

화학기상응축법에 의한 Fe/N 나노입자의 제조 및 특성 Preparation and properties of Fe/N nanoparticles by chemical vapor condensation process

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1. Introduction

There is growing interest in nanoscale metal and nitride particles for potential application as catalysts, sensors, electromagnetic shielding, magnetic recording, ferrofluids, and refrigeration [1,2]. In Fe-N system, Fe_4N , Fe_3N , and Fe_2N are known to be stable magnetic compounds of iron nitrides. The unique magnetic properties and high resistance to corrosion of these iron nitrides have attracted much attention in research of materials based on iron nitrides for magnetic applications. In this work, single Fe_3N nanoparticles were fabricated by the chemical vapor condensation (CVC) method, using the organometallic of iron pentacarbonyl ($\text{Fe}(\text{CO})_5$) and NH_3 as precursors under air/Ar atmospheres at different temperatures.

2. Experimental details

Iron nitrides nanoparticles were made by the chemical vapor condensation (CVC) process similar as that in the previous work [3]. To produce iron nitrides nanoparticles, a carrier gas of high purity NH_3 is fed through a heated bubbling units containing the liquid iron pentacarbonyl ($\text{Fe}(\text{CO})_5$) precursor. The flow of pure NH_3 or mixed with Ar as a carrier gas entraining $\text{Fe}(\text{CO})_5$ vapor passed through the heated tubular furnace into the work chamber. The precursors of $\text{Fe}(\text{CO})_5$ and NH_3 decomposed in that furnace in the range of 400-600C and condensed in clusters or nanoparticles. All nanoparticles were deposited in the work chamber at room temperature, from which powders can be collected.

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 analyzing transmission electron microscopy (TEM) images. Magnetic properties were measured by vibrating sample magnetometer (VSM) at room temperature in a field up to 10kOe.

3. Experimental results and discussions

Fig. 1 shows XRD patterns of nanoparticles prepared with different flow rate of NH_3 at 500°C . With a higher concentration of NH_3 , single Fe_3N is synthesized. When decreasing the NH_3 concentration, $\gamma\text{-Fe}_4\text{N}$ and $\gamma\text{-Fe}$ phases can be produced. The experimental conditions and phases are list in table 1.

TEM micrograph (Fig. 2) shows the morphology of Fe_3N nanoparticles, which are nearly spherical with a core-shell structure. Magnetic Fe/N nanoparticles form intricate long chains to reduce the magnetostatic energy.

The particle size dependence of the magnetic properties of the single Fe_3N nanoparticles was shown in Fig. 3. With increasing particle size, the saturation magnetization continuously increases (Fig. 3(a)), while no strong size dependence of coercivity (H_c) is found (Fig. 3(b)), similar to the results shown in [4].

4. Conclusions

The Fe/N nanoparticles have been successfully synthesized by chemical vapor condensation (CVC) process. By controlling the concentration of NH_3 , single Fe_3N and $\gamma\text{-Fe}_4\text{N}$ and $\gamma\text{-Fe}$ phases can be obtained. Saturation magnetization of Fe/N nanoparticles is a function of grain size. However, no distinct size dependence of H_c was found for these nanoparticles.

References:

- [1] S.T. Oyama, J. Solid-State Chem. 96 (1992) 442.
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- [3] C.J. Choi, X.L. Dong and B.K. Kim, Script Mater. 44 (2001) 2225.
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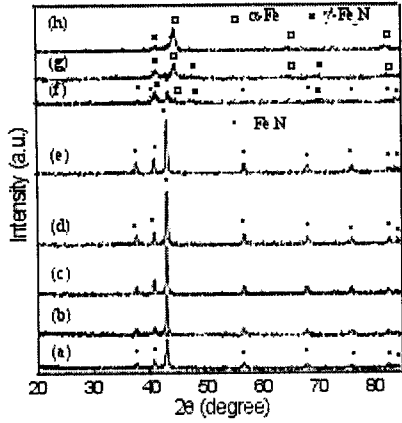


Fig.1 X-ray diffraction patterns of Fe/N nanoparticles prepared with different NH_3 concentration at 500°C



Fig. 2. TEM micrograph of Fe_3N nanoparticles

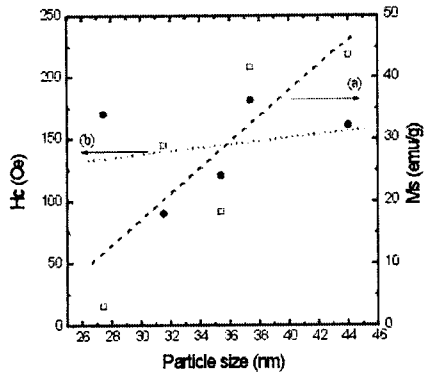


Fig.3 Variation of magnetic properties with particle size of Fe/N nanoparticles at room temperature

Sample	Flow rate (sccm)			Decomposition temperature (°C)	Chamber atmosphere (RT, 1bar)	Phase
	NH_3	Ar	$Fe(CO)_5$			
(a)	60	0	0.5	500	Air	Fe_3N
(b)	30	0	0.5	500	Air	Fe_3N
(c)	10	0	0.5	500	Air	Fe_3N
(d)	10	20	0.5	500	Air	Fe_3N
(e)	10	30	0.5	500	Air	Fe_3N
(f)	10	100	0.5	500	Air	Fe_3N , Fe_2N , $\alpha-Fe$
(g)	10	150	0.5	500	Air	Fe_3N , Fe_2N , $\alpha-Fe$
(h)	10	185	0.5	500	Air	Fe_3N , $\alpha-Fe$

Table 1