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# Characteristics of Fe/SiO<sub>2</sub> nanocomposite powders by the chemical vapor condensation process

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### Abstract

Fe/SiO<sub>2</sub> nanocomposite powders were produced by the chemical vapor condensation (CVC) process. Characteristics of the as-CVCed powders were investigated by XRD, TEM and VSM, etc. The Fe/SiO<sub>2</sub> powders could be sytheised from 700 °C. With increasing reaction temperature, the *a*-Fe phase peaks in the XRD became clearer. The size of CVCed powders was about 50 nm at 1100 °C. The powders showed intricate long-stand structure due to their magnetic characteristics. TEM results revealed that the Fe powders were covered by SiO<sub>2</sub> layer fully or partially depending on the experimental condition. The saturation magnetization and the coercive force of the as-CVCed powders were investigated with the decomposition temperature in this study.

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Keywords: Nanocomposite; Chemical vapor condensation; Magnetic property

## 1. Introduction

Recently, nanoparticles or nanocomposite powders with the size of 1–100 nm have been intensively investigated because of their wide range of potential applications in areas such as electronics, optics, catalysis, ferrofluids and magnetic data storage [1–3]. The nanocomposite powders could be fabricated by various methods such as inert gas condensation process (IGC), plasma arc discharge (PAD)[4] and chemical vapor condensation (CVC)[5]. Each fabrication technique has its own set of advantages and disadvantages and some limitations. However, as compared with other methods, the CVC process has been developed for preparation of almost all kinds of materials because a wide range of precursors are commercially available, and it can easily produce the nanocomposite or coating powders with high purity and non-agglomeration properties.

In the present study, we produced  $Fe/SiO_2$  nanocomposite powders by the CVC process using the metal–organic precursors. The effects of reaction parameters on phases, microstructures and magnetic properties of the as-synthesized nanocomposite powders were systematically investigated.

#### 2. Experimental

The basic setup for CVC is similar to that described in literature elsewhere [6]. Fe/SiO<sub>2</sub> nanocomposite powders were prepared using the iron pentacarbonyl (IP, Fe(CO)<sub>5</sub>) and the tetraethyl-orthosilicate (TEOS,  $C_8H_{20}O_4Si$ ) as precursors under flowing Ar atmosphere at different temperatures. Two bubblers were used in this work because the IP and TEOS have different physical and thermochemical properties. Flow rate of Ar carrier gas was fixed at the level of 600 cm<sup>3</sup>. Feeding ratio of IP to TEOS was kept with 9:1. A tubular furnace provided a heat source for the decomposition of precursors. The flow of the carrier gas entrained precursor vapor and passed through the heated tubular furnace in which the precursor is decomposed and condensed into clusters or powders. The synthesized powders were scraped off and collected from the chiller cooled by cold water. The CVC experiments were conducted at 700–1100 °C.

X-ray diffraction (XRD) with monochromatic Cu K $\alpha$  radiation ( $\lambda$  = 0.1542) was performed to identify the phases existing in as-prepared CVC powders. The morphology, size and lattices images of the powders were analyzed by high-resolution transmission electron microscopy (HR-TEM, JEOL) operated at 200 kV. Magnetic properties were measured using a vibrating sample magnetometer (VSM) at room temperature in a field up to 20 kOe.

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Fig. 1. XRD patterns of the as-prepared the CVC powders.

# 3. Results and discussions

By TGA analyses, the iron pentacarbonyl (Fe(CO<sub>5</sub>)) was decomposed from Fe(CO)<sub>5</sub> to Fe+5CO in the temperature range of 140–300 °C. Therefore, the liquid Fe(CO)<sub>5</sub> precursor was kept at the 140 °C to vaporize in evaporator. And the TEOS was vaporized from its liquid state at 103 °C in evaporator and fed into reaction furnace.

By the analysis of XRD patterns as shown in Fig. 1, we knew the nanopowders could be produced above 700 °C. No powders were obtained under 700 °C in this experiment. Fully amorphous halo pattern was found at 700 °C. With increasing reaction temperature, the XRD peaks became clearer. At 1100 °C, very sharp bcc Fe (*a*-Fe) peaks were detected, and week Fe<sub>2</sub>SiO<sub>4</sub> peaks were also traced, that means Fe and Si elements were reacted at this temperature. Amorphous SiO<sub>2</sub> phase was confirmed at near 22° of  $2\theta$ . No visual Fe oxide peaks were observed in this study. Fig. 2 showed the SEM micrographs of the CVC powder produced at 1100 °C. It showed the intricate long stand structure because of intrinsic mag-



Fig. 2. SEM micrographs of powder prepared at 1100 °C.

netic properties of Fe and agglomeration of the nanopowders itself.

The size of the as-CVCed powders was increased with increasing the reaction temperature as shown in Fig. 3. The size of the powders produced at 1100 °C was about 50 nm. In Fig. 3(a) shows that the CVC powders produced at 700 °C had amorphous phase. However, in Fig. 3(b), the image reveals that the Fe/SiO<sub>2</sub> nanopowders produced at 1100 °C had crystalline phase and were composed of core and shell. The shell thickness of the CVC powders was about ~5 nm.

In Fig. 4, TEM results revealed that the shell thickness of the Fe composite powders produced at 1100 °C was different with the surface position. As indicated by the point 2, the thickness of this part was about only 5 nm. The layer was composed with Si, O and C as shown in the micro-EDS (point 2), which was well matched with XRD results. In this figure, the shell showed random structure, i.e., amorphous phase. In fact, carbon or carbide was not detected in the XRD results. However, in point 2, we could find the crystalline carbon layer. The core phase was pure iron as shown in micro-EDS result of the point 1.

Fig. 3. TEM micrographs of the as-prepared powders: (a) 700 °C and (b) 1100 °C.



Fig. 4. (a) HR-TEM images and (b) micro-EDS results of the CVC powders produced at 1100 °C.

In this study, the CVC was performed under noble Ar atmosphere. As mentioned above, however, the carbon layer was found by the micro-EDS results. Carbon coating of Fe powders could occur during CVC process by the dissociation reaction of CO from precursors, Fe(CO)<sub>5</sub>. Generally, under the high temperature conditions and in the presence of metallic catalyst (Fe, Ni, Co, etc.), 2CO gas could be decomposed into  $CO_2 + C$  and react with metallic catalysis [7]. The chemical reaction, which is expected to take place, is as follows with increasing reaction temperature.

Pyrolysis of Fe(CO)<sub>5</sub>:

 $Fe(CO)_5 \rightarrow Fe + 5CO, T = 140-300 \,^{\circ}C$  (1)

CO gas dissociation:

 $Fe + 5CO \rightarrow Fe + \frac{5}{2}C + \frac{5}{2}CO_2, \quad T = 500 - 1100 \,^{\circ}C$  (2)

Pure iron formation or carbon coating:

$$Fe + C + CO_2 \rightarrow Fe + Fe(C)_{coating} + CO_2\uparrow,$$
  
$$T = 1100 \,^{\circ}C \tag{4}$$

From the XRD results of the Fe nanocomposites prepared at different temperatures, it is concluded that the presence of Fe influences the saturation magnetization of the Fe nanocomposites. Although the particles size increased with the reaction temperatures, the phase composition is the main reason for the increasing in the saturation magnetization. With the increase of reaction temperature, the saturation magnetization and coercivity increased from 3 emu/g and 37 Oe to 23 emu/g and 410 Oe,

respectively. The existence of pure Fe leads to increase in the saturation magnetization and coercivity of the  $Fe/SiO_2$  nanocomposites.

#### 4. Conclusions

In conclusion, the Fe/SiO<sub>2</sub> nanocomposite powders were successfully fabricated by the chemical vapor condensation process carried out in Ar atmospheres. The phase in the Fe nanocapsules was affected by the decomposition temperatures. The Fe/SiO<sub>2</sub> nanocomposite powders consisted of SiO<sub>2</sub> shell and Fe metal core. The size of the Fe nanocomposite was about 50 nm at 1100 °C, and it showed intricate long stands structure because of intrinsic magnetic properties of Fe. The saturation magnetization of the Fe/SiO<sub>2</sub> nanocomposite powders were increased with the increasing the reaction temperature because of the existence of Fe phase.

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