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화학기상응축법에 의한 Fe-Co 나노입자의 제조 및 특성

Preparation and properties of iron-cobalt alloyed nanoparticles by chemical vapor condensation process

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1. Introduction

Recently, research on the nanoparticles has attracted more attention because of their special characterization. The motivations of studies in this area are from both fundamentally scientific understandings of the physical properties and their technical applications in various fields [1].

Various physical, chemical and mechanical methods [2,3] have been used to synthesize the nanoparticles. The chemical synthesis of the nanoparticles is rapidly grown with grea potential in preparing the nanoparticles.

In this work, magnetic Fe(Co) alloyed nanoparticles are synthesized by chemical vapor condensation (CVC) process using the iron carbonyl ($Fe(CO)_5$) and cobalt carbonyl ($Co_2(CO)_8$) as precursors under flowing helium atmosphere at different temperatures.

2. Experimental Details

A carrier gas of high purity helium was fed through heated bubbling units containing the precursors. A tubular furnace provided a heat source for the decomposition of the precursors. The flow of the carrier gas entrained the precursors vapor and passed through the heated furnace and impinged on the liquid nitrogen cooled rotating chiller. Then the powders were scraped from the chiller.

X-ray diffraction (XRD) with CuK radiation was performed to identify the phases of as-prepared particles. The structure of the particles was determined by analyzing the high-reselution transmission electron microscopy (HRTEM) images. Magnetization was measured by a vibrating sample magnetometer (VSM) at room temperature in a field up to 20 kOe.

3. Results and Discussions

HRTEM micrograph (Fig.1) shows the typical morphology of passivated Fe-Co alloyed nanoparticles. The shape of the powders is nearly spherical with a core-shell structure. The black core is metallic and the light shell is metal-oxides. Magnetic Fe(Co) alloyed particles can form the intricate long strands when they agglomerate in order to minimize the magnetostatic energy.

The characterization of finally formed particles are affected by the preparation parameters, such as the flow gas rate of the carrier gas, the gas pressure in the work chamber, the decomposition temperature of the precursors, etc. Experiments show that particles formed at the higher pressure have a large size; particle size also increases with the increasing of

decomposition temperature. When the decomposition temperature changed from 400℃ to 900℃, the particle size changed from 11.8 nm to 18 nm at 200 mTorr, however, the particle size changed from 4.3 nm to 7 nm at 1 mTorr.

Experiment shows that the gas flow rate influenced the size of the particles. Table 1 shows clearly that both the particle size and the Co content are enhanced by increasing the gas flow rate. The particle size (Tab.1) determined by BET assuming of spherical shape ranges from 4.8 nm to 11 nm, in contrast, the particle size determined by TEM ranges from 6 to 14 nm. The systematic error in particle size calculated from BET surface area is caused by some surface relief and by imperfections in shape of nanoparticles.

Fig. 2 shows the Co content dependence of the magnetic properties of the Fe(Co) alloyed nanoparticles. With increasing Co content the saturation magnetization increases and reaches its maximum at near 40 wt Co, and then decreases (Fig.2 (a)) and coercivity continuously increases (Fig.2 (b)).

Fig.1

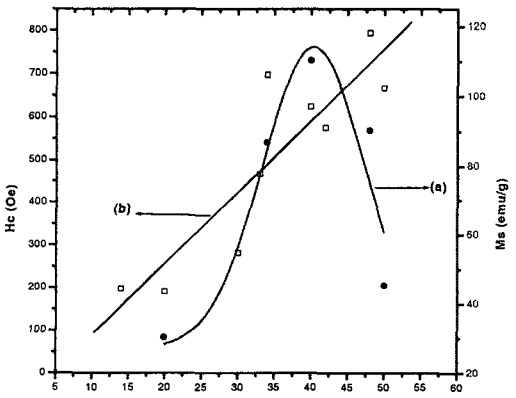

Fig.2

Table. 1

Gas flow rate (sccm)	Specific area (cm^2/g)	Size (nm)		Co content (wt.%)
		TEM	BET	
10	213.8	6±1	4.8	16
30	138.3	10±1	7.6	33
50	106.3	12±1	9.2	38

4. Conclusions

The Fe(Co) alloyed nanoparticles have been successfully synthesized by chemical vapor condensation (CVC) process. By controlling the preparation parameters, the Fe(Co) alloyed

nanoparticles with various content are obtained. In the CVC-prepared Fe(Co) alloyed nanoparticles, the core consists of metallic, while the shell is composed of metal oxides. Mean size of the Fe(Co) alloyed nanoparticles increases with increasing the decomposition temperature of precursor vapor, pressure in working chamber and carrier gas flow rate. The trace of FCC phase can be observed in nanoparticles at the Co content is more than 40wt.%. The variations in cobalt content strongly influence the saturation magnetization and the coercivity of nanoparticles.

References

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