Production and Characteristics of Fe/SiO_2 Nanopowders by Chemical Vapor Condensation Process

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1. Introduction

Researches on nanopowder materials with size of 1~100nm range have been intensively carried out in recent decades, because it has unique properties such as high catalysis and electromagnetic properties and high mechanical performance[1].

The nanostructured powders have been fabricated by various processes such as inert gas condensation process(IGC), plasma arc discharge(PAD), mechano-chemical process(MCP), and chemical vapor condensation(CVC). Among them, the CVC process is very attractive method because it can produce the nano composite or coating powders with high purity and non-agglomeration properties as well as grain size of <30nm[2]. CVC process has an advantage applicable to almost all materials because wide metal-organic precursors are available and it can also produce a large amount of nanopowders. The size of CVC powders are mainly affected by reaction temperatures, and the phase and composition are dependent on as carrier gases or reaction atmospheres.

In this study, we synthesized Fe, SiO_2 and Fe/SiO_2 nanopowders by chemical vapor condensation process by using of metal-organic precursors. Effects of the reaction parameters on the phase, microstructure and size of as-synthesized nanopowders were investigated.

2. Experimental Procedure

The CVC apparatus are similar with hot-wall reactor type furnace. Basic setup of the CVC adapted in this experimental is shown in Fig 1. Vaporization temperatures were optimized at 100℃. High-purity carrier gases fed through a heated bubbling unit containing the ironcarbonyl($Fe(CO)_5$) and TEOS(Tetraethyl-orthosilicate, $C_8H_2OO_4Si$) precursors. Nanopowders was produced under 1 atm. The CVC experiment was conducted at temperature range of 500~1100℃.

X-ray diffraction(XRD) with monochromatic CuKa radiation was performed to identify the phases existing in as-prepared powders. Chemical composite of CVC powders was examined by ICP-MS. The powder size was calculated by Scherrer equation from the half width of XRD peaks and BET specific surface area. The morphology and powder size were also analyzed by field-emission scanning electron microscope(FE-SEM, Philips) and transmission electron microscope(JEM-2000FXII). The powders for TEM investigations were ultrasonically dispersed in ethanol and dropped on a carbon-coated copper grid.

3. Results

By TGA analyses, the iron pentacarbonyl ($Fe(CO)_5$) was decomposed from $Fe(CO)_5$ to Fe + 5CO in the temperature range of 100~300℃. Also, SiO_2 nanopowders were produced from

700℃. No SiO_2 powders were obtained under 500℃. In order to investigate the effect of reaction temperature, several Fe/SiO2 powder samples were synthesized in various temperature range of 500~1100℃ as shown in Fig. 2. According to the analysis of XRD patterns, we knew that the powder produced at 700℃ had very broad peaks. With increasing reaction temperature, the XRD peaks were very clear.

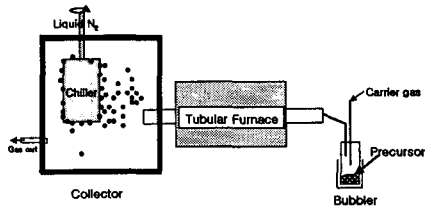
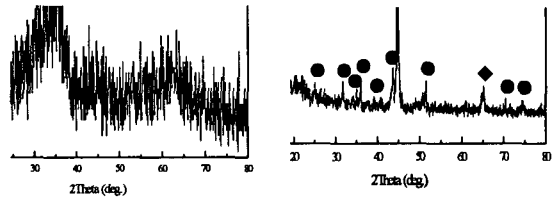


Figure 1. Schematic of CVC process.



(a) 700℃ (b) 1100℃

Fig. 2. XRD traces of the CVC powders under different temperatures.

TEM observation showed that the CVC Fe powders had intricate long stand structure because of intrinsic magnetic properties of Fe, but SiO_2 showed aggregates that consisted of point contacts of powders. The size of the CVC powders varied from 10nm to 40nm. The CVV powders had very smooth surface morphology and round shape.

4. Conclusions

Fe, SiO_2 and Fe/SiO_2 nanopowders were prepared by chemical vapor condensation process by using of metal organic precusors. Different Fe, SiO_2 and Fe/SiO_2 nanopowders were synthesized with changes of the reaction temperatures. Grain size of Fe/SiO_2 and SiO_2 powder was increased with increasing of reaction temperatures.

Acknowledgement

This research was supported by a grant (code #: 04K1501-01210) from 'Center for Nanostructured Materials Technology' under '21st Century Frontier R&D Programs'of the Ministry of Science and Technology

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