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Preparation of Nickel Ferrite from thermolysis of Nickel Hexa(Formato)-Ferrate(III)Hexahydrate

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Abstract. Thermal decomposition of nickel hexa(formato) ferrate(III) decahydrate i.e. $\text{Ni}_3[\text{Fe}(\text{HCOO})_6]_2 \cdot 10\text{H}_2\text{O}$ has been studied upto 973 K in static air atmosphere employing TG,DTG,DTA,DSC,XRD,ESR, Mössbauer and infrared spectroscopic techniques. Dehydration occurs in two stages in the temperature range of 348–423 K. Immediately after the removal of the last water molecule, the anhydrous complex undergoes decomposition till $\alpha\text{-Fe}_2\text{O}_3$ and nickel carbonate are formed at 588 K. In the final stage of remixing of cations, a solid state reaction between $\alpha\text{-Fe}_2\text{O}_3$ and nickel carbonate leads to the formation of NiFe_2O_4 at a temperature (873 K) much lower than for ceramic method. A high value of saturation magnetization i.e. 4440 gauss of the ferrite (NiFe_2O_4) shows its potential to function at high frequencies.

INTRODUCTION

Ferrites find extensive application in radio, television, microwave and satellite communication, bubble devices, audio-video and digital recording and as permanent magnets [1]. The synthesis of ferrites from thermolysis of iron carboxylate precursors has two major advantages over the conventional ceramic method viz. it does not involve milling of the starting material (precursor) and ferrites are formed at much lower temperature and in shorter time. The milling which is necessary in ceramic method imparts defects and strains to the precursor which in turn affect the permanent magnetic properties of the ferrites formed [2]. We have successfully obtained ferrites from the thermolysis of alkaline earth ferrimalonates [3-5] at temperature much lower in comparison to the ceramic method. The present investigation deals with the preparation of nickel ferrite from thermolysis of nickel ferriformate.

Experimental procedure

Nickel hexa(formato) ferrate(III) decahydrate i.e. $\text{Ni}_3[\text{Fe}(\text{HCOO})_6]_2 \cdot 10\text{H}_2\text{O}$ was prepared by mixing stoichiometric quantities of aqueous solutions of ferric chloride, nickel formate and formic acid. The reaction mixture was stirred vigorously and then concentrated on water bath until a brown colored product was formed. The brown product was filtered, washed with cold water and dried in air. The identity of the complex was established by elemental analysis. The infrared spectra of the complex (precursor) and products were recorded on Pye-Unicam SP3-300 IR Spectro-photometer in the range $4000\text{--}200\text{ cm}^{-1}$ using KBr pellet technique. Simultaneous TG, DTG and DTA curves were recorded on Stanton Redcroft STA-780 at a heating rate of 10° min^{-1} . DSC recording was performed on Mettler 25(Swiss) at a heating rate of 10° min^{-1} . ESR spectra were recorded on ESR spectrometer JES-FE3XG. Magnetic measurements were made at solid state physics laboratory, Ministry of Defence, New Delhi. XRD powder pattern was recorded at BARC, Bombay. Mössbauer measurements were made at USIC, University of Roorkee, Roorkee.

Results and Discussion

The infrared spectrum of the complex, $\text{Ni}_3[\text{Fe}(\text{HCOO})_6]_2 \cdot 10\text{H}_2\text{O}$ shows a broad band centered at 3300 cm^{-1} due to $\nu(\text{OH})$ of lattice water and a small but distinct band at 2900 cm^{-1} due to $\nu(\text{C-H})$. Broad bands centered at 1620 and 1350 cm^{-1} are attributed to $\nu_{\text{asym}}(\text{C=O})$ and $\nu_{\text{sym}}(\text{C=O})$ respectively of the coordinated formate groups. The bands at 570 cm^{-1} and 390 cm^{-1} due to $\nu(\text{Fe-O})$ suggest the presence of Fe-O (carboxylate) bonding [6,7]. The six coordination number of iron is satisfied by six formate ligands which are bound through oxygen atoms of the carboxyl groups. The transition metal and water molecules seem to be responsible for linking together the complex ion [8]. A 'g' value of 2.0 from esr spectrum indicates the presence of high spin iron(III) octahedral nature of the complex. Fig 1 shows the simultaneous thermal analysis (TG, DTG, DTA) curves of the complex at a heating rate of 10° min^{-1} . Presence of two plateau in TG curve suggests that dehydration occurs in two stages i.e. removal of two water molecules at 348 K and the remaining eight at 423 K. There are corresponding peaks in DTG at 323 K and 400 K respectively indicating that heat changes are accompanied by weight loss. DTA shows two endotherms centered at 325 K and 401 K for dehydration. DSC (Fig.2) exhibits a broad endotherm centered at 404 K showing a ΔH value of 3.306 kJ mol^{-1} for dehydration. The anhydrous complex then undergoes a gradual decomposition process until a weight loss of 57.8 % is obtained at 588 K indicating the formation of Fe_2O_3 and NiCO_3 .

(calc. loss = 57.48 %). The identities of these intermediates have been confirmed by Mossbauer and infrared spectra respectively. Mössbauer spectrum of the residue obtained after heating the precursor isothermally for 30 min at 573 K exhibits a symmetrical six-line pattern with isomer shift and internal magnetic field values of 0.38 mm s^{-1} and 510 KOe respectively indicating the formation of $\alpha\text{-Fe}_2\text{O}_3$ [9]. The corresponding DTG and DTA peaks exist at 568 K and 569 K(exothermic) respectively. DSC displays the corresponding exotherm at 570 K with a ΔH value of $4.594 \text{ kJ mol}^{-1}$ for the decomposition process. At higher temperature NiCO_3 completely decomposes into NiO as shown by a weight loss of 62 % at 873 K suggesting the presence of NiFe_2O_4 (calc. loss = 61.84%) formed as a result of solid state reaction between Fe_2O_3 and NiCO_3 . The final residue shows a Curie temperature of 864 K in good agreement with the reported value [1] of NiFe_2O_4 . The formation of the ferrite has also been confirmed by XRD powder pattern of the end product (Fig.3).

Fig. 1. STA curves of the complex at a heating rate of 10 K min^{-1} .

Fig. 2. DSC curve of the complex at a heating rate of 10 K min^{-1} .

Fig. 3. XRD powder pattern of the end product.

The ferrite (NiFe_2O_4) has been obtained at a temperature (873 K) much lower than that from ceramic method [10-11]. The saturation magnetization ($4\pi\text{Ms}$) value of 4440 gauss of the ferrite shows its potential to function at high frequencies [10].

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