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Synthesis of Ultrafine $\text{SrFe}_{12}\text{O}_{19}$ Particles with High Coercivity

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Abstract: Ultrafine particles of $\text{SrFe}_{12}\text{O}_{19}$ have been synthesized by citrate precursor technique. Solid state reactivity studies of citrate precursor lead to the formation of fine particles of $\text{SrFe}_{12}\text{O}_{19}$ below 823K. The size and morphology of these particles were studied by SEM. The surface area measured by BET method for 5.2nm crystallite is found to be $27.45 \text{ m}^2/\text{g}$, having particle size 42nm. Ultrafine particles of $\text{SrFe}_{12}\text{O}_{19}$ shows interesting magnetic properties which differs from conventional particles and are measured by VSM. The intrinsic coercive force of 6500Oe is observed for ultrafine $\text{SrFe}_{12}\text{O}_{19}$ which is close to the theoretical limit and is one of the highest value reported so-far for isotropic $\text{SrFe}_{12}\text{O}_{19}$ having 16..5nm crystallite size. The T_c value for ultrafine particles is found to be around 830K which is larger than the reported value of 748K for bulk materials. Mossbauer spectroscopy have ben explored to study these fine particles for non-collinear spin structure predominantly at the surface compared to the core of the particle.

1. Introduction:

Recently hexagonal hard ferrites $\text{SrFe}_{12}\text{O}_{19}$ has attracted much attention not only for permanent magnets and microwave devices but also for a promising material for perpendicular magnetic recording medium[1]. In this exciting area, the key to success in achieving high performance parameters is to prepare highly uniform and ultrafine particles and their subsequent processing. This has prompted the development of various chemical methods for the preparation of stoichiometric and chemically pure hexaferrites[2-4] Synthesis of strontium hexaferrites by precursor technique is a continuation of our work on ferrites and garnets[5-7]. The magnetic behaviour of $\text{SrFe}_{12}\text{O}_{19}$ fine particles is of considerable interest because some of the extraordinary phenomena have been recently recognized as arising from the effects of finite dimension. Solid state reactivity studies of citrate precursor plays an important role to control the ultrafine nature of $\text{SrFe}_{12}\text{O}_{19}$ particles. These particles exhibit high coercivity and also investigated for their interesting magnetic properties.

2. Experimental:

The thermal analysis of the citrate precursor was performed using TG/ DTA IR and gas analyses. The powder XRD patterns of the materials were recorded using $\text{CuK}\alpha$ radiation with Ni filter and crystallite size was measured using scherrer method. The surface area was measured using BET method. The morphology and particle size distribution were observed by SEM . Mossbauer spectra of $\text{SrFe}_{12}\text{O}_{19}$ particles were recorded using Co-57 (Rh) source. The magnetic properties of strontium hexaferrite particles were measured by means of vibrating sample magnetometer in conjunction with a Varian magnet in a maximum available field of 11KOE.

3. Results and discussions:

3.1. Thermal decomposition studies of $\text{SrFe}_{12}\text{O}_6(\text{C}_6\text{H}_6\text{O}_7)_{13}$:

The mode of citrate precursor decomposition has a special significance in yielding ultrafine $\text{SrFe}_{12}\text{O}_{19}$ particles and are discussed elsewhere[7]. Thermal studies show two exothermic peaks corresponding to the formation of acetone dicarboxylate complex and further decomposition to complex carbonate with the evolution of large amount of acetone and CO_2 gases. The small asymmetric endotherm corresponds to carbonate decomposition resulting to amorphous $\text{SrFe}_{12}\text{O}_{19}$. The citrate precursor and the decomposed products show characteristic absorptions in IR spectra and were X-ray amorphous which crystallises above 823K as $\text{SrFe}_{12}\text{O}_{19}$. The reactivity and particle size are influenced by the heat treatment temperatures.

3.2. Nature of ultrafine $\text{SrFe}_{12}\text{O}_{19}$ particles:

Ultrafine $\text{SrFe}_{12}\text{O}_{19}$ particles were characterized in detail by XRD, SEM and surface area measurements. The materials exhibit broad hump in the XRD pattern below 823K indicating an absence of crystalline periodicity and crystallisation occurs around 823K [7]. The XRD crystallite size is increased slowly from 5.2-20nm at temperature from 773 and 1073K corresponds to BET surface area of 27.45 and $10.12\text{m}^2/\text{g}$ with particle size of 42 and 116nm respectively. Ultrafine particle is a aggregate (magnetic cluster) of crystallites and observed as a platelet like morphology in the SEM studies. Upon crystallization, the destabilisation of characteristic platelet like aggregates occur because of localised crystallite growth having size of 30-40nm in SEM studies.

Fig.1 Mossbauer Spectra for $\text{SrFe}_{12}\text{O}_{19}$ having different crystallite size, a(4.2nm), b(10.3nm), c(16.5nm)

Fig.2. Magnetization curves showing σ/σ_s Vs. H (Inset) and σ/σ_s Vs. T plots for $\text{SrFe}_{12}\text{O}_{19}$ having different crystallite size, A(16.5nm), B(14.0nm), C(10.3nm), D(8.2nm).

Mossbauer absorption spectra at room temperature is shown in Fig.1 for $\text{SrFe}_{12}\text{O}_{19}$ particles measured from XRD line broadening. The 4.2nm particles show doublet indicating superparamagnetic nature. However, 10.3nm size particles exhibit the coexistence of ferrimagnetic and superparamagnetic particles. The five sextets pattern was observed for 16.5nm size particles of $\text{SrFe}_{12}\text{O}_{19}$ due to iron located in five different magnetic polyhedral coordinations corresponding to 12K, 4f1, 4f2, 2a and 2b respectively. The Mossbauer parameters have very good agreement with the literature reported values[8]. However, there is a decrease in the hyperfine field with the particle size which can be explained on the basis that the hyperfine field decreases with the surface spin non-collinearity. Fig. 2. shows the characteristics plots of magnetization (σ) Vs. temperature (T) at a field of 6KOe and the inset shows the relative magnetization (σ/σ_s) as a function of field for $\text{SrFe}_{12}\text{O}_{19}$ materials. The $\text{SrFe}_{12}\text{O}_{19}$ particles (14.0 nm) ordered magnetically around 748 K and show specific saturation magnetization σ_s comparable to bulk materials (67.7 emu/g). This effect disappears for $d < 14.0\text{ nm}$ particles where T_c is increased from 748 to 830 K due to the decrease in the particle size. The lower σ_s values were observed for these particles because of spin noncollinearity at the surface and superparamagnetic nature exhibited in the mossbauer studies. These samples attained saturation magnetization and exhibit ferrimagnetic behaviour (inset Fig.2). However the coercive force range 4900 - 6500 Oe is shown by 14.0 to 20.0 nm crystallite size. Interestingly the 16.5 nm crystallite corresponding to 116nm BET particle size show a coercive force (H_{ic}) of 6500 Oe when the field is applied upto 11.0 KOe. This value is very close to the theoretical limit and one of the highest value reported for isotropic $\text{SrFe}_{12}\text{O}_{19}$ particles[9].

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