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Synthesis and properties of magnetic fluid based on iron nanoparticles prepared by a vapor-phase condensation process

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Abstract

Magnetic fluid containing metallic iron nanoparticles was successfully fabricated in this work. The iron nanoparticles were synthesized by chemical vapor condensation process and then dispersed in water-base solution (pH 11) with oleic acid as surfactant. More than 80% of iron nanoparticles were fully dispersed in the fluid and remained stable without any further oxidation over 200 h. Both the iron nanoparticles and the subsequent magnetic fluid exhibited typical ferromagnetic behavior. © 2006 Elsevier B.V. All rights reserved.

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1. Introduction

Magnetic fluid has a great potential for magnetic seal, speaker damper, aerospace fuel injector as well as magnetic resonance imaging (MRI) agent [1]. So far, iron-oxide nanoparticles have been used in most magnetic fluids and some metallic iron particles whose size is somewhat larger than that of the iron oxides have also been tried [2]. However, relatively lower magnetization of the iron oxides and strong adhesion of the iron particles have limited practical application of such magnetic fluids. In this study, a feasibility of metallic iron nanopowder, fabricated by a vapor-phase condensation, as medium of magnetic fluid was investigated by examining dispersity and magnetic properties of both the iron nanopowder and the subsequent magnetic fluid.

2. Experimental

Iron nanoparticles were fabricated by chemical vaporcondensation(CVC) process using iron pentacarbonyl (Fe(CO)₅) as precursor [3,4]. The liquid precursor was vaporized in evaporator. The vapor was then transferred with high-purity Ar (0.01 Torr) into tubular reactor kept at 600 °C so that the precursor decomposed and condensed to nanoparticles. The condensed particles were then carried to collection chamber held at room temperature under highpurity Ar, in which the particles were deposited on the surface of the chamber. Finally, the particles were passivated with a mixed gas of Ar and O₂ before collection. The synthesized iron nanoparticles were dispersed in DI water (10 g/L) with oleic acid (5 cm^3) to make a magnetic fluid. The solution was subsequently ultra-sonicated for 30 min at 80 °C while NH₄OH was added to control pH of the solution. After the magnetic fluid was made, it was left alone in air and a fixed amount of the fluid was taken and weighted periodically to examine dispersity of the iron nanoparticles. Both the iron nanoparticles and the fluid were characterized by using Cu Ka X-ray diffraction, TEM, and VSM.

3. Results and discussion

Microstructures of iron nanoparticles synthesized at $600 \,^{\circ}$ C are shown in Fig. 1. These spherical particles with the average size of about 20 nm were dispersed pretty well,

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Fig. 1. TEM micrographs of iron nanoparticles synthesized at 600 $^{\circ}$ C by chemical vapor condensation (CVC) process.



Fig. 2. Dispersity vs. standing time and pH, for the iron nanoparticles shown in Fig. 1 in water-base magnetic fluids.

without forming big cluster. As shown in the right-hand figure, each particle had a typical core–shell structure: The metal iron core was enclosed with a layer of iron oxide, i.e., Fe_3O_4 [4].

In order to measure the dispersity of iron nanoparticles in the water-base fluid, a fixed amount of the fluid was taken from the equivalent upper position of the standing fluid, and subsequently dried and weighted. The dispersity was then expressed as the ratio of the weight, measured as a function of time, to the initial weight. As shown in Fig. 2, partially agglomerated or large iron particles were rapidly settled within 10 min. However, finer particles were suspended well in all fluids, resulting in slight decrease of dispersity with the increase of standing time. Furthermore, the dispersity of iron nanoparticles tended to increase with the increase of pH in the fluids, showing the optimum value at pH 11. But it decreased abruptly at pH 12. In general, the electric double layer on the surface of iron nanoparticle increases as pH of the fluid increases. It causes higher repulsive reaction between each electrically charged iron nanoparticle, thereby providing better dispersion of the particles in the fluid. The reason of the abrupt decrease in dispersity at pH 12 is uncertain. Nevertheless, it was found



Fig. 3. X-ray diffraction patterns of iron nanoparticles synthesized at 600 °C by CVC process and of those in a magnetic fluid (pH 11).



Fig. 4. Magnetization curve of a magnetic fluid (pH 11) made with iron nanoparticles synthesized at 600 °C by CVC process. Inset: M vs. H, for the iron nanoparticles themselves.

that the dispersity exceeding 80% could be obtained at pH 11.

As can be seen in X-ray diffraction patterns in Fig. 3, strong (110) peak of crystalline α -Fe was still detected, without showing any visible change in iron-oxide peaks, from the water-base magnetic fluid left alone for 200 h. It indicates that the iron nanoparticles suspended in the fluid can remain stable for a long time, obviously due to the oxide layer that encloses metal iron core, as shown in Fig. 1, and is stable enough to prevent further oxidation into the interior of the nanoparticles.

Fig. 4 shows magnetization curves measured from the iron nanoparticles shown in Fig. 1 and the magnetic fluid of pH 11 containing these particles. Comparing with the saturation magnetization of bulk Fe (220 emu/g), M_s of the

iron nanoparticles was considerably low (~125 emu/g). It is mainly due to the surface oxide layer that has much lower saturation magnetization and the surface anisotropy of the iron nanoparticles [5]. Because of the strong dilution of the iron nanoparticles in the fluid and, moreover, the removal of larger particles that were rapidly settled down in the fluid, $_iH_c$ and M_s of the fluid were significantly low, i.e., 27.4 Oe and 3.9 emu/g, respectively.

4. Conclusions

Magnetic fluid based on metal powder has been successfully made with iron nanoparticles synthesized by CVC process. The iron nanoparticles were about 20 nm in size and had core–shell type structure, i.e., metal iron core enclosed with thin layer of Fe₃O₄. More than 80% of these particles were fully dispersed in the magnetic fluid of pH 11

and remained stable without any further oxidation for a long time.

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