H curve was measured using a vibrating sample magnetometer (VSM). Pt peaks were confirmed from the XRD patterns and the average crystallite sizes of the sample was confirmed to be 4.9 ± 1.4 nm using TEM image. The EDS line profile of Fe_{1.5}@Pt/C_NH₃ revealed a core-shell structure with a Pt skin layer (0.3 nm). The saturation magnetization and coercivity at 295 K were 7.1 emu/g and 134.5 Oe, respectively. The Mössbauer spectrum of the Fe_{1.5}@Pt/C_NH₃ at 295 K were analyzed with 4 sets of doublets and the electric quadrupole splitting (ΔE_Q) values were $\Delta E_{Q,1} = 0.44$ mm/s, $\Delta E_{Q,2} = 0.17$ mm/s, $\Delta E_{Q,3} = 1.04$ mm/s, and $\Delta E_{Q,4} = 0.07$ mm/s, respectively. Also, the spectrum at 4.2K was measured with 3 sets of sextet and one doublet and the magnetic hyperfine field (H_{hf}) values were analyzed as $H_{hf,1} = 325.2$ kOe, $H_{hf,2} = 339.7$ kOe, and $H_{hf,3} = 323.6$ kOe, respectively. From the isomeric shift values, all Fe valence states was determined to be ferric.