Notes

In order to understand the effects of three important processing parameters on the quality of resulting ferrite powders/compacts (more than 10 parameters were optimized in Ref. 1), we synthesized Ni-Zn ferrite powder using the identical grades of chemicals with suitable changes in three processing parameters: (i) solution concentration, (ii) mode of washing and (iii) drying of the resulting precipitate. Due to these changes, the magnetic properties were found to be relatively poor (i.e. lower Ms value at the low applied magnetic fields). The resulting precipitate was further hydrothermally processed which substantially improved the saturation magnetization value, Ms = 50.7 emu/g. The experimental procedures, data acquisition and its analysis are presented in the paper.

Experimental

Chemical synthesis

The optimum concentrated solutions of nickel nitrate (3.95 M), zinc nitrate (4.2 M), ferric nitrate (2.87 M) and sodium hydroxide (5.3 M) were prepared using Loba and Merck LR grade chemicals. The solutions were filtered through sintered glass funnel (G4) to remove the insoluble impurities. The metal ion solutions were then premixed in a stoichiometric ratio to make 20 g of a typical Ni₀.₈Zn₀.₂Fe₂O₄ composition. The premixed nitrate solutions of Ni, Zn and Fe were added rapidly to the sodium hydroxide solution. The precipitation was carried out at room temperature with continuous stirring. Due to exothermic nature of reaction, the temperature of the slurry was found to rise around 50°C, forming the mixed hydrated oxides of iron, nickel and zinc. The precipitate was washed five times with distilled water using the centrifuge machine. The precipitate was further divided into four parts to give different thermal treatments: (i) the sample dried in an electrical oven at 80±5°C in air (Sample A), (ii) the sample dried under the IR lamp for 20 minutes (Sample B), (iii) the sample dried in microwave oven for 40 minutes (Sample C) and (iv) the sample treated hydrothermally (Sample D). For hydrothermal treatment, the ferrite slurry was added in a Parr bomb and it was sealed with Teflon. The contents were heated hydrothermally in a furnace at 100°C for 48 hr under saturated steam pressure. After...
the hydrothermal treatment, the Parr bomb was allowed to cool down naturally. The contents were taken out and dried at 80°C in oven. Then these as dried samples A, B, C, D were calcined at 1200°C for 4 hr in a furnace.

**Characterization**

All as-dried samples (A, B, C and D) and those further calcined at 1200°C were characterized using following physico-chemical techniques.

Powder X-ray diffractograms (XRD) were obtained using a Philips PW1730 diffractometer with CuKα radiation.

Saturation magnetization measurements were carried out at room temperature and at liquid nitrogen temperature by using a vibrating sample magnetometer EG&G PAR model 4500.

**Results and discussion**

XRD patterns of the samples A, B, C, D are shown in Fig.1. Powder pattern for sample A shows all peaks matching with the characteristic reflection of Ni-Zn ferrite as reported in Ref.1 indicating the formation of single phase spinel. Sample B and C do not show the characteristic spinel pattern, as the ferritization is incomplete. The XRD pattern for sample D shows well-defined spinel structure of single-phase compound with better crystallinity. On calcination to 1200°C all these samples show a well-defined and almost identical XRD pattern confirming the complete phase formation of Ni$_{0.8}$Zn$_{0.2}$Fe$_2$O$_4$.

The saturation magnetization values of samples A, B, C, D obtained are presented in Table 1. Fig. 2 indicates the nature of hysteresis loop for as-dried samples A, B, C, D. The sample A shows $M_s$ value of 17.7emu/g. This value is lower than the $M_s$ value 44 emu/g reported earlier$^1$ for Ni$_{0.8}$Zn$_{0.2}$Fe$_2$O$_4$. This decrease is observed because the amount of phase formation in sample A is less (as confirmed from XRD) as compared to that for the sample reported in Ref.1, which differs qualitatively. For sample B and sample C, the $M_s$ value still further decreases to 1.6

<table>
<thead>
<tr>
<th>Sample</th>
<th>As-dried sample</th>
<th>After calcination at 1200°C/4h</th>
<th>At liquid nitrogen temp. (83K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>17.7</td>
<td>67.0</td>
<td>74.0</td>
</tr>
<tr>
<td>B</td>
<td>1.6</td>
<td>63.0</td>
<td>73.5</td>
</tr>
<tr>
<td>C</td>
<td>4.0</td>
<td>62.3</td>
<td>73.0</td>
</tr>
<tr>
<td>D</td>
<td>50.7</td>
<td>63.2</td>
<td>77.0</td>
</tr>
</tbody>
</table>

![Fig. 1—X-ray diffraction patterns for the as-dried samples A, B, C, D](image)

![Fig. 2—Variation of magnetization with the applied magnetic field for as-dried samples A, B, C, D.](image)
and 4 emu/g because of the change in the drying condition. These drying conditions do not favour the crystallization/ferritization reaction and, therefore, it does not result in any phase formation of ferrite as seen from XRD plots (Fig. 1B / 1C).

The hydrothermal treatment improves the $M_s$ value to >50.7 emu/g (sample D). The improvement in the sample D as compared to the $M_s$ value (=44 emu/g) of sample reported in Ref.1 is because of the hydrothermal treatment given to sample D followed by drying at 80°C. Drying the sample at 80±5°C in oven alone does not increase the $M_s$ value. But the pressure and temperature acting together in hydrothermal synthesis enhance the rate of chemical reaction, which facilitates the crystallization/ferritization leading to well defined spinel formation as confirmed by XRD.

However, on calcination to 1200°C for 4hr of all these samples, the saturation magnetization value at room temperature is observed to be >60emu/g confirming the evolution of well-defined Ni-Zn ferrite. $M_s$ value when measured at liquid nitrogen temperature increases to >73emu/g for all the samples, approaching the saturation value. This behaviour is also supported by observation of the identical XRD patterns. The calcination at sufficiently high temperature helped (in all cases) in enhancing the rate of solid state diffusion reaction leading to (structurally and chemically on a microscopic scale) formation of well-defined spinel ferrite.

This study shows that any change in the processing changes the completion of ferritization. The hydrothermal method enhances the magnetization value substantially, avoiding further high temperature calcination step. This major achievement seems to be very promising for synthesis of active powders even when there are forced changes in processing parameters.

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References