Characteristics of Porous YAG Powders Fabricated by PVA Polymer Solution Technique

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Abstract

Pure and stable YAG ($Y_3Al_5O_{12}$) powders were synthesized by a PVA (polyvinyl alcohol) polymer solution technique. PVA was used as an organic carrier for the precursor ceramic gel. The precursor gels were crystallized to YAG at relatively a low temperature of 900 ℃. The synthesized powders, which have nano-sized primary particles, were soft and porous, and the porous powders were ground to sub-micron size by a simple ball milling process. The ball-milled powders were densified to 94% relative density at 1500 ℃ for 1h. In this study, the characteristics of the synthesized YAG powders were examined.

Keywords : Solution Technique, Polyvinyl Alcohol, Precursor, YAG powder, Porous Powder

1. Introduction

Ceramic processing is evolving in the direction of chemical synthesis of powders. Methods based on soft-solution processing provide powders with desired properties such as high purity and homogeneity. In recent, as one of the soft-solution processing, organic-inorganic solution technique using PVA (polyvinyl alcohol), PEG (polyethylene glycol) or EG (ethylene glycol) as a polymer carrier has been developed to make easier and high productive soft-solution processing.

Yttrium aluminum garnet (YAG, $Y_3Al_5O_{12}$) has high strength and good optical properties, so it is used widely as a high temperature engineering material. However, traditional mixed oxide methods require high-temperature heat treatments to obtain phase-pure YAG powder.

In this paper, YAG ($Y_3Al_5O_{12}$) powders are prepared by the organic-inorganic solution method, and the effect of polymer on the process of powder synthesis is studied. As well, porous structure of the synthesized powders are introduced. Finally, sintering behavior of the ball-milled YAG powders are examined.

2. Experimental and Results

Aluminum nitrate and yttrium nitrate were dissolved in stoichiometric proportions in D.I. water. After dissolving completely, a polymeric carrier was added, and then the mixture was heated up to 100 °C with stirring. As a polymeric carrier, polyvinyl alcohol (PVA) solution was added to the solution. The water in the solution was evaporated by continuous stirring during heating on a hot plate. The resulting gel-type precursors were completely dried after several hours at 100 °C. The dried precursors were calcined or crystallized at various temperatures in an air atmosphere in a box furnace. The calcined powders were ball milled with ϕ 5mm zirconia media for 10 h. Isopropyl alcohol was used as a solvent for milling. The ball-milled powders were uniaxially pressed at 10 MPa, and then the pellet-shaped green compacts were sintered in an air atmosphere at 1500 °C and 1600 °C for 1 h.

The precursor involving organic binder resembled an aero-gel and turned to a porous powder after drying. The porous structure was due to the development of foams in the thick solution during the stirring and drying process. The entrapment of NO_x gases produced by the decomposition of nitrate salts resulted in the generation of foams. The dried precursor gel was calcined and crystallized at various temperatures. The crystallized powder also showed porous structure. The crystalline phases of the YAG precursor at each temperature are summarized in Fig. 1. The first crystalline form was observed at 900 °C, and fully crystallized YAG was detected at 1000 °C. The crystallization temperature of 1000 °C is almost 100 °C lower than the temperatures of phase-pure YAG powders synthesized by previous methods.

In the thermal analysis examination, exothermic reactions occurred at the range of 300~500 °C and about 900°C. The temperature zones of decomposition, which is accompanied with weight loss, were 100~400 °C and 400~900 °C, respectively. The initial weight loss was due to the pyrolysis of nitrates and polymer chains of PVA. At 400 °C, oxidation of the residual carbon from pyrolyzed organics was occurred and continued up to 900 °C. Furthermore, a small exothermic peak at about 900 °C was corresponded to the first crystallization temperature of YAG precursor as shown in Fig. 1.

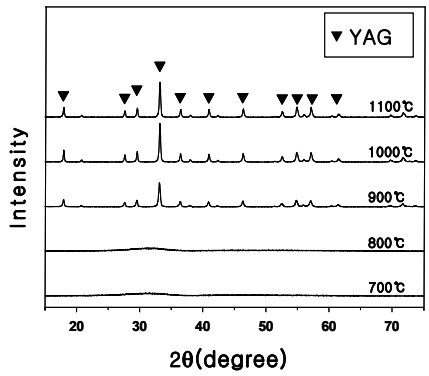


Fig. 1. XRD patterns of synthesized YAG powders at each temperature.

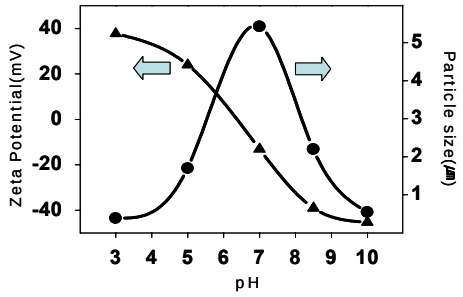
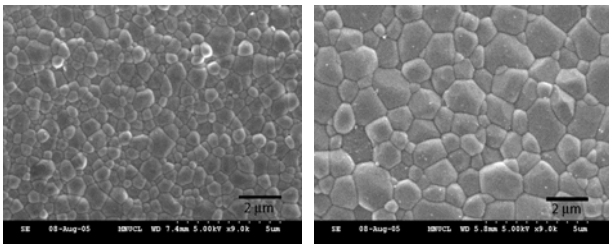


Fig. 2. Variation of particle sizes and zeta potentials of ball-milled powders at each pH condition.



(a) (b)

Fig. 3. SEM micrographs of sintered YAG at (a) 1500 °C and (b) 1600 °C for 1h.

The crystalline YAG revealed soft and porous powder structure. The powder milled for 10 h showed submicrometre particles with severe agglomeration. In the milling process, the efficiency was improved by the soft and porous powder structure in comparison with non-porous powders. The particle size of the ball-milled, fine powder was examined by electrophoretic light scattering spectrophotometer. The agglomerated, ball-milled powder was ultra-sonicated for 30 min in each pH solution, and then the particle size distribution and zeta potential at each suspension were measured. The results are presented in Fig. 2. According to the zeta potential result, it can be seen that the agglomerated YAG powder was dispersed well in the solution of pH 3. At this condition, the particle size was about 0.3 μm and the measured zeta potential was 40 mV.

Figs. 3 shows the SEM micrographs of the sintered YAG at 1500 °C and 1600 °C for 1 h. The densified YAG at 1500 °C had a 94% relative density with sub-micron grain size. At 1600 °C, the grain size was more increased and the densified YAG had a 98% relative density.

3. Summary

The organic-inorganic solution method was successfully used for the fabrication of porous YAG powders. The PVA polymer contributed to soft and porous powder microstructure, and simple ball milling with the porous powder was effective in making highly sinterable YAG powders.

The ball-milled, fine powders were densified at a relatively low temperature of 1500 °C for 1 h showing an average grain size of 0.7 μm. The organic-inorganic solution method may well be applied to the fabrication of many other highly sinterable oxide ceramic powders because of its merits, such as being a simple process, allowing easy control of powder size by simple ball milling.

4. References

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