

## Synthesis of ferromagnetic hollow Fe-Fe<sub>3</sub>O<sub>4</sub> spheres

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Ferromagnetic hollow Fe-Fe<sub>3</sub>O<sub>4</sub> spheres are successfully synthesized using ultrasonic spray pyrolysis and the subsequent reduction process with controlled reduction conditions. The reduction temperature ranged from 340°C to 400°C. X-ray diffraction analysis was utilized to identify the phase. X-ray absorption near-edge structure of Fe *K*-edge, Fe *L*<sub>2,3</sub>-edge and O *K*-edge were investigated to reveal the electronic structure, in particular the oxidization state of iron. It demonstrated that the as-prepared hollow sphere was  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. As increasing the reduction temperature, a reduction of Fe<sub>2</sub>O<sub>3</sub> to Fe<sub>3</sub>O<sub>4</sub> occurs. Fe was observed as the reduction temperature reached 360°C. Both Fe<sub>3</sub>O<sub>4</sub> and Fe were found to present simultaneously. TEM investigations showed that the average size and shell thickness of hollow sphere was about 300 nm and 30 nm, respectively. Fe and Fe<sub>3</sub>O<sub>4</sub> nanoparticles distributed randomly within the sphere. At last, among the hollow Fe-Fe<sub>3</sub>O<sub>4</sub> spheres, the strongest ferromagnetism was obtained in hollow spheres after reducing at 380°C. The value of saturation magnetization is 108.6 emu/g, which is higher than that of the reported form of pure Fe<sub>3</sub>O<sub>4</sub> nanoparticle and microsphere.

***Keywords – hollow sphere, Fe K-edge, Fe L-edge, O K-edge, room temperature ferromagnetism***