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Mössbauer study of iron sulfide nano-compound.

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Introduction

The Fe–S system has been studied to investigate a complex phase diagram by many researchers [1]. The iron (II) sulfide (FeS) has been much interested for the phase stability and physical properties under high pressure and temperature [2,3]. Stoichiometric iron (II) monosulfide (FeS), troilite has a hexagonal close packed structure with antiferromagnetic ordering at room temperature. It undergoes α transition into the ideal (NiAs) hexagonal structure which includes the pyrrhotites (Fe_{1-x}S) between 120 \sim 140 °C [1,4]. Besides, pyrrhotites (Fe_{1-x}S) show ferrimagnetic behavior due to unbalanced ferromagnetic coupling. In this paper, we report the ferrimagnetic behavior of nanocompound iron(II) monosulfide (FeS) at room temperature.

Experiments

The iron sulfide, FeS, was fabricated using a polyol method. The sulfur solution was the first prepared with sulfur powder and oleylamine. Iron(III) acetylacetonate was mixed in oleylamine for 30 min. under vacuum, then heated up to 80 °C to clearly dissolve it and maintained for 30 min. under Ar atmosphere. It reheated up to 340 °C and injected rapidly the sulfur solution under Ar atmosphere. Then, it cooled down to room temperature.

The phase purity and crystallinity of synthesized sample were characterized by X-ray diffraction (XRD) measurements using $CuK\alpha$ radiation ($\lambda=1.5406$ Å). Mössbauer spectrometer of the electromechanical type with a 50 mCi ⁵⁷Co source in Rh matrix was used in the constant-acceleration mode. The Mössbauer parameters were obtained by a least-squares fitting program assuming Lorentzian line shapes. The magnetizations were measured by using a vibrating sample magnetometer (VSM).

Results and discussion

XRD was used to confirm the crystal structure and crystallographic parameters of FeS nanocompound, as shown in Fig. 1. An analysis of XRD patterns by Rietveld refinement method using FULLPROF program shows that the sample has a troilite hexagonal structure (space group P-62C) with a lattice constant $a_0 = 5.985 \pm 0.001$, $b_0 = 5.985 \pm 0.001$ and $c_0 = 11.658 \pm 0.001$ Å.

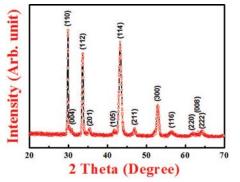
The hysteresis loop measured using VSM with maximum applied field 1 T at room temperature shows the ferrimagnetic behavior as shown in Fig. 2. The saturation magnetization (M_s) and the coercivity (H_s) values of the sample are found to be 1.8 emu/g and 251.1 Oe, respectively.

The Mössbauer spectra of FeS nanocompound were taken at various temperatures ranging from 4.2 to 295 K to understand localized nearest neighbor effects on effective field, as shown in Fig. 3. We notice that the Mössbauer absorption spectrum shows 2-sextets at room temperature, while it shows a clear 1-sextet at 4.2 K. We conclude that FeS nanocompound shows ferrimagnetic behavior at room temperature as pyrrhotites (Fe_{1-x}S) by analysis of Mössbauer spectrum. This is consistent with the result of hysteresis loop obtained by VSM measurement, too. The isomer shift value at room temperature is found to be $(0.60 \sim 0.64) \pm 0.01$ mm/s, relative to the Fe metal, which are consistent with the Fe²⁺ valence state. The ferrous character of the Fe ions is also manifested by the magnitudes of the magnetic hyperfine fields: the magnetic hyperfine field of FeS nanocompound is approximately 327 \pm 5 kOe at 4.2 K, which corresponds to ferrous ion.

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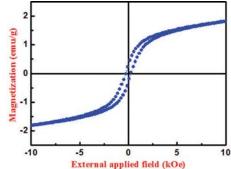


Fig. 1. X-ray diffraction pattern of FeS nanocompound prepared by polyol method.

Fig. 2. Hysteresis loop of FeS nanocompound at room temperature.

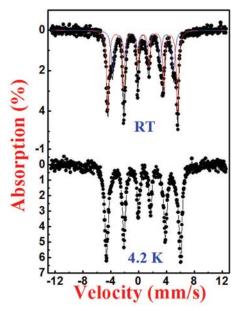


Fig. 3. Mössbauer spectra of FeS nanocompound at various temperatures.

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