

# **AEROSOL / PARTICULATE REACTORS**

## **I-25. Aerosol Routes for Synthesis of Nanostructured Magnetic Oxides: Characterization and Transport Behavior**

### **A. Problem Definition**

Small magnetic particles have drawn considerable attention due to wide range of innovative uses including recording media, pigments, magnetic fluids, and biomedical applications. Several preparation routes have been investigated for synthesis of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> including thermal decomposition of metal hydrazine complexes, wet chemical synthesis (Ennas *et al.* 1999), successive hydrolysis, and thermal decomposition of metal carboxylate. However, most of the conventional methods are rather complex, usually involving several steps and efforts have been made to establish direct preparation routes of these magnetic particles. Gas phase reaction and pyrolysis of dispersed droplets of Iron(III) compounds have been considered to be a promising approach for the preparation of nanoscale iron oxide particles. Earlier studies have shown that the iron oxide phase in such reactions depend on the preparation methods. Grimm *et al.* (1997) have studied the formation of  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> using the flame pyrolysis of Iron pentacarbonyl. However, detailed studies on the morphology and magnetic properties of synthesized  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> using flame pyrolysis have not been made.

### **B. Research Objectives**

In this study, effects of flame temperature profile, residence time have been investigated on the shape, size, and crystallinity of synthesized  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>. Flame combustion of ferrocene and ferric nitrate has also been investigated to study the mechanism of formation of the magnetic oxide.

### **C. Research Accomplishments**

The reactor configuration for this work was a non-premixed laminar methane/oxidizer flame as a reaction environment. The Schematic diagram of the experimental setup is shown in Figure (1).

Iron pentacarbonyl vapors were obtained by bubbling nitrogen gas through an impinger, maintained at 0°C isotherm by using melting ice around the container. Iron pentacarbonyl was added through the center port of the burner and mixed with fuel and oxidizer at the nozzle interface where the reaction takes place.

Flame temperature was measured using R-type thermocouple. In-situ characterization of particles was done using Scanning mobility particle sizer (Model 3080, TSI Inc.) to determine the size distribution and number concentration of particles. Ex-situ characterization methods of particles included: X-ray diffraction ( $CuK_{\alpha}$  radiation, DMax, Rigaku), Vibrating sample magnetometry (Model 4500, ES & G Princeton Applied Research), and Scanning electron microscopy (Model S4500, Hitachi).

X-ray diffraction (Figure 2) and VSM results of the powder collected show the presence of pure Fe<sub>2</sub>O<sub>3</sub> with high saturation magnetization. Rapid coalescence of particles caused by the cooling water at the surface of collecting interface produces unagglomerated particles. In the case of lower temperature in flame particles generated have high saturation magnetization and coercivity. In the case of high temperature in flame, particles are more spherical in shape, having low saturation magnetization and coercivity.

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## D. References and Supplemental Information

1. Ennas, G., G. Marongiu, et al. (1999). "Characterization of nanocrystalline gamma-Fe<sub>2</sub>O<sub>3</sub> prepared by wet chemical method." *Journal of Materials Research* 14(4): 1570-1575.
2. Grimm, S., M. Schultz, et al. (1997). "Flame pyrolysis - a preparation route for ultrafine pure gamma-Fe<sub>2</sub>O<sub>3</sub> powders and the control of their particle size and properties." *Journal of Materials Science* 32: 1083-1092.

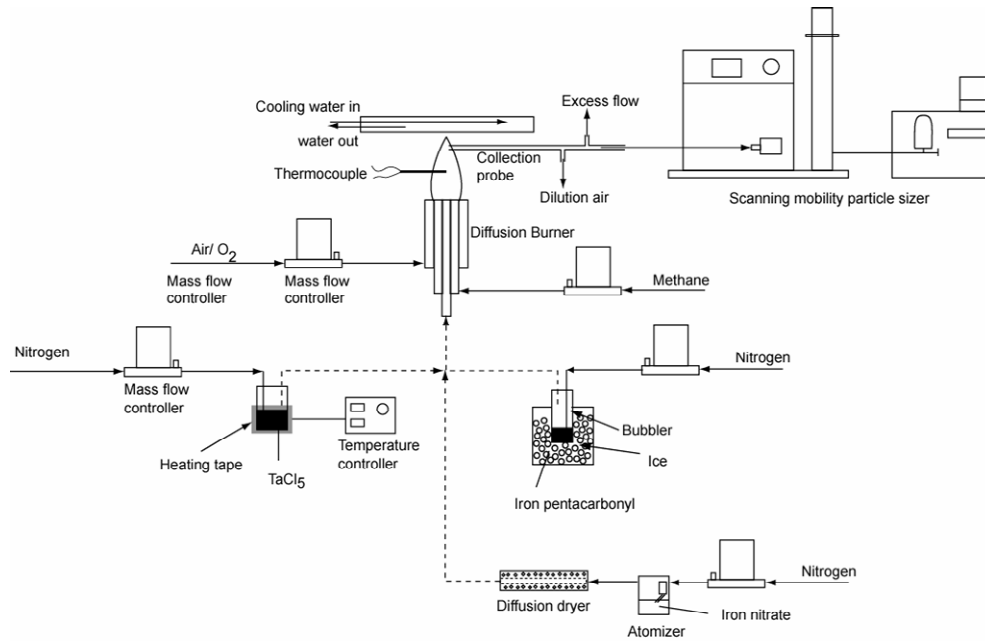


Figure 1: Schematic diagram of the experimental setup

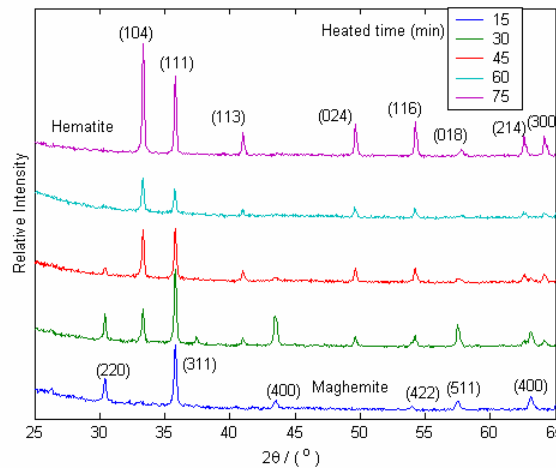


Figure 2: X-Ray diffraction pattern of CNA5 sample consisting of pure maghemite heated in the furnace at 500°C