The 2005 IEEE International Magnetics Conference
April 4 – 8, 2005 • Nagoya Congress Center • Nagoya, Japan

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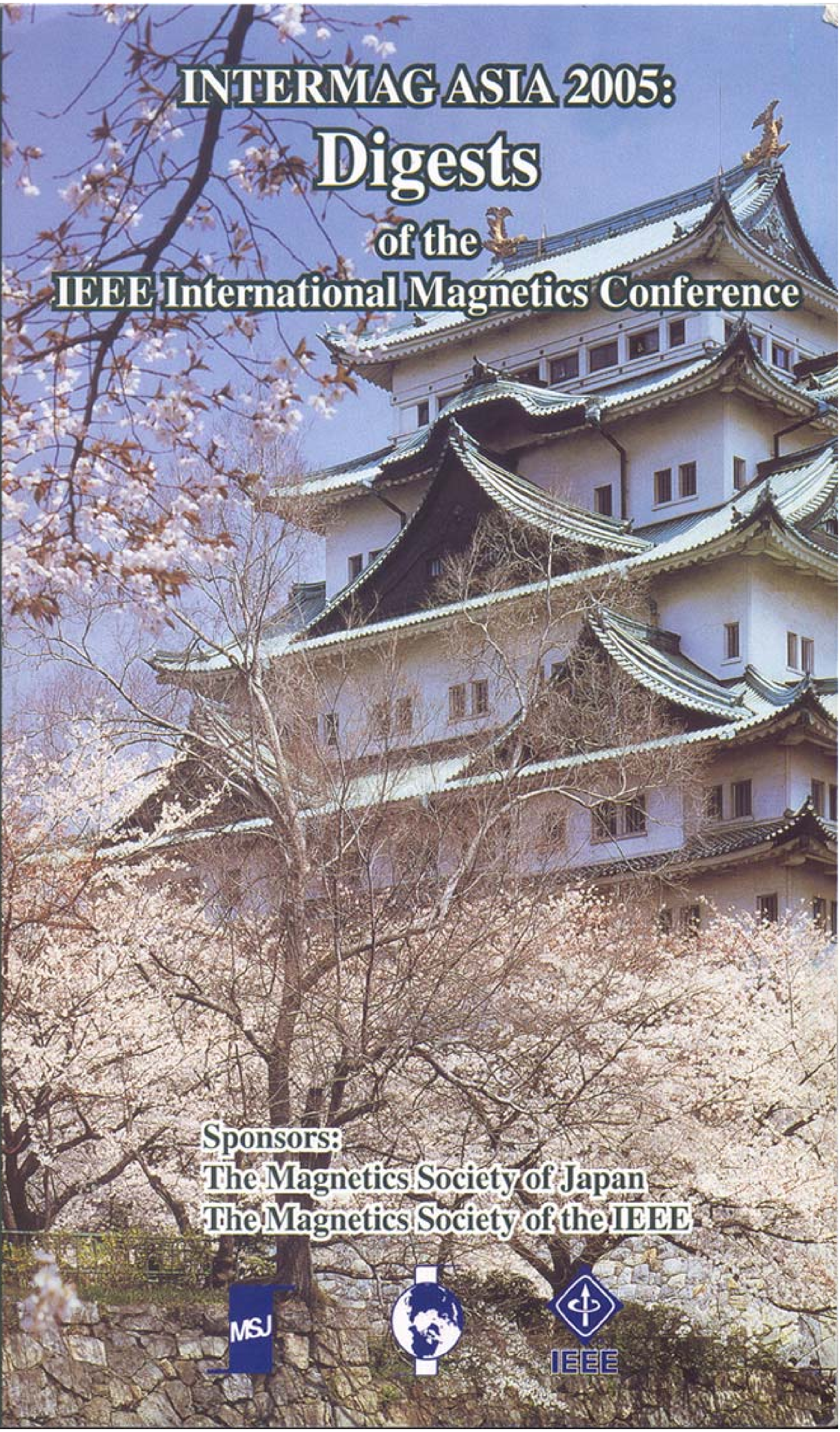

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MÖSSBAUER STUDIES OF NANO-SIZE CONTROLLED IRON OXIDE FOR BIOMEDICAL APPLICATIONS

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Introduction

The importance of magnetic nanoparticles is increasing for several biomedical applications. The use of particles that present superparamagnetic behavior at room temperature (no remanence along with a rapidly changing magnetic state) is preferred For biomedical applications [1]. Therefore, many researchers have reported a synthesis of magnetic nanoparticles with sizes variable from 1 to 20 nm by a various starting materials. However, performance and stable quality of the character of samples are not established, yet, because the analysis tools of nanoparticles are very weak.

Here we report a synthesis of iron oxide nanoparticles and correct characterizations by Mössbauer spectroscopy and transmission electron microscope (TEM). Mössbauer studies are essential tool because the only x-ray diffractometer (XRD) patterns in nanoparticles could not distinguish iron oxides from magnetite, maghemite, and spinel oxides because of similar crystal structure. Furthermore, we have coated the iron oxide nanoparticles by tetraethyl orthosilicate (TEOS) for biomedical applications.

Experiments and Results

MFe_2O_4 (M=Fe, Co) with spinel structure are made by reaction of iron(III) acetylacetonate [$Fe(acac)_3$] with surfactants at high temperature. We have used the phenyl ether, benzyl ether, and 1, 2-hexadecanediol as solvents [2]. $Fe(acac)_3$ was mixed in phenyl ether and benzyl ether for synthesis of the magnetite (Fe_3O_4). As boiling point of phenyl ether (259 ℃) is lower than that of benzyl ether (298 ℃), the size of magnetite nanoparticles can be controlled. And then, iron oxide nanoparticles have been coated by tetraethyl orthosilicate (TEOS) mixed ethyl alcohol and NH_4OH.

XRD measurements were performed using Cu$K\alpha$ radiation for interpretation of the existence of impurities in samples. Mössbauer spectra were recorded at room temperature and 15 K using a constant acceleration Mössbauer spectroscopy with a ^{57}Co in Rh matrix. The isomer shift values were reported with respect the to the Fe metal [3]. A drop of diluted nanoparticles solution in toluene was put onto a carbon coated copper grid and dried naturally for transmission electron microscopy (TEM) analysis.

The average particle sizes of iron oxides were 6, 13, and 18 nm, narrow size distribution was convinced by TEM. The Mössbauer spectrum for the 6 nm sample at room temperature displays a superparamagnetic behavior as demonstrated by the single quadrupole doublet

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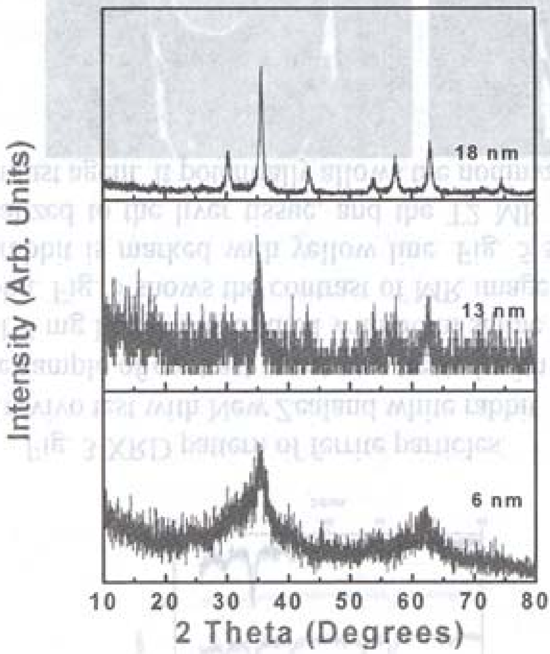
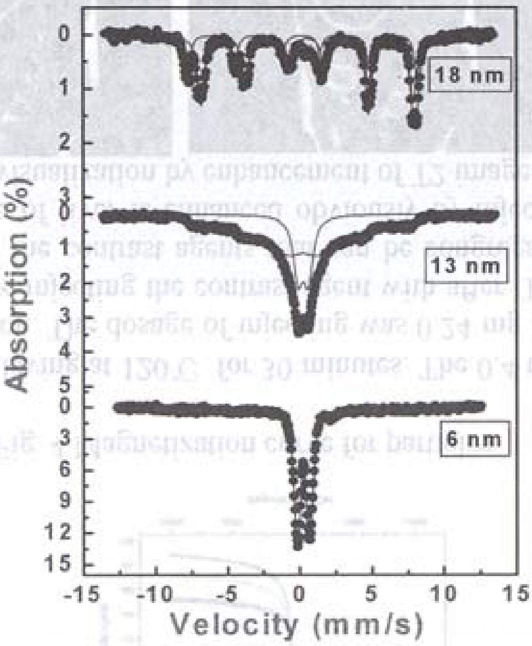
with zero hyperfine fields. While 13 and 18 nm particle show partially superparamagnetic behavior at room temperature. With only XRD patterns, the formation of samples could not distinguished easily because of similar crystal structure and line broadening of peaks in Fig. 1. 13 and 18 nm samples consist of magnetite or maghemite, but it can not be discriminated exactly. It is concluded that 13 nm and 18 nm samples are maghemite and magnetite, respectively, from the Mössbauer spectra. The analysis parameters, for the 13 nm sample, are hyperfine fields H_{hf} = 426 kOe, isomer shift δ =0.35 mm/s and quadrupole splitting E_Q= 0.69 mm/s, respectively. These values are typical Fe^{3+} ions in the high-spin state, and we suggest that this sample corresponds to a γ-Fe_2O_3. The spectrum for the 18 nm samples at room temperature shows a general sextet shape indicating ferrimagnetic behaviors. For this sample, the room temperature spectrum was fitted using two magnetic components of hyperfine fields H_{hf} = 494 and 458 kOe, isomer shifts δ = 0.18 and 0.59 mm/s corresponding to Fe^{3+} ions at sites A and Fe^{2+} ions at site B, respectively. And relative area ratio is A / B = 29 % / 71 %, therefore, it is concluded that this sample corresponds to a Fe_3O_4 ($[Fe^{3+}]_A[Fe^{2+}Fe^{3+}]_BO_4$).

It is suggested that 6 nm samples are available for biomedical applications such as hyperthermia and drug delivery system as a magnetic fluid carrier and *in vitro* and *in vivo*, they are coated with nontoxic materials such as Si or SiO_2 (from TEOS) essentially.





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