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화학기상응축법에 의한 Fe/N 나노입자의 성질 및 특성
(Characteristics and properties of Fe/N 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. There is a growing interest in nanoscale metal and nitride particles for potential application as catalysts, magnetic recording, ferrofluids [1,2].

The unique magnetic properties and high resistance to corrosion of Fe nitrides have attracted much attention for applications. Various physical, chemical and mechanical methods [3,4] have been used to synthesize the iron nitrides nanoparticles. The chemical synthesis of the nanoparticles is rapidly grown with great potential in preparing the nanoparticles.

In this work, composite Fe/N nanoparticles were fabricated by the chemical vapor condensation (CVC) method, using the organmetallic of iron pentacarbonyl ($Fe(CO)_5$) and NH_3 as precursors under Ar atmospheres at 500°C.

2. Experimental details

The carrier gases of ammonia and Ar were fed through heated bubbling units containing the precursor of $Fe(CO)_5$. The tubular furnace provided a heated source for decomposition of the precursor vapor and ammonia. The flow of the carrier gases entained the precursor vapor and passed through the heated furnace and impinged into the cooling chamber. All nanoparticles were deposited in Ar atmosphere at room temperature. Then the powders were collected from the work chamber.

X-ray diffractometry (XRD) with CuK_α radiation was performed to identify the phases of as-prepared particles. The structure of the particles was determined by high-resolution transmission electron microscopy (TEM) images. The composition of the surface layers of the particles is determined by XPS. Magnetic properties were measured by vibrating sample magnetometer (VSM) at room temperature in a field up to 10 kOe.

3. Experimental results and discussions

HRTEM micrograph (Fig. 1) shows the typical morphology of composite γ'-Fe_4N/α-Fe nanoparticles. The shape of the powders is nearly spherical with a core-shell structure. The core consists of Fe/N nanoparticles and the shell is iron oxide. Magnetic Fe/N nanoparticles can form intricate long chains when they agglomerate in order to minimize the magnetostatic energy.

Fig. 2 shows XRD patterns of nanoparticles prepared using the carrier gases of 10sccm NH_3 mixed with (a) 100sccm, (b) 150sccm and (c) 185sccm Ar gas at 500°C. As decreasing the NH_3 concentration, composite iron nitrides nanoparticles including Fe_3N, γ'-Fe_4N and α-Fe phases can be produced.

The binding energy of $Fe2p_{3/2\ (1/2)}$ electrons in the compacted composite γ'-Fe_4N/α-Fe sample is determined by analyzing the XPS results. Fig.3 represents that the iron oxides exist on the surface of as-prepared sample due to a much higher affinity of Fe for O than N. According to the analysis of binding energies, the iron oxides are FeOOH and Fe_3O_4.

Usually, magnetic properties reduce with decreasing the particle size of nanoparticles. However, no strong size dependence of magnetic performance of these composite Fe/N nanoparticles is found in this work. The magnetic properties of these composite Fe/N nanoparticles changed depending on the component transformation and

microstructure as shown in Fig. 4. With the amount of Fe_3N and γ'-Fe_4N reducing and that of α-Fe arising, the coercivity of these nanoparticles increase from 612.7Oe for (a) sample to 895.1Oe for (b) sample and 975.7Oe for (c) sample, while the saturation magnetizarion increase from 11.47emu/g for (a) sample to 21.24emu/g for (b) sample, but decrease to 17.47emu/g for (c) sample due to iron oxide formation.

4. Conclusions

The composite Fe/N nanoparticles have been successfully synthesized by chemical vapor condensation (CVC) process. By controlling the concentration of NH_3, Fe_3N and γ'-Fe_4N and α-Fe phases can be obtained. The as-prepared particle has a nearly spherical shape with a grain size of 15-30nm consisting of core-shell type structure. The core consists of composite Fe/N nanoparticles, and the shell is composed of the iron oxides, which can resist further oxidation of the core. The magnetic properties of these composite Fe/N nanoparticles change depending on the component transformation and microstructure

5. References:

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[2] L. N. Lewis, Chem. Rev. 93 (1993) 2693.
[3] W. A. Kaczmarek, Scr. Metall. Mater. 33, (1995) 1687.
[4] X. Bao, R. M. Metzger, and W. D. Doyle, J. Appl. Phys. 73, (1993) 6734.

Fig. 1 HRTEM of composite γ'-Fe_4N/α-Fe nanoparticles

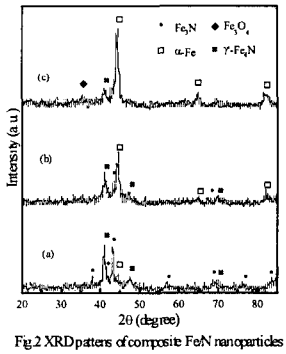

Fig.2 XRD patters of composite Fe/N nanoparticles prepared with different NH_3 concentration at 500°C

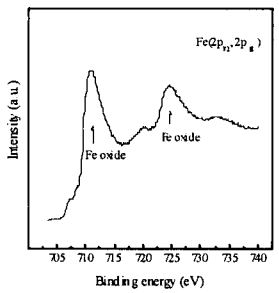

Fig.3 Fe(2 $p_{3/2,1/2}$) x-ray photoelectron spectra of the as-prepared composite γ'-Fe_4N/α-Fe nanoparticles

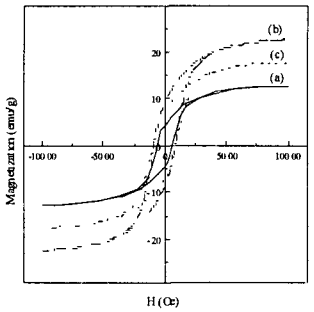

Fig.4 Hysteresis loop of composite Fe/N nanoparticles measured at room temperature