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Effect of titania on yttrium aluminum garnet fibers morphology development

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ABSTRACT

The titania-YAG (yttrium aluminum garnet) fibers were prepared by sol-gel method using aluminum chloride, titanium chloride, aluminum powder, yttrium oxide and acetic acid as raw materials. The effects of titania on the YAG fiber morphology development were studied. The result indicated titania could promote YAG grain growth. Titania-YAG fibers were obtained by sintering at 1000°C for 2 hours, with fine grain. But the fibers were obtained by sintering at 1400°C for 6 hours, with rough surface and coarse grains. © 2013 Trade Science Inc. - INDIA

INTRODUCTION

Yttrium aluminum garnet (YAG or $Al_5Y_3O_{12}$) possesses a cubic structure and constitutes a complex oxide of Al₂O₃ and Y₂O₃. YAG fibers, with low creep rates, high tensile strength, high module, excellent thermal stability, thermal shock resistance, oxidation and reduction atmosphere resistance at high temperatures, were widely used as high temperature structural materials and as a reinforcing phase to reinforce composites^[1].

Two main processes for the manufacturing of ceramic fibers existed, including melt-spinning processes and sol-gel spinning processes^[2]. López1 et al^[3] prepared YAG fibers by melt extraction technique. The calcined YAG powders were mixed with a plasticizer to extrude 3-mm-diameter rods that were dried in air for 24 h at room temperature. The rods were then sintered at 1500°C for 1 h to give them enough resistance for handling. The sintered rods were melted using an oxy-acetylene torch to form a small molten drop be-

neath a rotating Cu-Be wheel. The shallow contact of the wheel tip with the molten drop resulted in rapid solidification and formation of the fibers. Mileiko et al^[4] produced single crystalline YAG fibers by an internal crystallization method. The method was crystallization of the oxide melt infiltrated into continuous channels made in an auxiliary matrix, normally molybdenum, and then extracting the fibers from the auxiliary matrix by chemical dissolution of it.

Conventionally, melt-spinning methods are adopted for the synthesis of ceramic fibers with low-melting point, so it is difficult to prepare YAG fibers due to the high melting points (1970°C).

Many successful processes have been reported in the preparation of YAG fibers by the sol-gel method. Li et al^[1] prepared YAG fibers by sol-gel method using Al powder, Y(CH₃COOH)₃•4H₂O and HCl as raw materials, polyethylene oxide as viscosity adjusting agent and water as the solvent. YAG fibers were obtained by sintering at 900°C, the fibers with 25nm in grain size

KEYWORDS

YAG: Fibers: Sol-gel method; Titania.

MSAIJ, 9(7) 2013

Full Paper

and 970MPa in tensile strength. Pullar et al^[5] obtained sp

YAG fibers using alumina sol and yttria sol as precursors. The alumina sol was made from aluminium sulphate, and the yttria sol was made from yttrium chloride. Towata et al^[6] synthesized YAG fibers by sol-gel method, using aluminum isopropoxide and yttrium isopropoxide as raw materials, isopropanol solutions as solvent.

One of the directions of modification of ceramics in order to attain desirable properties is creating a secondary crystalline phase^[7]. The sintering temperature of YAG can be decreased and the YAG properties may be improved by adding low melting point phase (TiO₂)^[8].

It is desirable to using the raw materials with low cost but attribute high fiber quality. In the present work, long YAG fibers were prepared by the sol-gel method using aluminum powder, aluminium chloride and yttria as raw materials. The effect of titania on the YAG fiber morphology development was studied.

EXPERIMENTAL PROCEDURE

Preparation of samples

Starting materials used were aluminum powder (Chemically grade, Shanghai chemistry Co. Ltd., Shanghai, China), aluminum chloride hexahydrate (Chemically grade, Xi'an reagent factory, Xi'an, China), yttria (99.99wt%, Wanbao rare-earth Co. Ltd, Ganzhou, China), glacial acetic acid (Chemically grade, Tianjin Yaohua chemistry Co. Ltd., Tianjin, China), titanium chloride (Chemically grade, Yuda Reagent chemistry Co. Ltd., Shangyu, China) and polyvinylpyrrolidone (Chemically grade, Sinopharm Chemical Reagent Co. Ltd, Shanghai, China).

The titania-YAG fibers were prepared in the processing steps as shown in Figure 1. The yttria powder, titanium chloride, aluminum powder, and aluminum chloride were dissolved in acetic acid solution when the mixtures were heated and stirred using magnetic stirring under reflux at 80°C and then the precursor sol was obtained (a molar ratio of Al and Y, CH₃COOH and Y and H₂O and Al was 5:3, 3:1, 20:1 in precursor sol, respectively). Other more, an amount of titanium chloride was 1mol% in the YAG precursor sol. Then, spinning sols were obtained by condensing at 60°C.



Figure 1 : Schematic view of the production route for titania-YAG fibers.

The gel fibers were prepared by pulling a thin glass rod slowly from the sol after immersing. Then the gel fibers were dried at 60°C for 24 h in an oven. The dried gel fibers were then sintered at 1000 and 1400°C, with heating rate of 1°C/min.

Characterization techniques

X-ray diffraction analysis was carried out on an Xray diffractometer (DX-2500, Dandong Fangyuan instruments Co. Ltd., Dandong, China) using CuK α radiation with a step of 0.1°/s. The morphologies of fibers were characterized by scanning electron microscopy (JSM-6390LV, JEOL, Japan). All tests were done at room temperature.

RESULTS AND DISCUSSION

Aluminum chloride hexahydrate was firstly hydrolyzed with water and formed aluminum hydroxide in acid solution. The chemical reaction can occur as follows:

$$AlCl_3 + 3H_2O \rightarrow Al(OH)_3 + 3HCl$$
(1)

Hydrolysis reaction occurred because water molecules coordinated to metal ions were more acidic than in the noncoordinated state due to charge transfer from the oxygen to the metal atom^[9]. Yttria and aluminum pow-

Materials Science Au Indian Journal

(2)



der were dissolved during the stirring and heating in the mixed solution of aluminum chloride and acetic acid, and its main chemical reactions can be simplified in the following equation, though the actual reactions were complexity:

 $Y_2O_3 + 6HCl \rightarrow 2YCl_3 + 3H_2O\uparrow$

$$Al + 3HCl \rightarrow AlCl_3 + 3/2H_2 \uparrow$$
(3)

 $3CH_3COOH + Al \rightarrow Al(CH_3COO)_3 + 3/2H_2^{\uparrow}$ (4)

In addition, yttrium chloride was also hydrolyzed with water and formed yttrium hydroxide in the acid sol. The chemical reaction can occur as follows:

$$YCl_3 + 3H_2O \rightarrow Y_2(OH)_3 + 3HCl$$
(5)

The viscous sol was obtained by concentrating because poly-nuclear species are formed by condensation reactions (olation and oxolation) and formation of M– OH–M and M–O–M with linear or non-linear links^[9]. But, long fibers can be obtained, with 30cm in length, only by adding 1wt% PVP as spinning additive. Al, Y and Ti ions or particles would coordinate with N or O ions in PVP, resulting in the formation of the coordinative complex in aqueous solution. The reactions can be written as (6) and (7), Al³⁺ as an example^[10,11].

The X-ray diffraction patterns of gel fibers sintered at 1000°C are shown in Figure 2. YAG phase was obtained, but contained titania phase was not observed because the amount of titania was less and it was not detected by XRD.



Figure 2 : XRD pattern of the titania-YAG precursor gel fibers heated at 1000°C for 2h.

SEM micrograph of YAG and titania-YAG fibers sintered at 1000°C for 2h are shown in Figure 3. The YAG and titania-YAG fibers were obtained with all smooth surface.



(a) YAG fiber

(b) Titania-YAG fiber



SEM micrograph of YAG and titania-YAG fibers sintered at 1400°C for 6h are shown in Figure 4. The YAG fiber was obtained with smooth surface and fine grains. But the titania-YAG fiber was obtained with rough surface and some pores was observed on fiber surface. And coarse grains were observed with grain size about $0.5 \sim 1 \mu m$. So, titania promoted YAG grain growth and limited the YAG application in high temperature condition.



(a) YAG liber (b) Intania-YAG liber Figure 4 : SEM microstructures of the (a) YAG and (b) titania-YAG precursor gel fibers heated at 1400°C for 6h.

CONCLUSION

The titania-YAG fibers were prepared by sol-gel method using aluminum chloride, titanium chloride, aluminum powder, yttrium oxide and acetic acid as raw materials. The YAG fiber was obtained with smooth surface and fine grains were observed by sintering at 1400°C for 6h. But the titania-YAG fiber was observed with rough surface. And coarse grains were observed with grain size about $0.5~1\mu$ m.

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Full Paper 🛥

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