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Effect of sintering temperature on the structural and magnetic properties of $MgFe_2O_4$ ceramics prepared by spark plasma sintering

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Abstract

Spark plasma sintering (SPS) is a powerful technique to produce fine grain dense ferrite at low temperature. This work was undertaken to study the effect of sintering temperature on the densification, microstructures and magnetic properties of magnesium ferrite ($MgFe_2O_4$). $MgFe_2O_4$ nanoparticles were synthesized via sol–gel self-combustion method. The powders were pressed into pellets which were sintered by spark plasma sintering at 700–900 °C for 5 min under 40 MPa. A densification of 95% of the theoretical density of Mg ferrite was achieved in the spark plasma sintered (SPSed) ceramics. The density, grain size and saturation magnetization of SPSed ceramics were found to increase with an increase in sintering temperature. Infrared (IR) spectra exhibit two important vibration bands of tetrahedral and octahedral metal-oxygen sites. The investigations of microstructures and magnetic properties reveal that the unique sintering mechanism in the SPS process is responsible for the enhancement of magnetic properties of SPSed compacts.
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1. Introduction

Ferrite magnetic nanoparticles with a spinel structure have been widely used in many fields due to their outstanding electrical, optical and magnetic properties. Spinel type ferrites, with general formula MFe_2O_4 (M = Mg, Co, Ni, Zn, Cu, Mn etc.) are receiving increasing interest in recent years because of their stable structure, low cost and ease of formation. These materials offer a wide range of potential applications in telecommunication systems, microwave devices, computer memory chips, transmitting microwaves, heat transfer, low-magnetic materials due to their low magnetic loss, high resistivity and saturation magnetization [1]. The structural, magnetic, electrical and optical properties of ferrites are highly sensitive to chemical composition, the method of preparation and sintering time and temperature, etc. [2].

Among spinel ferrites, magnesium ferrite ($MgFe_2O_4$) is a soft magnetic n-type semiconducting material which is one of the most important and a plentiful magnetic material owing to its outstanding properties [3]. Recently, magnesium ferrite has attracted a lot of attention because of its excellent magnetic properties and potential applications in various fields such as microwave absorption, catalysis, semi-conductors, sensors, refractories, and low-magnetic materials [4–10].

Many synthesis techniques have been developed to prepare magnesium ferrites such as solvothermal reduction method [11], electrospinning technique [12], co-precipitation [13], sol–gel-auto combustion method [14], microwave hydrothermal method [15], reverse micelle processing [16] and high-energy ball milling method [17]. Among them, an auto-ignited technique is a promising way to synthesize various nanoparticles. It is a

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unique combination of the chemical gelation process and the combustion processes. This method exploits the benefits of inexpensive precursors, simple groundwork, short preparation time and homogeneous highly reactive powder [18].

From the industrial viewpoint, a significant consideration is being paid to design a simple, economic and environment-friendly approach to synthesize $MgFe_2O_4$ nanoparticles. It is well known that the magnetic properties of ferrites strongly depend on the microstructure, size and morphology of particles which depends on the preparation techniques.

Spark plasma sintering embodies an efficient and novel sintering technique for the fabrication of nanomaterials/nanoparticles. Compared with the conventional sintering process, SPS process has the salient merits of fast sintering, lower sintering temperature and the sintering time can be reduced by several orders of magnitude which are useful for the improvement of physical, chemical and magnetic properties of the product [19]. It is well-established fact that the most significant aspect affecting the evolvement of the microstructure is the sintering temperature.

Up to date, synthesis of ferrite nanoparticles of Fe_2O_3 [20], M-type barium ferrite [21], $NiZnFe_2O_4$ [22], $ZnFe_2O_4$ composite [23], $CoFe_2O_4$ [24], NiCuZn ferrite [25] and $MnFe_2O_4$ [26] through spark plasma sintering process has been reported. However, importantly, synthesis of $MgFe_2O_4$ through spark plasma sintering process has not yet been reported. The present study focuses on the effect of spark plasma sintering temperature on the density, structural and magnetic properties of the $MgFe_2O_4$ synthesized through self-ignited sol–gel method.

2. Experimental procedures

Nanocrystalline powder of $MgFe_2O_4$ was prepared by the self-ignited sol–gel method and the schematic representation of the detailed preparation process can be seen in Fig. 1. Briefly, stoichiometric amounts of $MgN_2O_6 \cdot 6H_2O$, $Fe(NO_3)_3 \cdot 9H_2O$ and citric acid ($C_6H_8O_7 \cdot H_2O$) were dissolved in deionized water under constant stirring to form a mixed solution at ambient temperature. The molar ratio of metal nitrate and citric acid was 1:1. Ammonia was slowly added to adjust the pH to 7. During the hydrolysis process, better condensation occurrs as the pH value of the aqueous solution approaches to 7 leading to the formation of nanocrystallites through single step decomposition process. The mixed solution was placed on a hot plate at 80 °C for 2 h with vigorous stirring. During the evaporation process the homogeneous sticky solution is finally converted into a gel. The so formed gel slowly bubbled, swelled (inset of Fig. 1) and finally burned on its own way to form a loose powder. The obtained powder was then sintered by spark plasma sintering (SPS) using a uniaxial pressure of 50 MPa under argon atmosphere.

The spark-plasma sintering experiments (SPS) [26] were performed in an SPS apparatus (Dr. Sinter Model 1050, Sumitomo Coal Mining Co. Ltd., Japan) in argon gas. The magnesium ferrite particles were loaded in a graphite die (10 mm in diameter) and punch unit. Graphite foils were used as spacers between the specimen and the graphite die and punches. The system was closed by carbon punches at both sides which transmit the uniaxial pressure. DC pulses were delivered to the die by the punches allowing the temperature to rise rapidly (about 100 °C/min). A low pressure of 30 MPa (2.0 kN) was initially applied. Each specimen was first heated to 500 °C, exposed for 1 min, and then heated to the present temperature (700, 800 and 900 °C) at heating rates of 100 °C min^{-1} at initial current of 300 A. The temperature was measured using a pyrometer. The specimen was gradually cooled down to 500 °C at a cooling rate of 50 °C min^{-1} and then furnace-cooled to room temperature.

After SPS sintering, the graphite sheet on the sample was scraped out and the pellets (10 mm in diameter and 2 mm in thickness) were annealed in air at 200 °C lower than the SPSed sample for 2 h to remove carbon contamination. The rate of heating and cooling in all heat treatments was maintained at 5 °C min^{-1}. For the whole process, it only took a short time to attain final ceramics.

The phase purity and structure of the precursor and SPSed products were recorded by Phillips X-ray diffraction (XRD, Bruker AXS-D8, Germany) with Cu-Kα radiation ($\lambda = 1.5405$

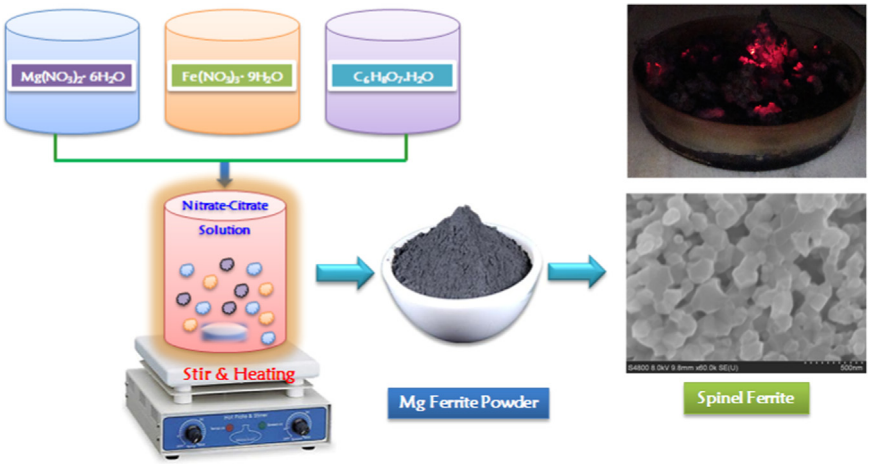

Fig. 1. Flowchart for the preparation of $MgFe_2O_4$ spinel ferrite by nitrate–citrate sol–gel auto-ignition method.

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