Synthesis and In-situ Characterization of γ-Fe_2O_3 Nanoparticles Synthesized by Chemical Vapor Condensation Process Using Scanning Mobility Particle Sizer

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1. Introduction

Recently, γ-Fe_2O_3 nanoparticles which are the most widely used for recording media, have found wide applications in hyperthermia and drug delivery system (DDS). To date, a number of methods for the production of γ-Fe_2O_3 nanoparticles gas phase syntheses have been reported. However, it is very important to understand the relation between magnetic properties of nanopowder and particle characteristics, which are basically determined in the reactor during synthesis. In this sense, it is significant to investigate particle characteristics in-situ during the formation of γ-Fe_2O_3 nanopowder and its influence on the magnetic properties. In order to interpret those matters, chemical vapor condensation process (CVC) and scanning mobility particle sizer (SMPS) were used for synthesis and real-time particle characterization of mono-dispersed pure γ-Fe_2O_3 nanoparticles respectively, in this study.

2. Experimental

Precursors with different concentrations were prepared by dissolving Fe(III) acetylacetonate in isopropyl alcohol. γ-Fe_2O_3 nanoparticles were synthesized at 40 mbar and 1000 mbar for direct aspiration into SMPS via sampling probe. Then, size distribution of γ-Fe_2O_3 nanoparticles was measured by SMPS in real-time. Besides, phase and crystallite size of γ-Fe_2O_3 nanopowder were analyzed by XRD, and specific surface area of the particle was obtained by BET measurements. Particle shape and average particle size were analyzed by TEM observation. Finally, magnetic properties were measured by VSM.

3. Results and discussion

The average size of Fe_2O_3 nanoparticles increased with increase of reaction temperature from 700°C to 1000°C, which showed the sintering effect clearly. XRD result revealed that α and γ phases coexisted below 900°C but only γ phase over 900°C. Thus, this indicates that α phase transformed into γ phase over 900°C. Thus, it was shown that 900°C was the critical reaction temperature for synthesis of γ-Fe_2O_3 nanoparticles in CVC process. Also, particle size and degree of agglomeration were increased with increase of precursor concentration.

In-situ characterization by SMPS showed that particle size decreased and size distributions became more homogeneous below 50 nm in particle size with decreasing reaction temperature, pressure and precursor concentration. This results were due to the reduction of particle growth and degree of agglomeration. These results suggested that particle growth and number of agglomerates decreased by reducing process variables, which were confirmed and quantified theoretically by calculation of reaction factors and experimentally using XRD, BET, TEM and VSM.

4. Conclusion

γ-Fe_2O_3 nanoparticles were successfully synthesized by CVC process and their microstructure and properties were controlled by changing process variables. The relation between powder properties and process variables were quantified by calculating reaction factors for synthesis conditions of γ-Fe_2O_3 nanoparticles. Also, in-situ particle characterization by SMPS measurements showed their feasibilities of applications to various nanoparticles synthesized in other gas phase syntheses such as spray, flame, and pulsed wire evaporation.