Direct use of celestite to prepare presintered \( \text{SrFe}_{12}\text{O}_{19} \) powders

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Abstract

Mineral celestite was directly used to prepare presintered single-phase Sr-ferrite powders for fabrication of sintered permanent magnets. The starting materials were high-purity domestic celestite, modified iron oxide and sodium carbonate. The role of sodium carbonate is firstly to promote the displacement solid-state reaction and secondly to lower the temperature during calcining stage. A vibrating sample magnetometer was used to measure magnetic parameters of the powders. The results were compared with those of the powders prepared by conventional ceramic process. Lower reaction temperature of 1000 °C and low-cost celestite show that this process is more economical for production of presintered \( \text{SrFe}_{12}\text{O}_{19} \) powders than the conventional one. © 2002 Elsevier Science B.V. All rights reserved.

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1. Introduction

The most important factors in the production of strontium ferrite (\( \text{SrFe}_{12}\text{O}_{19} \)) apart from the processing method are the cost and availability of the raw materials [1]. The usual raw materials used commercially for production of strontium ferrites are iron oxide and strontium carbonate [2]. The iron oxide is generally derived from Ruthner process in steel plants [3] and strontium carbonate is mainly derived from mineral celestite [4,5]. One of the ways to reduce the cost of production is to use celestite directly for strontium carbonate. In this work we have used refined iron oxide by-

product of Mobarakeh Steel Complex and mineral celestite from Kashan mines located in central Iran to prepare strontium ferrite powders, suitable for production of sintered magnets.

2. Experimental procedure

Three mixtures of modified mineral celestite together with refined iron oxide and sodium carbonate with different mole ratios (Table 1) were used to synthesize single-phase strontium ferrite. The mixtures denoted by S8, S11 and S14 were milled in water and atmospheric air for 24 h, using rotary ball mill with a ball to powder mass charge ratio of 6:1. The milled powders were then calcined at an optimum temperature of 1000 °C for
10 h. After cooling, the melt chunks were crushed and milled again such that they could pass through a 180 μm mesh. The meshed powders were then washed few times in distilled water. The particles, which were magnetic at room temperature, were collected by means of magnetic separation, while the compounds dissolved in water and nonmagnetic particles in suspension were discarded. Any residual salts were finally washed and removed with a dilute solution of HCl.

The crystal structure of presintered powders was specified by X-ray diffraction (XRD), using Cu Kα radiation. Particle size and morphology were studied by scanning electron microscopy (SEM), using a Philips XL30 model. The magnetic measurements on cold pressed pellets 5 mm in diameter and about 0.5 mm in thickness were made at room temperature, using a vibrating sample magnetometer (VSM) with a maximum field of 5 T. The as-milled powders were annealed at 900°C for 3 h to remove any surface strains due to ball milling. The powders were then wet pressed anisotropically, dried and were sintered at 1200°C for 3 h. Magnetic parameters of the sintered magnets were measured by a permeameter with a maximum field of 1.2 T.

### 3. Results and discussion

The spectra of the powders show that they are single phase and Fig. 1 shows a typical XRD spectrum of S11 sample.

### Table 1

<table>
<thead>
<tr>
<th>Parameters</th>
<th>S8 Presintered</th>
<th>S11 Presintered</th>
<th>S14 Presintered</th>
<th>Conventional method presintered</th>
<th>S11 Sintered at 1200°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Celestite (wt%)</td>
<td>15.28</td>
<td>14.28</td>
<td>15.13</td>
<td>—</td>
<td>14.28</td>
</tr>
<tr>
<td>Fe₂O₃ (wt%)</td>
<td>73.25</td>
<td>74.25</td>
<td>72.52</td>
<td>—</td>
<td>74.25</td>
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<tr>
<td>Na₂CO₃ (wt%)</td>
<td>11.74</td>
<td>11.47</td>
<td>12.32</td>
<td>—</td>
<td>11.47</td>
</tr>
<tr>
<td>σₘ (emu/g)</td>
<td>72.0</td>
<td>69.9</td>
<td>73.3</td>
<td>60.3</td>
<td>61.2</td>
</tr>
<tr>
<td>σₜ (emu/g)</td>
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<td>32.2</td>
<td>32.8</td>
<td>30.1</td>
<td>56.5</td>
</tr>
<tr>
<td>Hₘ (Oe)</td>
<td>1938</td>
<td>1760</td>
<td>2048</td>
<td>5230</td>
<td>3022</td>
</tr>
</tbody>
</table>

Fig. 1. XRD spectrum of S11 sample.
spectrum of the washed sample S11 at 1000°C. The peaks in the spectrum indicate that the reaction has completely occurred during the calcining stage and a single-phase SrFe$_{12}$O$_{19}$ has been obtained.

A SEM of S11 sample is shown in Fig. 2. The micrograph shows that the particles are hexagonal platelet crystals with average diameter of 0.5–2 µm. Fig. 3 shows a part of the hysteresis loops of the samples. Numerical values of the saturation magnetization $\sigma_s$, remanent $\sigma_r$ and coercive force $H_c$ are given in Table 1. As can be seen the values of $\sigma_s$, and $\sigma_r$ are higher than the values of the powders that were prepared by conventional ceramic process ($\sigma_s = 60.3$ emu/g, $\sigma_r = 30.1$ emu/g and $H_c = 5230$ Oe) [1], whereas $H_c$ values are much smaller.

This discrepancy is due to the fact that, $H_c$ is a function of particle size, annealing temperature and packing factor. The single-domain size for Sr-ferrite powders is about 1 µm [6], which in our powders varies from 0.5 to 2 µm. The variation of $H_c$ with particle size can be discussed on the basis of single-domain theories [7]. Also by annealing the prepared powders, the value of $H_c$ will drastically increase [8]. The magnetic parameters quoted in Table 1 are related to cold pressed pellets (low packing factor) of unannealed powders. To see the effect of particle size, annealing temperature and packing factor, a sintered magnet was prepared, using S11 powders. The magnetic parameters of the magnets quoted in Table 1, show reasonable agreement with those related to other sintered magnets [1]. During sintering process, small particles have grown to single-domain size which leads to higher $H_c$ [7], whereas lower $\sigma_s$ is due to the lower maximum field of 1.2 T used in permeameter. The results obtained in this work show that, due the lower reaction temperature and low cost of mineral celestite, the process is more economical than the conventional ceramic method.

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References