

Effect of synthesis conditions on the particle size and morphology of YAG powder

Jie-Guang Song*, Fang Wang, Ming-Han Xu, Yin-Yan Ju, Yang-Liang Li, Shi-Bin Li and Gang-Chang Ji

Jiujiang Key Laboratory of Green Remanufacturing, School of Mechanical and Materials Engineering, Jiujiang University, Jiujiang 332005, China

Yttrium aluminum garnet (YAG) is currently a widely applied structural and functional material. To prepare high-performance YAG ceramics we prepared pure YAG powder, after calcining the precursor at 1100 °C, by co-precipitation with ammonia as the precipitant and aluminum nitrate as well as yttrium nitrate as raw materials. The preparation conditions for the YAG precursor were a pH of 9, a titration rate of 10 ml/minute and a reaction time of 60 minutes. The mean particle size of the YAG powder was 11 μm. The mean particle size of the YAG powder increased with an increase in the pH and at a higher pH the YAG particles were more irregular in shape. The mean particle size and particle size distribution range of the YAG powder increased with an increase in the titration rate. For the YAG powder with a higher mean particle size, the particle size distribution range was wider but the size of most YAG particles was still small and the YAG particles were nearly spherical.

Key words: YAG powder, Co-precipitation method, Synthesis condition, Particle size, Micrograph.

Introduction

Yttrium aluminum garnet (YAG) with a chemical composition of $Y_3Al_5O_{12}$ is an important advanced structural and functional material [1-3]. Because of its relatively stable lattice structure and large thermal conductivity, it is used as a host for solid-state laser materials in luminescence systems and in the window material for a variety of lamps. YAG also has great potential application as a high-temperature engineering material because of its good high-temperature strength as well as its superior creep resistance. In addition, YAG is a promising fiber material for the preparation of ceramic composites [4-6].

However, the wider application of YAG is limited because the manufacture of jumbo size YAG single crystal requires special equipment, complicated technology, long production periods, has a high cost and the product has a defective index. By comparison with single crystals, the mass manufacture of YAG polycrystal powder materials is easily realized [7-8]. The performance of polycrystal materials is better than that of single crystal materials under specific conditions so they can be used as a substitute for single crystal materials to produce structural and functional materials [9-10].

The manufacture of YAG powders can be carried out by sol-gel processing, metal-organic preceramic processing, hydrothermal synthesis and co-precipitation [11-13]. Co-precipitation is widely applied because of advantages in sampling methods and the ease of controlling the accuracy.

In this study we investigated the effect of synthesis conditions on the particle size and morphology of YAG powders produced by co-precipitation. This study lays a foundation for the preparation of high-performance superfine YAG powder materials.

Experimental Procedures

Preparation of YAG powder

We used analytical grade aluminum nitrate, yttrium nitrate and ammonia. A solution of aluminum nitrate (0.0126 mol/l) and yttrium nitrate (0.00756 mol/l) were mixed to produce an $Al(NO_3)_3$ and $Y(NO_3)_3$ composite solution. This solution was added slowly (3 ml/minute) to deionized water to form a suspension, which was then stirred. Ammonia was added to the suspension at different rates until a specific pH was reached. The suspension was then stirred for 1 hour, filtered and the precipitate was washed four times (three times with deionized water and once with absolute ethyl alcohol). The precipitate was dried at 100 °C and a YAG precursor was obtained. The YAG precursor was calcined at different temperatures for 1 hour and superfine and pure YAG powder was obtained. Fig. 1 shows a flow diagram of the preparation process.

Characterization of YAG powder

The phase of the YAG precursor and the superfine YAG powder was identified by X-ray powder diffraction (XRD) (Model: D/Max-RB, Japan). The particle size analysis of the superfine YAG powders was performed with a laser grain size analyzer (Model: Zetasizer Nano ZS90, English). A microstructural analysis of the superfine YAG powders was performed with a scanning electron microscope (SEM) (Model: TESCAN VEGA II, Czech Republic).

*Corresponding author:
Tel : +86-792-8313632
Fax: +86-792-8311239
E-mail: songjieguang@163.com.

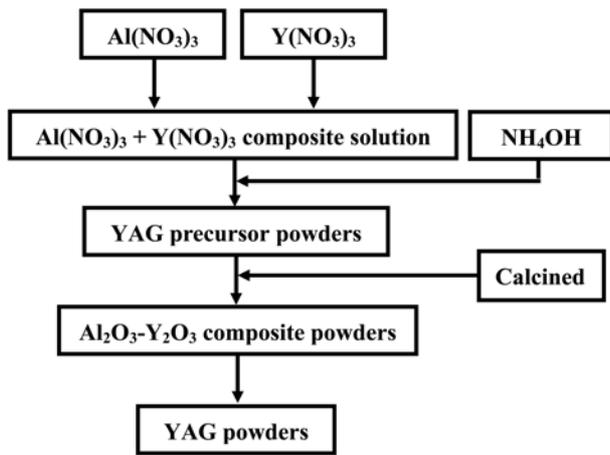


Fig. 1. Process flow diagram for the preparation of YAG powder.

Results and Discussion

Effect of the calcination temperature on phase of YAG powder

A phase analysis of the calcined YAG precursor prepared using different temperatures is shown in Fig. 2. The other preparation conditions were a pH of 9, a titration rate of 10 ml/minute and a reaction time of 60 minutes. The analysis shows that the YAG precursor does not have an obvious characteristic crystalline diffraction peak (Fig. 2(a)) because the YAG precursor is amorphous. An obvious diffraction peak was present at a 2θ of $25\text{--}35^\circ$ after the calcination of the YAG precursor at 600°C for 1 hour (Fig. 2(b)), which indicates that the powder remains in an amorphous state but small quantities of a crystalline

phase are produced. At higher calcination temperatures the characteristic diffraction peaks of Y_2O_3 and Al_2O_3 are present. After calcining the YAG precursor powder at 800°C for 1 hour (Fig. 2(c)), we found that $\text{Al}(\text{OH})_3$ and $\text{Y}(\text{OH})_3$ both decomposed. The characteristic diffraction peak of YAG was found after the calcination of the YAG precursor at 1100°C for 1 hour (Fig. 2(d)), which indicates that both Y_2O_3 and Al_2O_3 reacted to produce the YAG crystalline phase [14]. The characteristic diffraction peak of YAG is strong, which indicates that the YAG crystalline phase is integrated and pure.

Effect of the pH value on the particle size and morphology of YAG powder

SEM micrographs of the YAG powder prepared at different pH values are shown in Fig. 3 and the other preparation conditions were a titration rate of 10 ml/minute, a reaction time of 60 minutes and a calcination temperature of 1100°C . The mean particle sizes of the YAG powders are $6\ \mu\text{m}$, $11\ \mu\text{m}$ and $18\ \mu\text{m}$ respectively under pH = 7, 9 and 11. The mean particle size of the YAG powder increased with an increase in the pH and at a higher pH the YAG particles were more irregular in shape. The particle size distribution is shown in Fig. 3 and the particle size distribution is wider at a pH of 11. The higher pH means that more $[\text{OH}]^-$ is present during the process [15-16] and the concentration range of $[\text{OH}]^-$ is wider during the entire reaction process. This concentration is a very important factor for the nucleation and growth of the YAG precursor particles. $\text{Al}(\text{OH})_3$ and $\text{Y}(\text{OH})_3$ powders with different particle sizes were produced using different concentrations and this leads to the wider particle size distribution of the

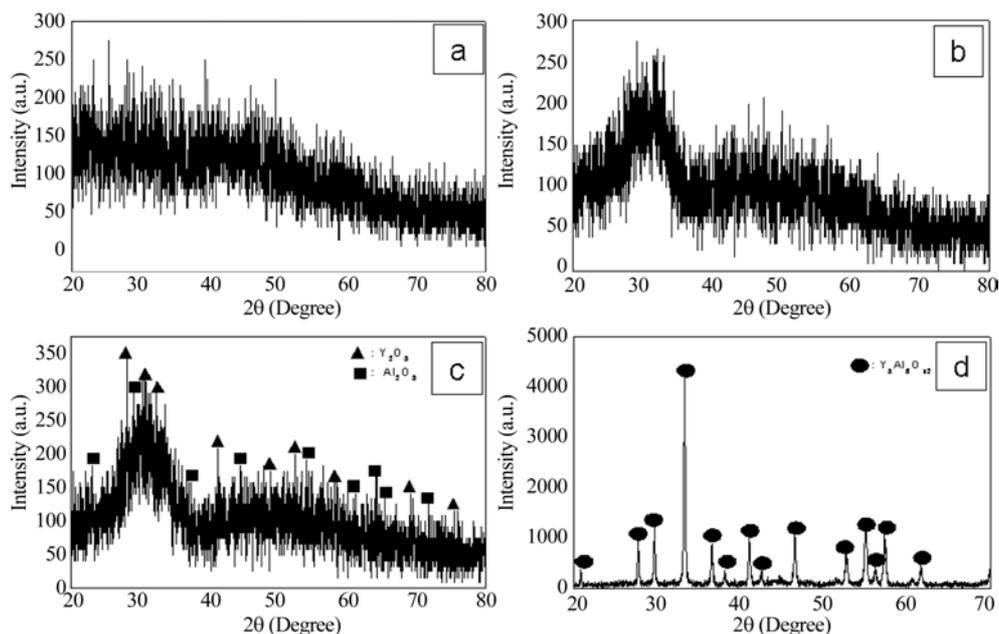


Fig. 2. Phase analysis of (a) the YAG precursor powder calcined at (b) 600°C , (c) 800°C and (d) 1100°C .

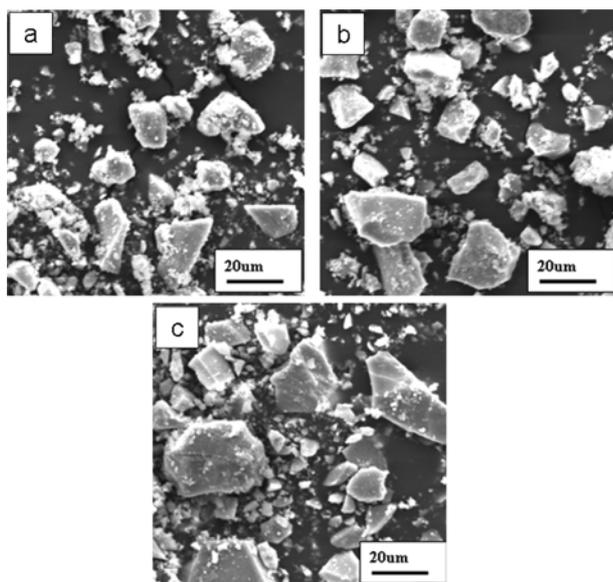


Fig. 3. SEM of the YAG powder synthesized at (a) pH = 7, (b) pH = 9 and (c) pH = 11.

YAG powders. The better dispersion characteristics of the YAG powders are shown in Fig. 3.

Effect of the titration rate on the particle size and morphology of YAG powder

SEM micrographs of the YAG powders prepared using different titration rates are shown in Fig. 4 and the other preparation conditions were a pH of 9, a reaction time of 60 minutes and a calcination temperature of 1100 °C. The mean particle sizes of the YAG powders are 5 μm, 11 μm and 21 μm respectively under 5 ml/minute, 10 ml/minute and 30 ml/minute. The mean particle size and particle size distribution range of the YAG powders increased with higher titration rates. At a titration rate of 5 ml/minute, the particle size was smaller, the particle size of most of the YAG particles was about 2 μm and they were better dispersed. The dispersion of the fine and uniform particles is better under lower titration rate conditions. A lower titration rate results in less available Al^{3+} and Y^{3+} and the probability of crystal nucleus production is higher but the probability of crystal nucleus growth is lower. Therefore, uniform and smaller YAG particles were prepared under the lower titration rate conditions. At a titration rate of 10 ml/minute, smaller particles are mainly present. At a titration rate of 30 ml/min, the YAG particles are inhomogeneous and irregular. Because the Al^{3+} and Y^{3+} concentrations are higher at a higher titration rate, enough Al^{3+} and Y^{3+} is available for nucleus growth [17-18]. A larger particle precursor powder is produced and this leads to the formation of larger particle size YAG powders. When the concentration of Al^{3+} and Y^{3+} is varied the concentration distribution of Al^{3+} and Y^{3+} is wider and this causes the particle size distribution of the YAG precursor powder to be wider. Therefore, the particle size distribution of the prepared YAG powder is wider.

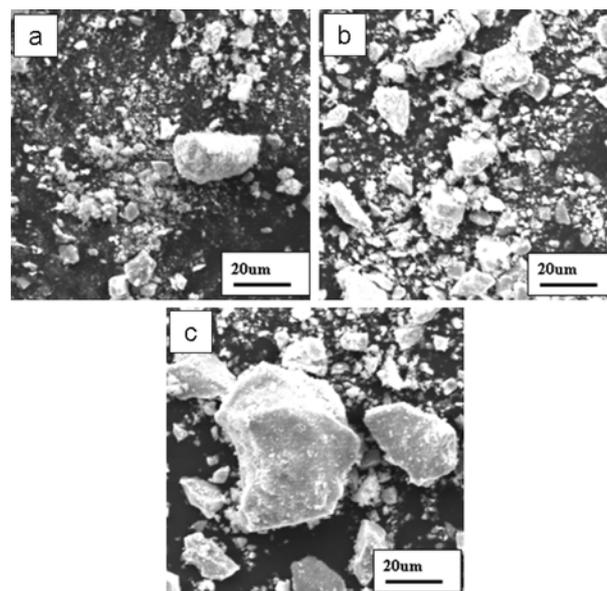


Fig. 4. SEM of the YAG powder synthesized at titration rates of (a) 5 ml/minute, (b) 10 ml/minute and (c) 30 ml/minute.

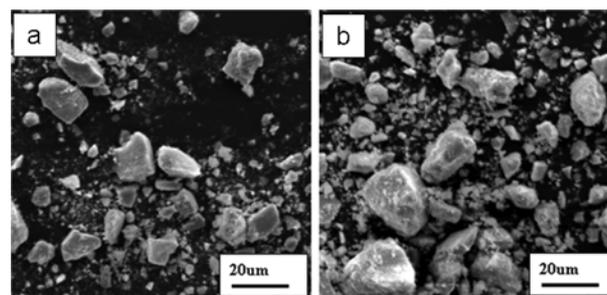


Fig. 5. SEM of the YAG powder synthesized at reaction times of (a) 30 minutes and (b) 60 minutes.

Effect of the reaction time on the particle size and morphology of YAG powder

SEM micrographs of the YAG powders prepared using different pH values are shown in Fig. 5. The other preparation conditions were a pH of 9, a titration rate of 10 ml/minute and a calcination temperature of 1100 °C. The mean particle sizes of the YAG powders are 7 μm and 11 μm respectively for 30 minutes and 60 minutes. With a shorter reaction time, the YAG particles have a better dispersion, are finer, more inhomogeneous and irregular. With a reaction time of 30 minutes, the particle size of most of the YAG particles are less than 10 μm and the smallest particle is about 2 μm. The mean particle size of the YAG powder was higher at a reaction time of 60 minutes and the particle size distribution range was wider, but the particle size of most of the YAG particles is less than 15 μm and they are nearly spherical. Because of the longer reaction time, the crystal growth time is adequate and the integrity of the crystals are better. In addition, YAG is known to have a cubic structure [19-20] and the YAG particles are nearly spherical as well.

Conclusions

Pure YAG powder was synthesized, after calcining the precursor at 1100 °C, by co-precipitation with ammonia as a precipitant and aluminum nitrate as well as yttrium nitrate were used as raw materials. The preparation conditions for the YAG precursor were a pH of 9, a titration rate of 10 ml/minute and a reaction time of 60 minutes. The mean particle size of the YAG powder was 11 µm. The mean particle size of the YAG powder increased as the pH increased, and at a higher pH, the YAG particles were found to be more irregular. The mean particle size and particle size distribution range of the YAG powder increased with an increase in the titration rate. As the mean particle size of the YAG powder increased, the particle size distribution range became wider but the most of the YAG particles were still small. The YAG particles were nearly spherical.

Acknowledgments

The authors are thankful for the financial support provided by The Natural Science Foundation for Young Scholar of Jiangxi Province, China.

References

1. J.G. Song, G.C. Ji and S.B. Li, *J. Disper. Sci. Technol.* 30 (2009) 529-533.
2. S.J. Wang, *Mater. Sci. Eng. B.* 127 (2006) 203-206.
3. J.G. Song, G.C. Ji and S.B. Li, *J. Ceram. Process. Res.* 10 (2009) 428-432.
4. Zarzecka, M., *J. Eur. Ceram. Soc.* 27 (2007) 593-597.
5. A. Tarafder, A.R. Molla and B. Karmakarw, *J. Am. Ceram. Soc.* 93 (2010) 1-8.
6. J.G. Song, D.M. Du, and Y.Y. Ju, *J. Reinf. Plast. Comp.* 29 (2010) 710-717.
7. A. Fernandez, *J. Mater. Sci.* 38 (2003) 2331-2335.
8. J.G. Song, *Synth. React. Inorg. M.* 39 (2009) 83-86.
9. S.J. Wang, B. Xu and P. X. Lu, *Mater. Sci. Eng. B* 127 (2006) 203-206.
10. A. Lukowiak, R.J. Wiglusz and M. Maczka, *Chemical. Physics. Letters.* 494 (2010) 279-283.
11. Y.H. Sang, H. Liu and Y.H. Lv, *J. Alloy. Compnd.* 490 (2010) 459-462.
12. J.G. Song, G.C. Ji and S.B. Li, *Surf. Rev. Lett.* 15 (2008) 581-585.
13. Y.M. Zhang and H.M. Yu, *Ceram. Int.* 35 (2009) 2077-2081.
14. L. Yang, T.C. Lu, H. Xu and N. Wei, *J. Alloy. Compnd.* 484 (2009) 449-451.
15. K. Guo, H.H. Chen and X.X. Guo, *J. Alloy. Compnd.* 500 (2010) 34-38.
16. J. Su, Q.L. Zhang and C.J. Gu, *Mater. Res. Bull.* 40 (2005) 1279-1285.
17. Z.H. Chen, Y. Yang and Z.G. Hu, *J. Alloy. Compnd.* 433 (2007) 328-331.
18. N. Kaithwas, M. Deshmukh and S. Kar, *Cryst. Res. Technol.* 42 (2007) 991-994.
19. X. X., Li and W.J. Wang, *Powder. Technol.* 196 (2009) 26-29.
20. Q.X. Zheng, B., Li and H.D. Zhang, *J. Supercrit. Fluid.* 50 (2009) 77-81.