

# Synthesis and characterization of NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> superparamagnetic hollow spheres

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We have synthesized a series of hollow NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> nanospheres to study the NiFe<sub>2</sub>O<sub>4</sub> particle size effect on their magnetic properties. In this work, we used the functional polymer poly(MMA-co-MAA) latex (500 nm) as a core template to prepare hollow spheres. The NiFe<sub>2</sub>O<sub>4</sub> nanoparticles were firstly deposited on the surface of the polymer sphere using the co-precipitation method. Then, the silica layer was coated on the PMMA/NiFe<sub>2</sub>O<sub>4</sub> core-shell spheres by the sol-gel method. To create silica-coated nickel ferrite hollow spherical structures, these hybrid PMMA/NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> core-shell spheres were subsequently calcined in the temperature range from 450 to 900 °C for 4h. X-ray diffraction pattern shows that the coated phase has a cubic spinel ferrite structure. The average crystallite sizes of the coated NiFe<sub>2</sub>O<sub>4</sub> nanoparticles, depending on the calcined temperature, are in the range from 2.1 to 8.9 nm. The scanning electron microscope (SEM) and transmission electron microscope (TEM) micrographs show that the hollow spheres are uniform and the thickness of the composite shell of NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> sphere is about 50 nm. Based on the thermogravimetric analysis (TGA), we find that the content of NiFe<sub>2</sub>O<sub>4</sub> is 83.1 wt% in the NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> composite shell. We have measured the magnetization  $\sigma(H)$  isotherms for all samples at room temperature. All hollow spheres exhibit the superparamagnetic behaviour. The spontaneous magnetization  $\sigma_s(300\text{ K})$  clearly decreased as the particle size decreased. This phenomenon can be interpreted as the effect of surface spin canting when the particle size is reduced. It is supposed that the complete silica network formed through sol-gel method based on hydrolysis and condensation, which in turn hinders the growth of NiFe<sub>2</sub>O<sub>4</sub> nanoparticles and results in an effective refinement of NiFe<sub>2</sub>O<sub>4</sub> particles.

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Fig. 1. Flow chart of synthesis and experiments for NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> hollow spheres.

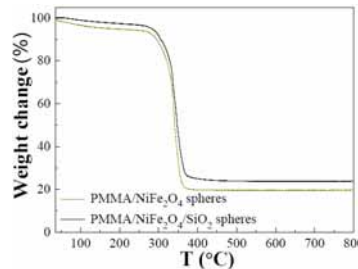


Fig. 2. TGA graphs of PMMA/NiFe<sub>2</sub>O<sub>4</sub> core-shell spheres and PMMA/NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> core-shell spheres. for magnetic hollow NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> spheres. The content of NiFe<sub>2</sub>O<sub>4</sub> is 83.1 wt% in the NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> composite hollow spheres.

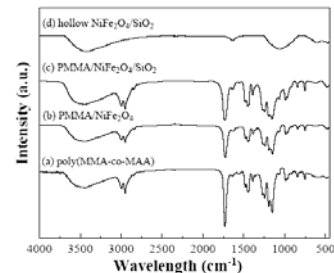


Fig. 3. FTIR spectra of ionic poly(MMA-co-MAA) polymer spheres, PMMA/CoFe<sub>2</sub>O<sub>4</sub> core-shell spheres, PMMA/NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> core-shell spheres and NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> hollow spheres (calcined at 450 °C).

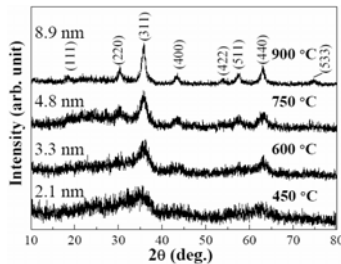


Fig. 4. XRD patterns of the NiFe<sub>2</sub>O<sub>4</sub>/SiO<sub>2</sub> hollow spheres calcined at various temperatures for 4 hrs. Crystallite sizes estimated by the Scherrer's formula.

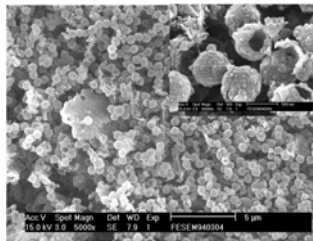


Fig. 5. SEM micrograph of hollow spheres created at 450°C

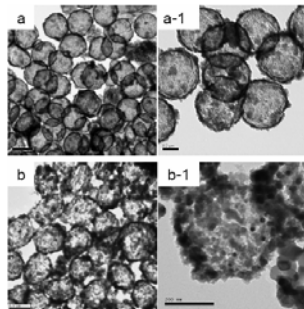


Fig. 6. TEM micrograph of hollow spheres created at (a) 450 °C and (b) 900 °C.

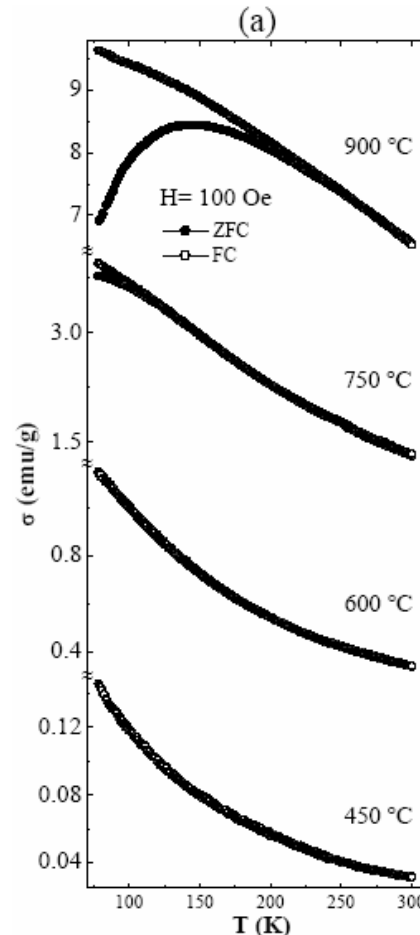
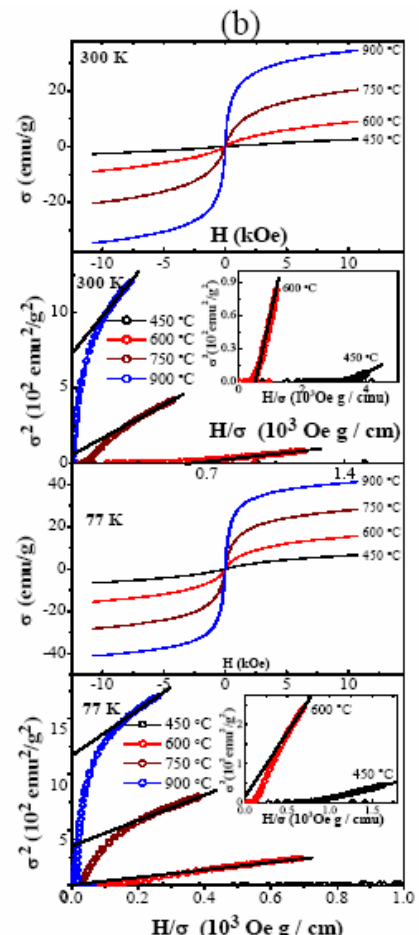


Fig. 7. (a) Temperature dependence of the ZFC and FC magnetization curves for SiO<sub>2</sub>/CoFe<sub>2</sub>O<sub>4</sub> hollow spheres prepared at various calcined temperatures. (b) Hysteresis loops and Arrott plots obtained at 300 K and 77 K for SiO<sub>2</sub>/CoFe<sub>2</sub>O<sub>4</sub> hollow spheres prepared at various calcined temperatures.



References:

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