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Structural transformation with milling on sol–gel precursor for BaM hexaferrite

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Abstract. The structural modification upon milling in an intermediate step precursor of the sol–gel method for $BaFe_{12}O_{19}$ hexaferrite BaM production is discussed. The milling of the precursor diminishes the powder particle size, leads to a more homogeneous matrix and induces a solid-state transformation, from γ-Fe_2O_3 to α-Fe_2O_3. The induced modifications of the precursors change the magnetic and structural properties of the final BaM hexaferrite compared to the BaM obtained from the non-milled precursor.

1. Introduction

In the last few years nanocrystalline magnetic materials have proven to develop exciting magnetic behaviours that are attractive for a wide range of applications. Alternative routes of production to the ceramic method, such as chemical coprecipitation, crystallization of glass, radiothermal synthesis and the sol–gel method among others, have emerged as suitable methods for the production of nanocrystalline $BaFe_{12}O_{19}$ hexaferrite BaM powders with a narrow grain size distribution, single-domain behaviour and perfect crystal structure [1–4].

The sol–gel technique is one of these new chemical routes which, allows good control of the structural and magnetic characteristics of the final BaM powder [4]. Mechanochemistry has also been used as a valid route for BaM nanoparticle production [5]. In this technique, sintered BaM particles are milled in order to reduce the grain size down to the nanometre scale. The powder after milling exhibits a large disruption of its structural characteristics with extensive crystalline disorder. Additional heat treatment is then given in order to eliminate the amount of amorphisized phase and reduce the crystalline imperfections. A compromise must be reached between the grain growth and the attained crystalline perfection. The grain size distribution is usually not as well controlled as in other chemical routes.

Recently, Martinez *et al* [6] reported a modification of the sol–gel technique. They introduced ball milling into 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 favour the synthesis of the BaM hexaferrite at a lower temperature, resulting in a better specific magnetization. The possibility of lowering the synthesis temperature and still obtaining a pure BaM powder allows the reduction of the grain size in the final product.

In this paper we study the structural modification of the intermediate precursor upon milling in order to explain the observed effect on the final BaM powder.

2. Experiment

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 is derived during an exothermal reaction with an increase of temperature up to 130 °C. The obtained gel is dried at 250 °C and then heated up to 450 °C, which eliminates the organic precursors.

The mixture was then divided into several samples and milled in a vibrating ball mill (Spex 800) for 2, 6 and 10 h. Samples were studied by x-ray diffraction (XRD), infrared (IR) spectroscopy, differential thermal analysis (DTA), Mössbauer spectroscopy and scanning electron

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