IEEE
International Magnetics Conference
San Diego, California
May 8-12, 2006

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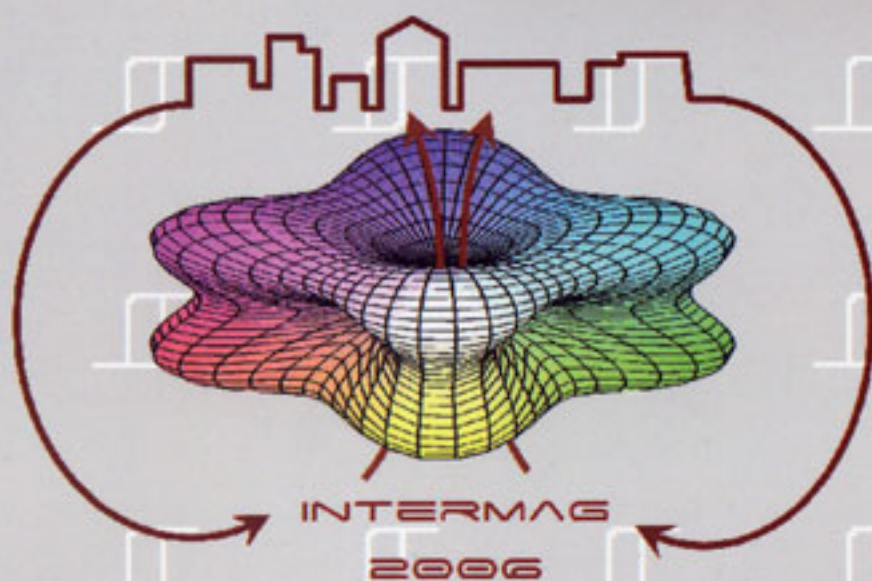
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HW-06

Synthesis and Magnetic Properties of LiFeO₂ Powders by a Sol-Gel Method.

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Introduction

The commercial lithium battery with high energy density and good cycle life has been studied as a power source for portable electronics. Many research groups have investigated various cathode materials for the lithium secondary batteries such as a layered oxide; $LiMO_2$ (M=Co, Ni, Mn, Fe), which consists of alternating layers of trigonally distorted MO_6 and LiO_6 octahedral sharing edges[1]. Lithium iron oxides such as $LiFeO_2$ and $LiFe_5O_8$ are increasing scientific interest and are also promising candidates for cathode materials in rechargeable lithium batteries as well as low-cost substitutes to garnet materials ($Y_3Fe_5O_{12}$) in microwave frequency applications[2].

Experiments

Ordered $LiFeO_2$ powders were prepared by the sol–gel method. Lithium acetate[CH_3CO_2Li] and iron nitrate nonahydrate[$Fe(NO_3)_9H_2O$] were dissolved in ethanol and distilled water.$LiFeO_2$ powders were annealed in the range of 300–1000 °C in air atmosphere for 6 h. The crystal structures were measured by X-ray diffraction (XRD) using CuKα radiation. Thermal analysis such as thermogravimetry analysis (TGA) and differential thermal analysis (DTA) were performed on the dried powder obtained from the $LiFeO_2$ precursor solution. The particle morphology of the samples was observed using a scanning electron microscope (SEM). Magnetic properties were measured using a vibrating sample magnetometer (VSM). Mössbauer spectra were recorded using a 40 mCi ^{57}Co source in a Rh matrix with the spectrometer working at constant acceleration.

Results and Discussion

DTA analysis indicated an exothermic peak. The peak at 245 °C is associated with the organic weight losses. However, the final peak appears at about 550 °C, indicating the complete thermal decomposition and crystallization of α–$LiFeO_2$ occur simultaneously. α–$LiFeO_2$ powders that were annealed at and above 600 °C have a single-phase. The x-ray diffraction patterns were refined by Rietveld refinement using the FULLPROF program. The crystal structure of the α–$LiFeO_2$ at room temperature, was determined to be cubic of *Fm3m* space group with its lattice constant a = 4.1610 Å ±0.0005. The Bragg factors R_B and R_F were 5.56 % and 3.79 %, respectively. The SEM micrographs indicate the distribution of grains uniform with really spherical shape. The powders present average particle sizes of 110 nm. Mössbauer spectra of the α–$LiFeO_2$ were taken at various absorber temperatures from 4.2 to 295 K. The Néel temperature of α–$LiFeO_2$ is found to T_N=90±2 K. Figure 1 illustrates some of the spectra of the sample, which were composed of two sixline hyperfine patterns. The spectra for the samples at 4.2 K exhibit general sextets shape indicating ferrimagnetic behaviors. The spectrum was fitted using the two magnetic components of hyperfine fields H_{hf}= 506 and 478 kOe, isomer shifts δ = 0.37 and 0.36 mm/s corresponding to Fe^{3+} ions. Mössbauer spectra of ^{57}Fe at room temperature for α–$LiFeO_2$ powders are shown paramagnetic behavior as demonstrated by the single quadrupole doublet with zero hyperfine fields. The hyper-

fine parameters for the sample are isomer shift δ= 0.24 mm/s and quadrupole splitting E_Q = 0.61 mm/s, respectively. Figure 2 shows the fractional change of the average magnetic hyperfine field,$[H_{hf}(T)-H_{hf}(0)]/H_{hf}(0)$, as a function of reduced temperature T/T_N. The average magnetic hyperfine field decreases with increasing temperature according to $[H_{hf}(T) - H_{hf}(0)] / H_{hf}(0) = -0.36(T/T_N)3/2 - 0.27(T/T_N)5/2$ for $T/T_N < 0.7$, indicative of spin-wave excitation. $ln\,F$ versus T_2 for α-$LiFeO_2$, where *F* stands for the total resonance absorption to the recoil-free fraction *f*. From the temperature variation of the total Mössbauer absorption areas, we obtained the Debye temperatures of α–$LiFeO_2$ to be Θ=253 ±5 K. The temperature dependence of the inverse molar susceptibilities was linear above 200 K for α–$LiFeO_2$. The calculated Curie-Weiss temperature (θ_P) value was -160±5 K for α–$LiFeO_2$.

[1] Y. S. Lee, C. S. Yoon, Y. K. Sun, Y. Sato, Electrochemistry Communications, **4**, 727(2002).
[2] K. U. Kang, H. N. Oak, and C. S. Kim, J. Appl. Phys. **97**, 10F102(2005).
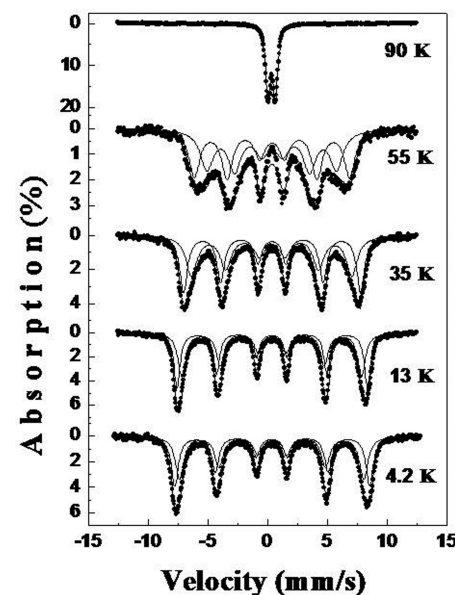

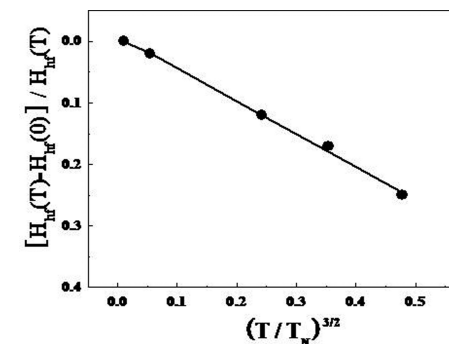**Fig. 2. Fractional change of the average magnetic hyperfine field, H_{hf}, as a function of $(T/T_N)^{3/2}$ for α-LiFeO₂.**

Fig. 1. Mössbauer spectra of α-LiFeO₂ at various temperatures.