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*San Diego, California*

*May 8-12, 2006*

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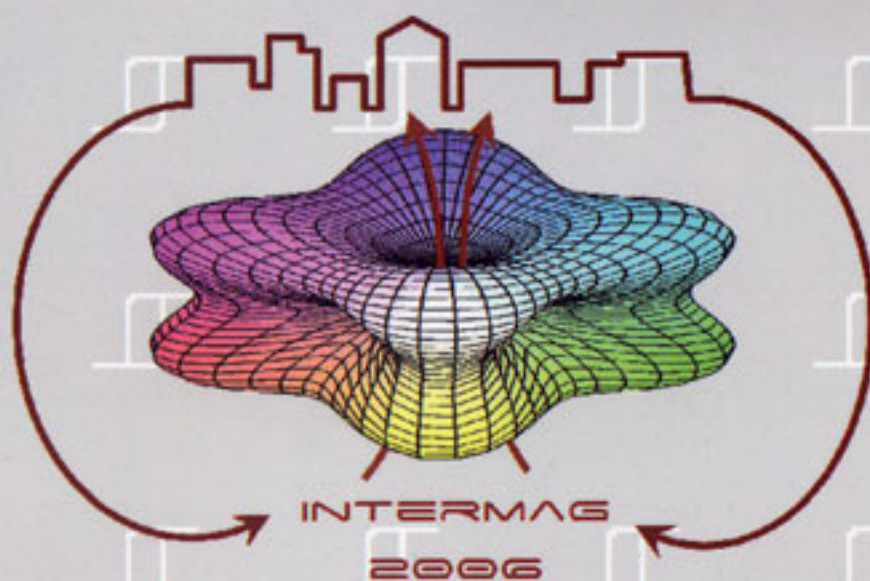
Exhibits

Magnetism Society Information

Program

Technical Digests

Author Index



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## CR-04

## The Effect of Cation Ion Ordering in In- Cr- Sulphur Spinel.

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## Introduction

Chromium chalcogenide spinel  $M\text{Cr}_2\text{X}_4$  ( $M = \text{Fe}, \text{Co}, \text{Cu}, \text{Cd}, \text{X} = \text{S}, \text{Se}$ ) shows various magnetic properties with M ions.  $\text{CuCr}_2\text{Se}_4$  and  $\text{CdCr}_2\text{Se}_4$  are known to show metallic conduction and large magneto-optical effect [1]. In addition to colossal magnetoresistance (CMR) effect, metal-insulator transition and structural phase transition appear in  $\text{FeCr}_2\text{S}_4$  [2], spin-frustration effects reveal in  $\text{FeSc}_2\text{S}_4$  and  $\text{MnSc}_2\text{S}_4$  [3]. These systems have been revisited relaxor ferroelectricity and colossal magnetocapacitive effect [4]. These features were attributed to competition of isomorphic ions with the topological frustration, Jahn-Teller distortion, and geometric frustration of magnetic moment. Here, we report the magnetic properties of the  $\text{FeCr}_2\text{S}_4$  and  $\text{FeIn}_2\text{S}_4$  with special emphasis on cation ordering related to the quadrupole interactions.

## Experiments

Syntheses of the  $\text{FeCr}_2\text{S}_4$  and  $\text{FeIn}_2\text{S}_4$  were accomplished by the solid state reaction of the high-purity elements Fe, Cr, In and S in an evacuated quartz tube. The crystal structure of the samples was examined using x-ray diffractometer (XRD) with  $\text{Cu-K}\alpha$  radiation and analyzed by Rietveld refinement. The Mössbauer spectra were recorded using a conventional spectrometer of electro-mechanical type with a  $^{57}\text{Co}$  source in a rhodium matrix.

## Results and discussions

Fig.1 shows the x-ray diffraction refinements for the  $\text{FeCr}_2\text{S}_4$  and  $\text{FeIn}_2\text{S}_4$  samples at room temperature, respectively. The spectra shown in Fig. 1 demonstrate the absence of any impurity phases. The determined crystal symmetry of samples is a cubic spinel structure  $Fd3m$ .  $\text{FeIn}_2\text{S}_4$  is an inverse spinel, with In atoms occupying both tetrahedral (A) and octahedral (B) sites. On the other hand,  $\text{FeCr}_2\text{S}_4$  has a normal spinel with Fe atoms occupying A site and Cr atoms occupying B site. The determined lattice constant  $a_0$  for  $\text{FeCr}_2\text{S}_4$  and  $\text{FeIn}_2\text{S}_4$  were  $a_0=10.011$  and  $a_0=10.616$  Å, respectively, since result might be the larger ionic radius for In ions than for Cr ions.

In order to study microscopic interaction mechanism, the Mössbauer spectra of sulphur spinel  $\text{FeCr}_2\text{S}_4$  and  $\text{FeIn}_2\text{S}_4$  have been studied. Fig. 2 shows Mössbauer spectra for the  $\text{FeCr}_2\text{S}_4$  and  $\text{FeIn}_2\text{S}_4$  at 4.2 K and room temperature, respectively. The Néel temperatures were found to be 175 and 15 K for the  $\text{FeCr}_2\text{S}_4$  and  $\text{FeIn}_2\text{S}_4$ , respectively, by Mössbauer spectroscopy. It can be understood as the strength of inter-sublattice exchange interaction  $\text{Fe}^{2+}(\text{A})-\text{S}^{2-}-\text{Cr}^{3+}(\text{B})$  is stronger than that of the intra-sublattice exchange interaction  $\text{Fe}^{2+}(\text{B})-\text{S}^{2-}-\text{Fe}^{2+}(\text{B})$ . The large asymmetrical line broadening of Mössbauer absorption lines is shown for the samples at 4.2 K. We note that the  $\text{FeCr}_2\text{S}_4$  shows a single line resonance spectrum with an isomer shift of 0.72 mm/s at room temperature, while  $\text{FeIn}_2\text{S}_4$  at room temperature has an isomer shift of 0.74 mm/s and a electric quadrupole splitting ( $\Delta E_Q$ ) of 3.22 mm/s. The charge state of Fe ions is ferrous ( $\text{Fe}^{2+}$ ) as characterized by isomer shift ( $\delta$ ) for the samples. We interpret that the presence of the  $\Delta E_Q$  is attributed to the trigonal field at the octahedral site, according to  $\text{Fe}^{2+}$  ions enter to octahedral B site.

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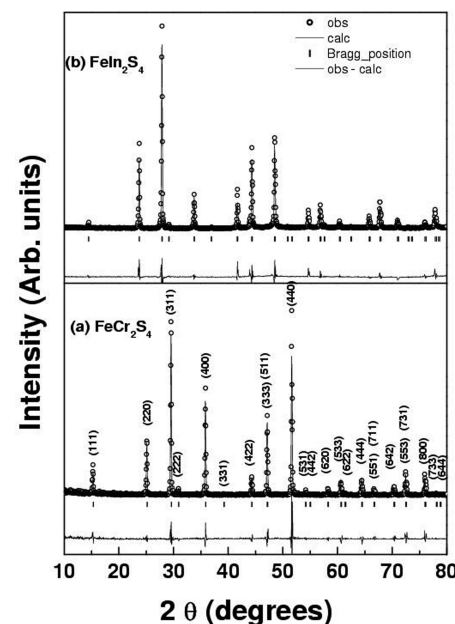


Fig. 1. Refined x-ray diffraction patterns of the (a)  $\text{FeCr}_2\text{S}_4$  and (b)  $\text{FeIn}_2\text{S}_4$  at room temperature.

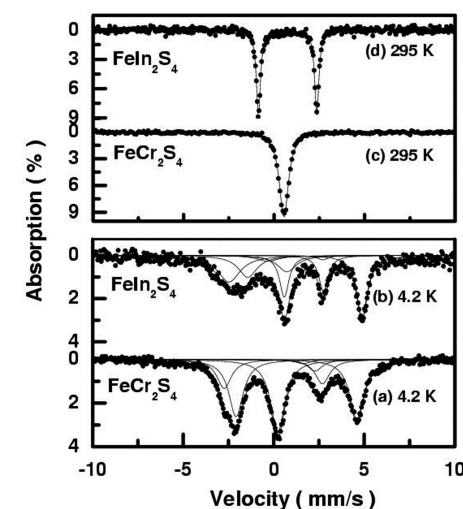


Fig. 2. Mössbauer spectra of  $\text{FeCr}_2\text{S}_4$  and  $\text{FeIn}_2\text{S}_4$  at 4.2 K and room temperature.