ELSEVIER

Ceramics International 27 (2001) 721–723

CERAMICS INTERNATIONAL

www.elsevier.com/locate/ceramint

Short communication

Preparation and microstructure of polycrystalline Al_2O_3–YAG composites

Hongzhi Wang, Lian Gao *

State Key Laboratory on High Performance Ceramics and Superfine Microstructure, Shanghai Institute of Ceramics, Chinese Academy of Sciences, Shanghai 200050, People's Republic of China

Received 28 August 2000; received in revised form 21 September 2000; accepted 8 November 2000

Abstract

Polycrystalline Al_2O_3–YAG composites were obtained from the powder prepared by a co-precipitation method. After the calcination at different temperatures, the crystallization of YAG was found at about 1000°C. Dense sintering bodies could be obtained by hot-press sintering at 1550°C. Bending strength and fracture toughness of Al_2O_3–5 vol.% YAG and Al_2O_3–25 vol.% YAG composites were 604 MPa and 5.0 MPa $m^{1/2}$, 611 MPa and 4.5 MPa $m^{1/2}$ respectively. It indicated that the incorporation of YAG benefited to the mechanical property improvement of Al_2O_3 ceramics. TEM observation revealed that large YAG particles (about 1 μm) located on the grain boundaries of Al_2O_3, and some small YAG particles (100–200 nm) were inside Al_2O_3 grains in both composites. Many white areas were found in Al_2O_3–5 vol.%YAG composite, which probably results from low concentration of yttrium.

Keywords: B. Composites; YAG; Co-precipitation

1. Introduction

Al_2O_3 is one of the most frequently utilized high-temperature structural ceramics, but relative low mechanical properties limit its application. A second phase is often considered to reinforce Al_2O_3, and yttrium aluminum garnet (YAG) is believed to be a very suitable candidate for its excellent creep resistance, similar coefficient of thermal expansion and no reaction with Al_2O_3 [1].

Parthasarathy and Mah [2,3] studied the processing and mechanical properties of Al_2O_3/YAG eutectic composites, and found that the composites have a flexural strength of 373 MPa and a fracture toughness of 4.3 MPa $m^{1/2}$ at room temperature. In comparison with sapphire and single-crystal YAG, the composite has a significantly higher fracture toughness at elevated temperatures. However, the reports about polycrystalline Al_2O_3/YAG composites were still few. Duong et al. [4] have studied the creep behavior of fine-grained Al_2O_3–$Y_3Al_5O_{12}$ composites, but no mechanical properties were given.

In this work, we would fabricate polycrystalline Al_2O_3–YAG composites with higher mechanical properties than monolithic Al_2O_3 ceramics. So a homogenous microstructure, especially uniform distribution of YAG particle, was an importance problem to be considered. If the Al_2O_3 and YAG mixed powder was used as starting materials to fabricate Al_2O_3–YAG composites, it would be very difficult to get well dispersion of the particles. Comparing with the particle mixing, a solution mixing would benefit to the uniform distribution of each element. In the phase diagram of Y_2O_3–Al_2O_3 system [5], it can be found that Al_2O_3 and YAG can coexist until 1800°C in the Al_2O_3-rich field. If the solution including Y and Al elements was used to produce hydroxides, and then oxides after heat-treatment, a homogenous microstructure would be hopeful to obtain. So the co-precipitation method was utilized in the experiments to get starting powder.

2. Experimental

Two composition powders, Al_2O_3–5 vol.% YAG (Al_2O_3–3.2 wt.% Y_2O_3) and Al_2O_3–25 vol.% YAG (Al_2O_3–15.8 wt.% Y_2O_3), were prepared by a co-pre-

* Corresponding author. Tel.: +86-021-62512990, ext. 6321; fax: +86-021-62513903.

E-mail address: liangaoc@online.sh.cn (L. Gao).

0272-8842/01/$20.00
PII: S0272-8842(01)00009-8

cipitation method. $Al(NO_3)_3$ and $Y(NO_3)_3$ were mixed in aqueous solution with the proper ratios. This salt solution and ammonia were added into distilled water simultaneously while keeping pH values between 8 and 9. After washing with distilled water and ethanol, and drying at 100°C, the precipitates were calcined at 700°C for 2 h to get oxides. The oxide powders were hot-pressed at between 1450 and 1650°C for 1 h under 30 MPa of pressure in N_2 atmosphere to obtain sintered bodies.

To learn the forming process of YAG phase, a part of the precipitate was heat-treated at different temperatures, and the phases in powders and sintered bodies were determined by XRD, respectively. The densities of sintered samples were measured using the Archimedes method. For mechanical properties testing, the hot pressed samples were cut and ground into rectangular bar specimens (4×3×30). The fracture toughness was evaluated from the indentation fracture method, and fracture strength was measured by the three point bending test. Microstructures of sintered bodies were observed under TEM. EDS was used to detect elements in composites.

3. Results and discussion

X-ray diffraction patterns of the co-precipitated powder indicated that the precipitate consists of bayrite ($Al(OH)_3$) and yttrium hydroxide(amorphous). When calcination temperature was elevated above 400°C, bayrite disappeared, while the weak peaks of γ-Al_2O_3 occurred and persisted until 1000°C. After the calcination at 1200°C, the powder shows a high degree of crystallinity, the peaks of YAG, α-Al_2O_3 and $YAlO_3$ were observed. The XRD pattern of the powder calcined at 1300°C shows well-defined peaks whose positions are almost identical to those of stoichometric YAG and α-Al_2O_3. The results indicate that crystallization of YAG occurs at ≈1000°C. $YAlO_3$ is metastable and transforms to YAG by reacting with Al_2O_3 when the temperature is increased up to 1300°C.

The powders calcined at 700°C were hot-pressed at the temperatures between 1450 and 1650°C. Although

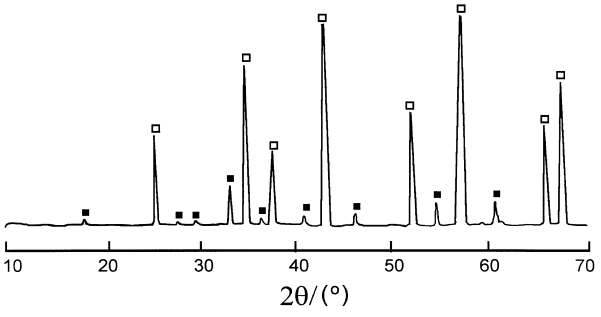

Fig. 1. XRD spectrum of Al_2O_3–5 vol.% YAG composite hot-pressed at 1550°C (■ represents YAG, □ represents α-Al_2O_3).

the crystallization and transformation of YAG phase happened during the sintering, no cracked bodies have been obtained in our experiments. Both Al_2O_3–5 vol.% YAG and Al_2O_3–25 vol.% YAG composites reached to their theoretical densities after the hot-press sintering at 1550°C for 1 h. XRD revealed that only two phases, α-Al_2O_3 and YAG, existed in all sintered bodies. Fig. 1 showed the XRD spectrum of Al_2O_3–5 vol.% YAG composite hot-pressed at 1550°C.

Mechanical property testing at room temperature indicated that: bending strength of Al_2O_3–5 vol.% YAG composite was 604 MPa, and fracture toughness was 5.0 MPa $m^{1/2}$; those of Al_2O_3–25 vol.% YAG composite were 611 MPa and 4.5 MPa $m^{1/2}$ respectively. All these data were higher than those of monolithic Al_2O_3 ceramics reported by other researchers (300–400 MPa for strength, 3–4 MPa $m^{1/2}$ for toughness) [6,7]. It indicates that the incorporation of YAG benefited to the mechanical property improvement of Al_2O_3 ceramics.

Fig. 2 shows TEM pictures of Al_2O_3–5 vol.% YAG composite. It can be noted that the grain size of Al_2O_3 matrix was 2–4 μm, some large YAG particles (about 1 μm) located on the grain boundaries of Al_2O_3, while some small YAG particles (100–200 nm) were inside Al_2O_3 grains [Fig. 2(a)]. To our surprise, a lot of small white areas were found in Al_2O_3 grains. These white areas were usually close to intragranular YAG particles, even a part of them included half of YAG particle [Fig. 2(b)]. EDS revealed that no other element but Al and O could be detected in these white areas. The solubility limit of yttrium in Al_2O_3 has been evaluated to 10 ppm weight yttrium in other literatures [8,9], which could not be detected by EDS. So we presumed that these white areas lacked yttrium, which would lead to light contrast under TEM, because yttrium segregated from these areas and congregated to form YAG. In the TEM picture of Al_2O_3–25 vol.% YAG composite as shown in Fig. 3, white areas were seldom observed. Comparing to Al_2O_3–5 vol.% YAG composite, a high concentration of yttrium satisfied the requirement of YAG formation in Al_2O_3–25 vol.% YAG. However, the existence of white areas seems to have little influence on mechanical properties of the composites. This is corroborated by the similar properties of the materials differing in Y_2O_3 concentration. From Fig. 3, it could be also observed that Al_2O_3 grain size was about 1–2 μm, which was smaller than that of Al_2O_3–5 vol.% YAG composite. This difference is plausibly caused by higher concentration of YAG particles in Al_2O_3–25 vol.% YAG composites, which inhibited the grain growth of Al_2O_3 during sintering.

Microcracks were frequently observed under TEM as shown in Fig. 4. It may be attributed to anisotropy of Al_2O_3 grains and the different thermal expansion coefficients between Al_2O_3 and YAG. The creation of the microcracks was advantageous to the mechanical property improvement of composites.

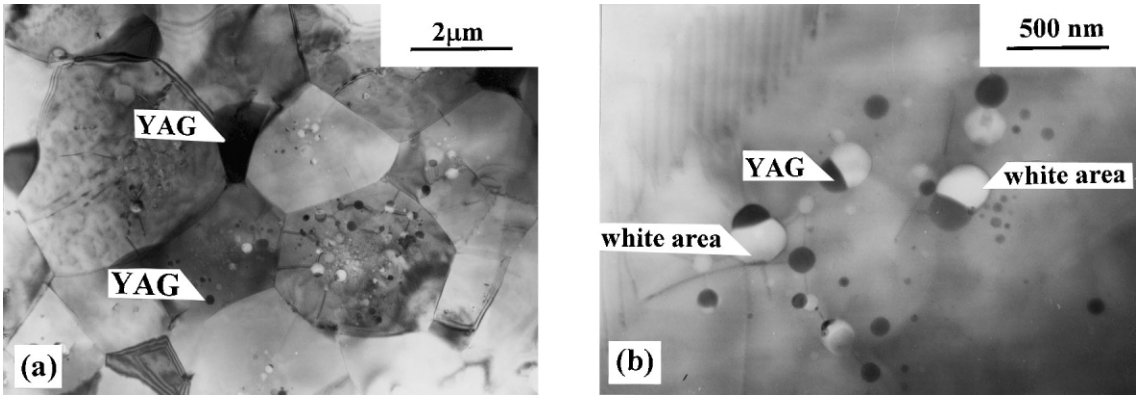

Fig. 2. TEM pictures of Al_2O_3–5 vol.% YAG composite.

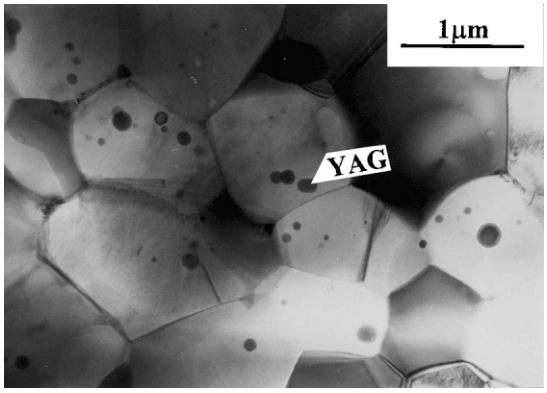

Fig. 3. TEM pictures of Al_2O_3–25 vol.% YAG composite.

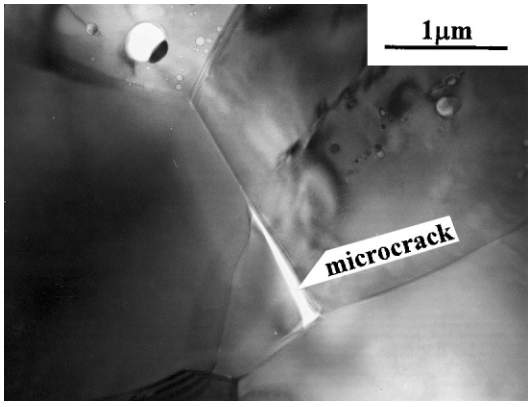

Fig. 4. Microcrack in Al_2O_3–5 vol.% YAG composite.

4. Conclusions

Composite powders Al_2O_3–5 vol.% YAG and Al_2O_3–25 vol.%YAG were prepared by a co-precipitation method. The crystallization of YAG occurred at about 1000°C. After hot-press sintering at 1550°C for 1 h, nearly dense bodies could be obtained. Bending strength and fracture toughness of Al_2O_3–5 vol.% YAG and Al_2O_3–25 vol.% YAG composites were 604 MPa and 5.0 MPa $m^{1/2}$, 611 MPa and 4.5 MPa $m^{1/2}$ respectively. Large YAG particles located on the grain boundaries of Al_2O_3, while some small YAG particles were inside Al_2O_3 grains. A lot of white areas were observed in Al_2O_3–5 vol.% YAG under TEM, which may be relative to insufficiency of yttrium to form YAG.

References

[1] K. Keller, T. Mah, T. Parthasarathy, Processing and mechanical properties of polycrystalline $Y_3Al_5O_{12}$, Ceram. Eng. Sci. Proc. 11 (1990) 1122–1133.
[2] T. Mah, T. Parthasarathy, L. Matson, Processing and mechanical properties of $Al_2O_3/Y_3Al_5O_{12}$(YAG) eutectic composite, Ceram. Eng. Sci. Proc. 11 (1990) 1617–1633.
[3] L. Matson, R. Hay, T. Mah, Characterization of alumina/yttrium–aluminum garnet and alumina/yttrium–aluminum perovskite eutectics, Ceram. Eng. Sci. Proc. 11 (1990) 995–1003.
[4] H. Duong, J. Wolfenstine, Creep-behavior of fine-grained two-phase Al_2O_3–$Y_3Al_5O_{12}$ materials, Mater. Sci. Eng. A172 (1993) 173–179.
[5] E. Levin, C. Robbins, H. McMurdie (Eds.), Phase Diagrams for Ceramists 1969 Supplement, The Am. Ceram. Soc. Pub., Columbus, OH, 1969, p. 96, Figs.2344–2347.
[6] W. Gitzen (Ed.), Alumina as a Ceramic Materials, The Am. Ceram. Soc. Pub. Columbus, OH, 1970, p. 43.
[7] K. Niihara, New design concept of structural ceramics-ceramic nanocomposites, J. Ceram. Soc. Jpn 99 (1991) 974–982.
[8] M. Gulgun, V. Putlayev, M. Ruhle, Effect of yttrium doping alpha-alumina: I. microstructure and microchemistry, J. Am. Ceram. Soc. 82 (7) (1999) 1849–1859.
[9] E. Sato, C. Carry, Yttria doping and sintering of submicrometer-grained alpha-alumina, J. Am. Ceram. Soc. 79 (8) (1996) 2156–2160.