Microstructural study of $BaFe_{12}O_{19}$ fine particles obtained from a milled precursor

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Abstract

$BaFe_{12}O_{19}$ fine particles were synthesized after milling an intermediate step precursor in the sol-gel method. The milling process results in a better transformation of the initial mixture into BaM ferrite compared to the non-milled precursor heat treated at the same temperature. Microstructural study by SEM of the modification of the intermediate step precursor upon milling is carried out. The observed structural modifications are discussed with regards to its influence in the final obtained BaM hexaferrite.

keywords: BaM, milling, solid state phase transformation, sol-gel method, g-Fe_2O_3, a-Fe_2O_3

1. Introduction

The hexagonal M-phase ferrite $BaFe_{12}O_{19}$ has been studied intensively for many years due to their importance as permanent magnets, potential high-density magnetic recording and other application [1]. Some of these applications require a strict control of homogeneity, particle size and shape and crystalline perfection, which governs their magnetic behavior.

The sol-gel route has emerged as a new method for synthesizing barium ferrite [2]. By this route the final BaM powder exhibits a fine grain size with a narrow grain size Recently, R. Martinez et al. reported a modification of the sol-gel technique [3]. They introduced ball milling in a intermediate step of the processing route. After eliminating the organic precursors at 450°C, the resulting mixture is milled for several hours. The milled precursor was then heat treated at different temperatures. As a result of milling the resulting sample showed improved magnetic properties compared to the non-milled sample heat treated at the same temperature. The milling stage seemed to favor the synthesis of the BaM hexaferrite at lower temperature resulting in a better specific magnetization. The possibility of lowering the synthesis temperature an still get a pure BaM powder allows the reduction of the grain size in the final product.

In this paper we study the microstructure modifications of the intermediate precursor upon milling and in the final products in order to explain the observed effect on the final BaM powder.

2. Materials and Methods

A precursor of barium hexaferrite was prepared by the sol-gel method using ethylenglycol as coordinating agent and atomic Fe:Ba ratio of 12. Details of this method are given in [2]. Briefly, iron and barium nitrates in a Fe:Ba ratio of 12:1, were dissolved in ethylene glycol at 40°C. After heating the sol of the dissolved metal compounds to around 100°C, a wet gel derives during an exothermal reaction with increase of temperature up to 130°C. The obtained gel is dried at 250°C and then warm up to 450°C, which eliminates the organic precursors. The mixture was then divided into several samples and milled in a ball mill for 2h, 6h and 10h.

The intermediate precursors of the non-milled and 10h-milled samples were heat treated at 800 °C in order to obtain BaM hexaferrite.

Samples of the intermediate precursor were studied by scanning electron microscopy (SEM) in a Jeol JSM-6300 equipped with a energy dispersive Si-detector.

3. Results

X-ray powder diffraction patterns of the precursors are shown in figure 1. The non-milled precursor is formed by $BaCO_3$ and α-Fe_2O_3 (maghemite), upon milling the maghemite