



## Original Research Paper

# Synthesis and characterization of yttrium aluminum garnet nanostructures by cathodic electrodeposition method

Mojtaba Hosseinifard<sup>a</sup>, Alireza Badiiei<sup>a,\*</sup>, Kamran Ahmadi<sup>b</sup><sup>a</sup> School of Chemistry, College of Science, University of Tehran, Tehran, Iran<sup>b</sup> Semiconductors Department, Materials and Energy Research Center, P.O. Box 14155-4777, Tehran, Iran

## ARTICLE INFO

## Article history:

Received 31 July 2016

Received in revised form 1 October 2016

Accepted 12 October 2016

Available online 15 November 2016

## Keywords:

Cathodic electrodeposition

Hydroxide

Nanostructure

Yttrium aluminum garnet

## ABSTRACT

Pure nanostructures of yttrium aluminum garnet (YAG) was prepared based on the cathodic electrodeposition method from the mixture of  $YCl_3$  and  $AlCl_3$  dissolved in water/ethanol solution. At first, hydroxide precursors cathodically were grown on the steel substrates then, the hydroxide powders heat treated at  $850\text{ }^\circ\text{C}$  for 4 h in dry air atmosphere. The formation of crystalline YAG nanopowder was confirmed by X-ray diffraction (XRD), thermogravimetric analysis (DSC-TGA), scanning electron microscopy (SEM) and Fourier transformed infrared spectroscopy (FT-IR). The results of the SEM showed that applied current density and bath temperature have the prominent effect on the morphology and particle size of the products. The results revealed that cathodic electrodeposition followed by heat-treatment can be used as a facile method for preparation of YAG nanostructures with different morphology.

© 2016 The Society of Powder Technology Japan. Published by Elsevier B.V. and The Society of Powder Technology Japan. All rights reserved.

## 1. Introduction

Yttrium aluminum garnet ( $Y_3Al_5O_{12}$ ), or YAG, is a crystal with low-thermal expansion, high-optical transparency, low-acoustic loss and a high threshold for optical damage [1,2]. Due to its structural, optical and luminescence properties, it can be considered as a solid-state laser, phosphor, and window material for several of lamps [3–5]. Its hardness as well as its thermal and chemical stability makes it suitable to use in a widely of high-temperature ceramic composites [6–8]. YAG-based phosphors doped with different rare earth ions are also promising phosphor candidates in contrast enhanced display application and scintillation [9,10]. For application of YAG powder in applied fields it is necessary to achieve circumstances such as obtaining pure phase YAG powders with optimum morphology, mono-dispersed spherical shape, suitable average size and narrow size distribution. However, obtaining YAG in a single phase is not a simple task even when strictly adhere with the stoichiometric ratio to the ingredients. This is due to the three well-known intermediate compounds in the  $Y_2O_3$ – $Al_2O_3$  binary system of the crystal including yttrium aluminum monoclinic ( $Y_4Al_2O_9$ , YAM), yttrium aluminum perovskite ( $YAlO_3$ , YAP), and  $Y_3Al_5O_{12}$ (YAG) [11].

YAG is prepared by conventional solid-state reaction at high temperatures ( $>1600\text{ }^\circ\text{C}$ ) between aluminum oxide and yttrium oxide and repeated mechanical mixing to obtain pure phase YAG powders [12,13]. These processes lead to large particle sizes and heavy agglomeration. Furthermore, the conventional process has some disadvantages i.e. inappropriate particle sizes, stoichiometry impurities, the formation of undesirable phases, etc. While high temperature sintering is necessary for crystallization, it can introduce defects and impurities [14,15]. Therefore, various synthesis methods have been developed to fabricate YAG-based materials for overcoming the drawbacks of the solid-state reaction process.

YAG nano-powders allow for the careful control of the final grain structure in dense polycrystalline YAG used for structural applications, whereas nano-sized spherical particles allow for higher definition and brightness in phosphor applications [16–19]. Sintered micron-sized YAG powders also provide efficient, transparent, polycrystalline YAG lasers. In addition, a wide variety of nano-powders exhibits lasing properties not obtained with micron-sized powders. Hence there is significant motivation to develop methods for preparing large-scale quantities of high-quality YAG nano-powders. In recent years, many techniques have been developed and successfully used to synthesize YAG nano-powders including microwave-assisted [20], solvothermal [21], sol-gel [22,23], co-precipitation [24–26], spray-pyrolysis synthesis [27,28], and solid-state combustion methods [29,30]. Notably, the critical issue for the preparation of YAG transparent optical ceram-

\* Corresponding author. Fax: +98-21-61112614.

E-mail address: [abadiei@khayam.ut.ac.ir](mailto:abadiei@khayam.ut.ac.ir) (A. Badiiei).

ics and YAG-based phosphors is to synthesize high-quality powders with fine particle size and good disparity. Although YAG is the thermodynamically stable phase; and kinetically favored phases (hexagonal YAP, cubic YAG and monoclinic YAM). For example, hexagonal YAP and orthorhombic YAP are the common kinetic phases formed by using gas-phase synthesis techniques. Moreover, efforts to transform these nano-powders to phase-pure YAG by heating has led to coincidental particle necking followed by excessive grain growth. Wet-chemical routes such as co-precipitation, sol-gel, combustion, and spray pyrolysis have been proposed to lower the temperature in order to obtain single phase YAG with fine particle size. However, the aggregation is still inevitable due to the post heat treatment in these methods.

Among the different methods, the electrochemical method is a new and completely unique approach to overcome the difficulties of the above-mentioned methods. The other advantages of this method are energy reduction, simplicity, flexibility and suitability for the preparation of nanostructures metal oxides and hydroxides. The electrochemical route, i.e. cathodic electrodeposition, can be used as an effective method for the synthesis of YAG due to its powerful control on the properties of the final products. In this method, metal hydroxide as a precursor is prepared by electrodeposition process and then converted into metal oxide via heat-treatment. In the electrodeposition process, the rates of electrochemical reactions, nucleation and growths can be controlled by changing various experimental parameters such as current density, potential, bath temperature, and reaction medium. These parameters can be adjusted in order to design new products with the desired crystallinity and morphology [31–33]. Various metal oxides forms of  $Y_2O_3$  e.g. thin films, nanoparticles, nanospheres and nanorods have been fabricated via electrochemical method in recent years [34–37] and there are few reports on the electrochemical synthesis of YAG [38] but to the view best of our knowledge is the effects of different current densities and bath temperatures on the morphology, crystal structure and particle size of YAG nanostructure this characteristic haven't been studied yet. In this work, YAG nano-powder was prepared via cathodic deposition followed by heat-treatment and the effect of different current densities and bath temperatures on the morphology and particle size of YAG nanostructure was investigated.

## 2. Experimental procedure

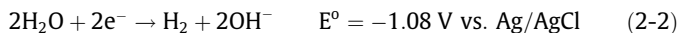
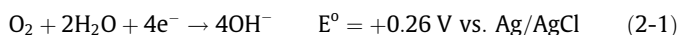
### 2.1. Chemicals

Yttrium chloride hexahydrate ( $YCl_3 \cdot 6H_2O$ ) and anhydrous aluminum chloride ( $AlCl_3$ ), (Aldrich) were used as raw materials without further purification. All solutions were prepared in a mixture of water/ethanol solutions [39]. An aqueous solution of 0.005 M  $YCl_3$ , 0.0083 M  $AlCl_3$  was prepared for electrodeposition.

### 2.2. Sample preparation

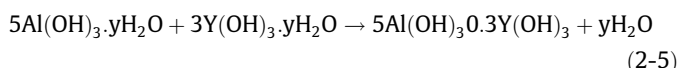
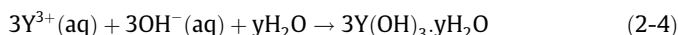
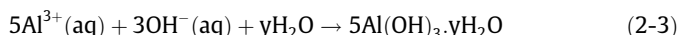
The electrochemical cell included a cathodic stainless-steel (316L, size 1 cm × 1 cm × 0.3 mm) substrate centered between two parallel graphite anodes. Prior to each deposition, the steel substrates were given a galvanostatically electropolishing treatment at a current of 20 A for 5 min in a bath (70 °C) containing 50 vol.% phosphoric acid, 25 vol.% sulfuric acid and balanced deionized water. The experimental conditions have been listed in Table 1. All the deposition tests performed indifferent current densities and bath temperatures. According to the required stoichiometric ratio of YAG, the solutions of 0.005 M  $YCl_3$  and 0.0083 M  $AlCl_3$  were dissolved in a water/ethanol solution, therefore used as an electrolyte.

The hydroxide is formed on the cathode surface through electrochemical steps as shown in Eqs. (2-1) and (2-2) [45–48]



Measuring the potential values during the deposition process (−0.98 V vs. Ag/AgCl) revealed that the reduction of water had a major role in the base electrogeneration at our experimental conditions.

Then according to Eqs. (2-3)–(2-5)  $Al^{3+}$  and  $Y^{3+}$  cations can react with produced hydroxide ions at the cathode surface, results in formation and deposition of hydroxide gel on the surface of cathode. The overall steps are schematically shown in Fig. 1.



After the electrodeposition, the steel substrates dried at 25 °C for 5 h. Finally, the deposits were separated from the substrates and heated under the air flow at 850 °C for 4 h.

### 2.3. Characterization

The crystal structure of the prepared samples was investigated by X-ray diffraction (XRD), and it was recorded on a Phillips PW-1800 diffractometer with a  $Cu K_\alpha$  radiation in the  $2\theta = 10\text{--}65^\circ$  ranging with a scanning rate of 5 deg/min. FTIR spectra of the samples were obtained using the Bruker Vector 22 Fourier transformed infrared spectroscopy with samples in a KBr wafer at ambient temperature, in the range of 400–4000  $cm^{-1}$ . The FTIR spectra were acquired after 20 scans at a resolution of 4  $cm^{-1}$  from 400 to 4000  $cm^{-1}$ . The morphologies of the prepared YAG powders were studied using the scanning electron microscope (SEM, LEO 1455 VP, Oxford, UK, operating voltage 30 kV). The thermogravimetric analysis of the hydroxide deposit was performed using the TGA/DSC1- METTLER TOLEDO- STARE in oxygen flow between 25–1100 °C with a heating rate of 10 °C/min.

## 3. Results and discussion

### 3.1. Structural analysis

The crystal structure of the prepared samples was investigated by X-ray diffraction (XRD), FTIR spectra, thermogravimetric analysis (TGA-DSC), scanning electron microscope (SEM) and results showed the formation of pure YAG.

#### 3.1.1. XRD

Fig. 2a–d shows the XRD patterns of prepared powders that were calcined at various temperatures for 4 h in a mixture of water/ethanol. According to the yielded results, diffraction peaks of the samples which calcined at 500 and 650 °C were not significant. The results indicating that the powders are amorphous in these temperatures but the formation of crystalline YAG phase can be confirmed by XRD analysis at higher temperatures (>750 °C). As shown in Fig. 2c when the calcinations temperature increased to 750 °C the XRD diffraction peaks was appeared that can be refer to YOOH and AlOOH structure (JCPDS card No. 074-2350, 005-0355). By increasing the calcination temperature to 850 °C, all of the observed diffraction peaks fully match with pure

Download English Version:

<https://daneshyari.com/en/article/6464619>

Download Persian Version:

<https://daneshyari.com/article/6464619>

[Daneshyari.com](https://daneshyari.com)