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Synthesis of yttrium iron garnet (YIG) by citrate–nitrate gel combustion and precursor plasma spray processes

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Abstract

The influence of synthesis conditions on the formation of yttrium iron garnet (YIG) powders starting from the same precursor solution was investigated by employing a citrate–nitrate (C–N) gel combustion process and a precursor plasma spraying technique. Two different C–N ratios were used in the synthesis and their influences on phase formation were studied by thermal analysis (DTA/TGA) and X-ray powder diffraction (XRD). Time-resolved powder XRD experiments were performed for the first time on these C–N precursor materials to understand their mode of decomposition. For a C–N = 0.75, the ex-situ XRD data confirmed a single-step conversion to YIG from the amorphous precursor powder without any intermediate phase formation. However, the use of the same C–N precursor solution as a liquid feedstock material in the precursor plasma spraying (PPS) technique revealed an entirely different transformation mechanism to YIG through the intermediate phase YFeO_3 . The measured values of saturation magnetization (M_s) as well as coercive field (H_C) of the powder samples annealed at 1500 °C are close to those reported for bulk YIG.

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1. Introduction

Yttrium iron garnet ($\text{Y}_3\text{Fe}_5\text{O}_{12}$, YIG) and derived compounds constitute a very important

class of materials due to their magnetic and magneto-optic properties leading to several applications in telecommunications and data storage. Numerous studies reported the synthesis of YIG, in bulk polycrystalline, single crystal and in thin film forms. The conventional method of preparing YIG powders from the corresponding oxides (Y_2O_3 and Fe_2O_3) is a very tedious process requiring high processing temperatures followed by a grinding operation, yielding large particles of several tens of micrometers. Nevertheless in recent years, soft-chemical routes have been developed to circumvent the synthetic difficulties to prepare fine particles of phase pure garnet powders. A variety of soft chemical approaches including sol–gel, co-precipitation, and glycothermal synthesis have been reported to prepare submicron to nanocrystalline powders of YIG [1–5]. It is well known that deviations from stoichiometry have a strong influence on the magnetic properties of the ferrites. Additionally, the magnetic properties of a given particle also depend in a sensitive manner on size and shape.

Vaqueiro et al. [1] reported the dependence of magnetic properties of YIG particles prepared by citrate–nitrate (C–N) gel process on annealing. In sol–gel methods, the preparation of a homogeneous gel with respect to the distribution of cations is very important to achieving the compositional homogeneity of final oxide powder. In C–N methods, the role of citric acid is to form a complex with metal ions and is expected to reduce segregation of cations. Recently we have reported a C–N gel combustion method to prepare fine powders of yttrium aluminum garnet (YAG) [6] and lanthanum strontium manganate (LSM) [7]. The combustion process has the advantage of using inexpensive precursors in a very simple way to produce nanocrystalline materials. Of late, there have been many reports on the auto-combustion process to synthesize fine particles of ceramic oxides [8–11]. Formation of mixed Y–Fe(III) citric complexes and their thermal decomposition were studied by Todorovsky et al. [12]. Influence of two different complexing agents (citric acid and malonic acid) and pH on the synthesis of YIG has also been reported [13]. Preparation of YIG films by spray pyrolysis was reported by Deschanvres et al. [14].

Recently, using radio-frequency (RF) plasma, we developed a novel precursor plasma spraying (PPS) technique to deposit thin/thick coatings of spherical, nano- to micron-sized deposits of yttrium aluminum garnet, which basically avoids the handling and selection of powders [6,15]. Currently we are making efforts to directly synthesize YIG coatings from precursor solutions using this technique. No data were found in the literature on the application of thermal spray method for the preparation of YIG coatings. Obviously, a good knowledge of thermal decomposition of the precursors is of prime importance for the optimizing the thermal spray parameters.

In this paper, we report the effect of different (C–N) ratio on the conversion mechanism from the combustion formed precursor to crystalline YIG. The same precursor solution was further utilized to explore its use in PPS technique to produce garnet coatings and powders. An in situ X-ray monitoring of the crystallization of YIG from an amorphous C–N precursor, using synchrotron radiation, is also reported for the first time.

2. Experimental method

$Y(NO_3)_3 \cdot 6H_2O$ (Aldrich, 99.9%), $Fe(NO_3)_3 \cdot 9H_2O$ (Aldrich, 99.9%), citric acid mono hydrate (Alfa Aesar, 99.5%), de-ionized water and ethanol were used for the synthesis of materials. All chemicals were used as received without further purification.

The YIG precursor solution was prepared by dissolving and mixing the required amount of the metal nitrates in a stoichiometric ratio of Y:Fe = 3:5 in a 1:1 ethanol de-ionized water mixture. An appropriate amount of citric acid in water was added to the above mixture while stirring. The ratio between nitrates was kept constant, corresponding to the stoichiometric YIG composition, where as 2 mol ratios of citric acid to nitrates (C–N) were prepared; one with C–N = 0.15 and the other with C–N = 0.75. The solution obtained above was divided into two portions with one being transferred to an alumina crucible where it was allowed to evaporate on a

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