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Magnetic Properties of Fe Nanoparticles Synthesized by Chemical Vapor Condensation

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1. Introduction

Research on nanomaterials has been stimulated by the interest for basic scientific investigations and their technological applications (1). Chemical synthesis of nanoparticles is a rapidly growing field with a great potential in making useful materials. Generally, chemical techniques are very versatile in that they can be applied to almost materials and can usually produce a large amount of materials. Fine particles can be synthesized using organic and organometallic reagents. In this paper, we synthesized Fe nanoparticles by Chemical Vapor Condensation (CVC) method from the precursor of iron carbonyl ($Fe(CO)_5$. The structural and magnetic properties of free-standing Fe particles were discussed.

2. Experimental Details

The basic setup for CVC is similar to that described in literature elsewhere (2). Carrier gases such as He, Ar, CO, O_2, etc. are fed through a heated bubbling unit containing the liquid precursor. A tubular furnace provides a heat source for the controlled decomposition of the precursor. The flow of the carrier gas entrains precursor vapor and pass through the heated furnace in which the precursor pyrolyzes and condenses into clusters or particles that impinge upon the liquid nitrogen cooled rotating chiller from which powders can be scraped off and collected. Experiments were conducted with a tubular furnace uniformly heated at a temperature over the range 400 – 1100°C. Fe nanoparticles were synthesized by the pyrolysis of $Fe(CO)_5$. Characterization and magnetic properties of Fe nanoparticles were investigated by HRTEM, X-ray diffraction, VSM, BET, Mössbauer spectroscopy, etc.

3. Results and Discussion

Spherical particles of different sizes within the nanometer range and very uniform in size were obtained by CVC. During the fabrication processes, the precursor of iron carbonyl can be easily transported into a bubbler by a micro-pump due to its liquid state at room temperature. Vaporization of liquid $Fe(CO)_5$ was realized in the bubbler at 150°C and subsequently passed into the heated furnace by the carrier gas for a decomposition. The minimum decomposition temperature for $Fe(CO)_5$ is about 350°C. Powders were well deposited on the surface of liquid nitrogen-cooled chiller in the work chamber. After passivating, the as-prepared powders were collected.

High Resolution Transmission Electron Microscopy (HRTEM) results show that the Fe particle shape is nearly spherical with a core-shell type structure. The core is metallic Fe and the shell is composed of crystalline Fe oxides. By adjusting the carrier gases, the decomposition temperature and the cooling of powder, its particle size and microstructure can be controlled. As a result, it can be realized to control the magnetic states of particles for the specific applications. Fig. 1 shows X-ray diffraction patterns of Fe nanoparticles, which were synthesized at the decomposition temperature of 1000 °C with carrier gas of He (sample (a)), mixed carrier gas of (Ar + O_2) (sample (b)) and carrier gas of He without liquid nitrogen-cooling (sample (c)). Fig. 2 shows Mössbauer spectra of above samples at room temperature. It is illustrated that sample (a) exhibits superparamagnetism, sample (c) is a distinct ferromagnetism and sample (b) is a mixture of two kinds of magnetic states. Saturation magnetizations, coercivities, oxygen contents and the mean

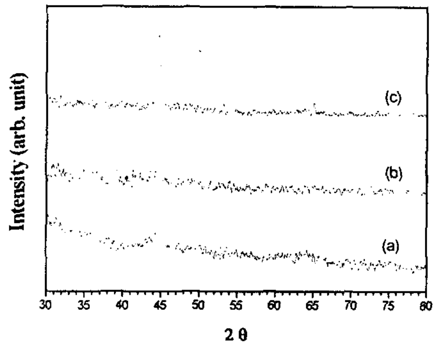

Fig. 1. X-ray-diffraction patterns of Fe nanoparticles samples (a), (b) and (c).

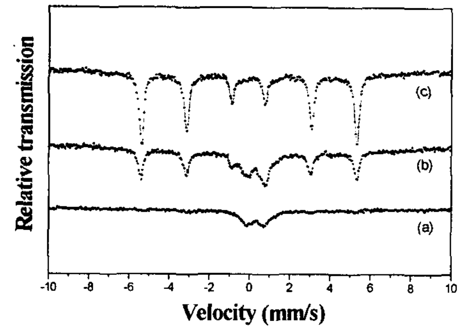

Fig. 2. Mössbauer spectra at room temperature for Fe nanoparticles.

Table 1. Saturation magnetization (σ_S), coercivity (H_C), oxygen content and mean particle sizes for Fe nanoparticles samples (a), (b) and (c).

Samples	Magnetization σ_S (Am^2/kg)	Coercivity H_C (Oe)	Oxygen content (wt.%)	Mean particle size (nm)
(a)	32.49	176.0	30.8	7.6
(b)	81.69	494.4	26.8	10.3
(c)	153.60	591.7	10.1	12.3

particle sizes of Fe nanoparticles samples (a), (b) and (c) are displayed in Table 1 for comparison.

4. Summary

Magnetic Fe nanoparticles were synthesized by CVC process using $Fe(CO)_5$ as precursors. The nanoparticles have core-shell structures with uniform dispersion. For the specific purposes, the micostructures as well as the magnetic states of Fe nanoparticles can be controlled by adjusting the process parameters, such as the carrier gases, the decomposition temperature, the cooling of powder, etc.

References

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