

# Aerosol Synthesis and Growth Mechanism of Magnetic Iron Nanoparticles

O.V. Tolochko<sup>1,a</sup>, E.S. Vasilieva<sup>1,b</sup>, D. Kim<sup>2,c</sup>, D.W. Lee<sup>2,d</sup>, B.K. Kim<sup>2,e</sup>

<sup>1</sup> State Polytechnic University, Polytechnicheskaya Str., 29, St. Petersburg, 195251, Russia <sup>2</sup> Korea Institute of Machinery and Materials, 66, Sangnam-Dong, Changwon, Kyungnam, 641-010, South Korea <sup>a</sup>oleg@ftim.spbstu.ru, <sup>b</sup>katerina@ftim.spbstu.ru, <sup>c</sup>ktcmspb@hanmail.net, <sup>d</sup>ldw1623@kmail.kimm.re.kr, <sup>e</sup>kbk1649@kmail.kimm.re.kr

#### Abstract

Magnetic oxide-coated iron nanoparticles with the mean size ranging from 6 to 75 nm were synthesized by aerosol method using iron carbonyl as a precursor under the flowing inert gas atmosphere. Oxide shells were formed by passivation of asprepared iron particles. The influence of experimental parameters on the nanoparticles' microstructure, phase composition and growth behavior as well as magnetic properties were investigated and discussed in this study.

Keywords : iron, nanoparticles, Mössbauer spectroscopy, structure, magnetic properties

## 1. Introduction

Synthesis and application of magnetic nanoparticles are emerging in wide areas, especially in electronics and biotechnology due to their special characteristics of superparamagnetic properties and single-domain magnetism. The magnetic properties of fine particles have been studied intensively with both technological and theoretical interest connected with magnetic phenomena related to size effect. Furthermore, this research field is in rapid progress because of wide potential application areas, such as components of magnetic tapes, ferrofluid, magnetic refrigerants, and the wide range of medical applications. A wide range of techniques to fabricate magnetic nanoparticles has been developed rapidly over the past decades, such as mechanical milling, spray drying, sol-gel, sonochemistry, etc. Among them, chemical synthesis of nanoparticles is a rapidly growing field, because of its versatile applicability to almost all materials and high rate of production capability with little agglomeration. In this study, the structure and magnetic properties of magnetite coated iron nanoparticles synthesized by Chemical Vapor Condensation (CVC) method were examined and discussed.

## 2. Experimental and Results

The basic equipment for CVC process was described previously [1].

Structure, phase composition and magnetic properties were studied by X-ray diffraction analysis, scanning electron microscopy, Mossbauer spectroscopy and vibration sample magnetometry methods. The morphologies and particles size distribution were determined by transmission electron microscopy.

Fig. shows TEM micrographs of oxide coated nanoparticles with the mean size of 8 and 28 nm. Electron diffraction patterns show the same phase composition of such particles: iron core and oxide shell. The shells consisted of magnetite [2] and their thickness was evaluated about 2-3nm.



TEM images and electron diffraction patterns of nanoparticles with the mean size of (a) 8nm and (b) 28nm.

At the synthesis process particles did not appear at the temperatures lower than 260°C. The increasing decomposition temperature and residential time of nanoparticles in reactor increased the mean particles size and lead to more asymmetric size distribution. Unagglomerated magnetic nanoparticles form intricate long threads to minimise the magnetic energy. By Mossbauer study it was shown, that according to CVC parameters, particles' structure contains about 15-100% of bulk iron and up to 45% of Fe<sub>3</sub>O<sub>4</sub>, another phases can be Fe

(Fine),  $\alpha$ -FeOOH (Fine and middle), or  $\gamma$ -FeOOH.

At the higher temperature, particles coagulated and structure completely consisted of filaments of 40-100nm in diameter depending on experimental parameters. Such structure is so unstable that new large particles are formed in the temperature of higher than 700°C and does not grow significantly as the temperature increases up to 1000°C. The mean size of these particles depends on size of initial iron particles.

Maximal magnetization of the powder with the average particle size of 75 nm is about 210 emu/g, which is almost theoretical value of 225 emu/g known in pure bulk iron. At the smaller mean size of particles, magnetization is also in the good correspondence with values which were calculated in speculation that particles consist of iron core and magnetite shell of 2 or 3nm thickness (Ms ~90emu/g). However, when the particle size is smaller than 12nm the maximum magnetization of the particles decreases continuously. Such decrease of magnetization comes from the increase of portion of superparamagnetic particles with the decrease of mean particle size. Particles smaller them 8 nm behave like paramagnets.

The maximum coercivity obtained so far is about 1 kOe, measured from the iron nanoparticles with the average size of 20-25 nm. It means that Fe core size is at least 15-21nm, which is much bigger than theoretically estimated domain size.

#### 3. Summary

Iron-based magnetic nanoparticles of 6-75nm can be successfully produced by CVC method. Particle growth behavior was investigated in different conditions through microscopic examination considering formation parameters, size and structure of nanoparticles. Ultrafine particles of less than 8nm size were superparamagnetic, maximum coercivity of iron nanoparticles obtained in this study (~ 1 kOe) was shown in the size of 20-25 nm and maximum magnetization was 210 emu/g, almost approaching the theoretical value known in pure bulk iron. Produced particles would be applicable to magnetic liquids, magnetic recording media or permanent magnets.

## 4. References

- O.V. Tolochko, A.G. Nasibulin, C.-J. Choi, I. Altman, B.-K. Kim, and E.I. Kauppinen : J. Aerosol Sci Vol. 34 (2003), p. 301.
- 2. C.J. Choi, O.V. Tolochko, and B.K. Kim: Mater. Letters Vol. 56 (2002), p. 289.