FABRICATION OF POLYCRYSTALLINE YAG/ALUMINA COMPOSITE FIBERS AND YAG FIBER

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SUMMARY: Polycrystalline YAG fiber and α-alumina and YAG matrix composite fiber have been prepared by sol-gel method. α-alumina and YAG matrix composite fiber with fine and homogeneous microstructure could be successfully fabricated by interpenetrating YAG in alumina matrix and adding α-alumina of seed particles to fibers. Effect of α-alumina seed particles and YAG on crystallization and microstructure of composite fiber were discussed. And the size of Alumina matrix of the composite fibers heated at 1600^{o}C for 4hours was below 2 μm.

KEYWORDS: Sol-gel, Al_2O_3, YAG, Composite fiber, Seed particle, Tensile strength, Microstructure, Creep

INTRODUCTION

Continuous fiber-reinforced ceramic composites (CFCCs) show improved toughness and great potential for high temperature applications, for example, gas turbine engine. Fibers used in these CFCCs are required to exhibit good thermal and mechanical properties in high temperature oxiding environments. Oxide fibers have the potential to maintain durability and mechanical properties in oxidizing environments. However, the applications of commercially available oxide fibers are limited by the degradation in fracture strength and creep properties above 1200°C. For example, Al_2O_3 fibers derived by sol-gel process, which have been commercially used for a number of years, contained a mixture of alumina spinel and amorphous silica, resulting in poor creep resistance [1]. Thus, recent researches have been focused on developing fiber materials with inherently better high-temperature properties.

The creep resistance of polycrystalline ceramics can be improved by the addition of hard second-phase particles [2-3]. The second-phase particles can affect the deformation rate of a polycrystal in two ways [4]. One is macroscopic modification of the continuum deformation mechanics, and the other is microscopic change of the interface -related deformation characteristics such as suppressing interface reactions (vacancy or interstitial creation

/annihilation) or hindering grain boundary sliding. In addition, the second-phase particles may affect fiber grain growth, microstructure, and therefore creep rate by grain boundary pinning.

YAG (yttrium-aluminum garnet, $Y_3Al_5O_{12}$) is a prime candidate for hardening Al_2O_3 due to its excellent creep resistance [5,6]. Parthasarathy et al. [7] investigated the creep behavior of hot-pressed polycrystalline YAG with the grain size of 3 m and found that it indicated a lower creep rate than that calculated for polycrystalline Al_2O_3 with a similar grain size. Directionally solidified Al_2O_3-$Y_3Al_5O_{12}$ eutectic composites have been also shown to exhibit better high-temperature properties.

This paper described new fabrication process of Al_2O_3 and YAG composite fiber and YAG fiber. Transformation behavior and microstructure development of Al_2O_3 based composite fiber were studied. The effect of α-Al_2O_3 and YAG seed particles and mechanical properties of these fibers were also examined.

EXPERIMENTAL PROCEDURE

YAG and alumina composite fibers and YAG fiber were prepared. The composite fibers were 0.5 vol% and 40vol% yttrium content.

In order to synthesis precursor fiber, aluminum isopropoxide (Nakarai Chemical Co., Japan) and yttrium isopropoxide (High Purity Chemical Co., Japan) were prepared. Aluminum isopropoxide and yttrium isopropoxide stabilized with 2-isobutoxyethanol were mixed in the isopropyl alcohol and ethyl 3-oxobutanate, according to the procedure described by Yogo et al [8]. YAG fiber was synthesized the mole ratio of aluminum isopropoxide : yttrium isopropoxide = 5 : 3. In the case of fiber with seed particles, a-alumina (Average particle size : 0.2 mm, TMDA, Taimei Chemical Co., Japan) were added to the solution. The solution was hydrolyzed by adding the hydrochloric acid, and was condensed, and then was spun to the precursor fiber. The precursor fiber was dried atmosphere, and then heat-treated at various temperatures in air.

The crystalline phase of the composite fiber was identified by X-ray powder diffraction (RU-200B, Rigaku Co. Ltd., Japan).The crystallization temperature of the fiber was investigated by DSC (Shikuu-rikou,Japan). The fiber morphology and microstructure were observed by a scanning electron microscope (SEM, JSM-6320FK, JEOL, Japan). The ratio of Y/Al of the fiber was measured by inductively coupled plasma-emission spectrometry (ICP-ES, ICAP-1000S, NIPPON Jarrell-Ash, Japan).

RESULTS AND DISCUSSION

Figure 1 shows the SEM photographs of the seeded composite fibers heated at 1500°C for 4 h with 3.7 vol% YAG. Figure 2 shows DSC curves of the composite fibers. The transformation temperature from θ to α phase alumina was 1130°C in the case of the seed particles of 0.2 wt %. The size of the alumina matrix was 0.7 -0.8 :m and was much smaller than that of the unseeded fiber. It was obvious that intra- and inter-granular pore were frequently observed for unseeded composite fibers. They showed many large pores and in homogeneous microstructure compared with that of the unseeded Al_2O_3 fiber. This could be explained by the obstruction of crystallization by yttrium and abrupt grain growth during the transformation to α-phase. Since the crystallization temperature largely increased by 100 to 200°C during to yttrium addition, sudden grain growth at high heat-treating temperatures resulted in homogeneous grain structure and large inter-granular pores for the unseeded composite fiber. In the case of 3 wt % α-alumina seed particles, the transformation temperature was 1110°C and the size of the alumina matrix was 0.5 :m. The grain size of the alumina matrix decreased with increasing the content of the α-alumina to 3 wt %.

However the size of 10 wt % seed particles suddenly grew up to 5 m. This is due to the sintering of the α-alumina seed particles. In the case of alumina / YAG composite fibers, similarly to alumina fibers, the addition of a-alumina seed particles enhanced transformation of alumina matrix from theta to alpha phase. The temperature of the peak decreased from 1210 to 1090°C when the amount of seeds reached 10 wt %. The critical quantity of seeds needed to shift the transformation amounts approximately to 1-2 wt %. The results were consistent with other reports [9,10]

Figure 3 shows the X-ray diffraction patterns of YAG fiber heat-treated at 700^{o}C , 800^{o}C and 900^{o}C. An amorphous YAG was observed at 700^{o}C, the crystallization of YAG started at 800^{o}C.

Figure 4 shows DSC curves of the YAG fibers included with YAG seed particles and YAG fiber not included with YAG seed particles. The seeded fiber had two peak from 700^{o}C to 900^{o}C, but unseeded fiber had only one. The first peak of seeded fiber at 740^{o}C seemed to result from the crystallization of hexagonal YAP and second peak at 850^{o}C seemed to the transformation hexagonal YAP phase into YAG phase [11]. The peak of seeded fiber appeared at lower temperature than unseeded YAG fiber.

Figure 5 shows the X-ray diffraction patterns of YAG and alumina composite fiber (YAG was included of 40 vol%) heat-treated at 1000^{o}C, 1100^{o}C, 1200^{o}C, 1300^{o}C, 1400^{o}C and 1500 ^{o}C. It seemed that yttrium reacted with alumina as the heat-treatment temperature increased, to produce an hexagonal YAP above 1000^{o}C. At same time, YAG appeared at 1100 ^{o}C. While increasing the heat-treatment temperature, the relative intensity of YAG increased, and YAP completely disappeared at 1300^{o}C and the composite fiber consisted of α-alumina and YAG at 1300^{o}C.

Figure 6 shows the SEM photograph for thermally-etched surface of the alumina/YAG composite fiber included with 40 vol% YAG heat-treated at 1600^{o}C for 4 h. The matrix size of YAG fiber grew up to 5 μm. This is smaller than alumina fiber. In the case of alumina / YAG composite fibers, similarly to YAG fibers, the addition of yttrium control grain growth of matrix. The composite fiber was consisted with the 1μm alumina and YAG grains intertwined.

CONCLUSIONS

Polycrystalline alumina and YAG composite fibers and YAG fiber was successfully fabricated by sol-gel process containing α-alumina or YAG seed particles. This α-alumina seed particle accelerated the phase transformation from θ to α-phase. The YAG seed particle effected multi-step of the yttrium and aluminum oxide. The composite fibers with YAG (YAG= 40 vol%) consisted of alumina and YAG grains with size of 1- 2 μm at 1600^{o}C.

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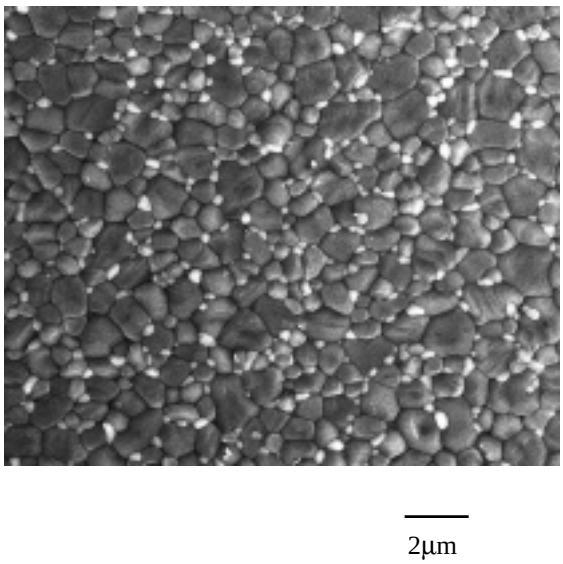

Fig. 1. SEM photograph of alumina/YAG composite fiber (YAG : 3.7 vol%).

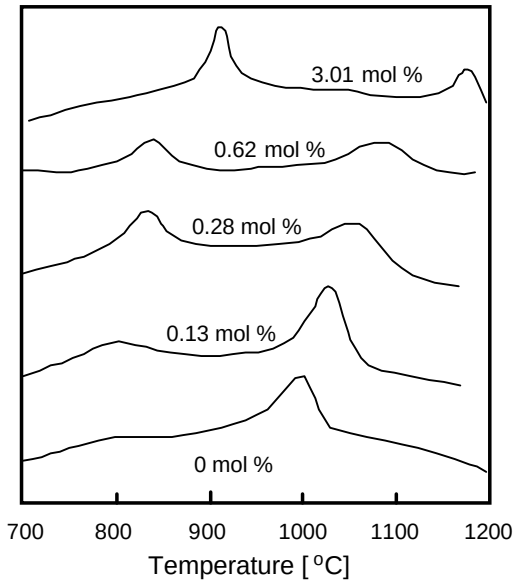

Fig. 2. DSC curve of composite fibers . Samples with various seed contents were heated at 10℃ min^{-1} in air.

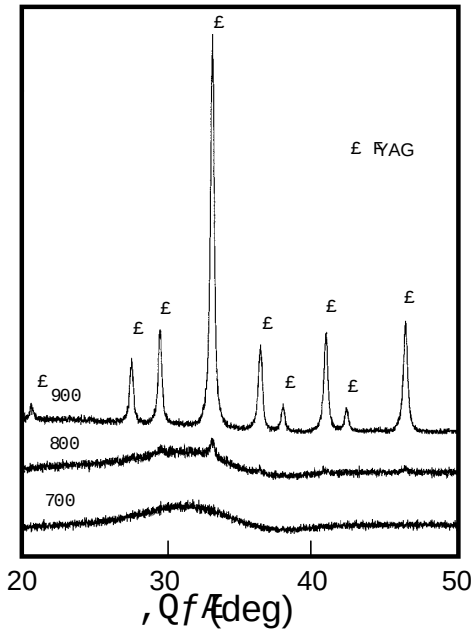

Fig. 3 X-ray diffraction pattern of YAG fibers heat-treated at various temperature for 4 hours.

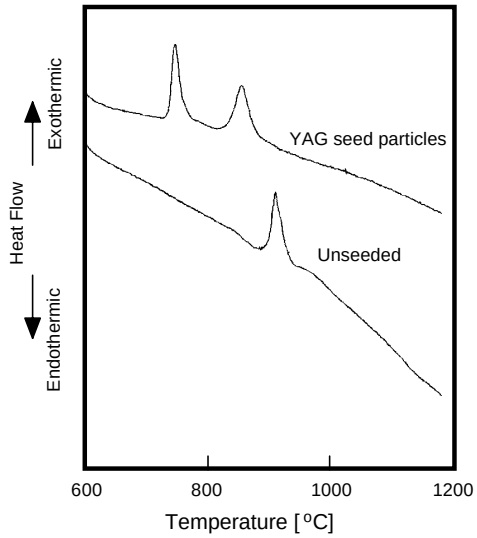

Fig. 4. DSC curve of YAG fibers heated at 10°C min^{-1} in air.

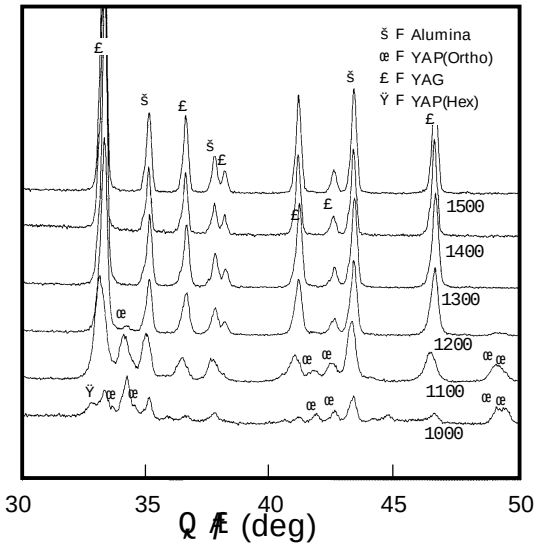

Fig. 5 X-ray diffraction pattern of Al_2O_3/YAG composite fibers (YAG : 40 vol%) heat-treated at various temperature for 4 hours.

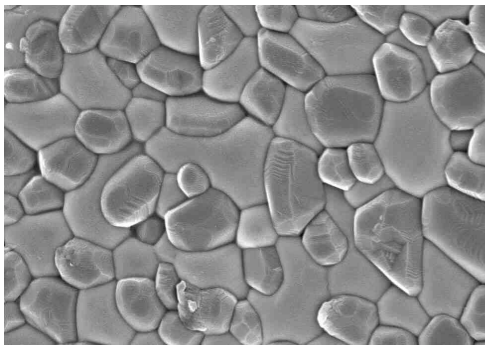

Fig. 6. SEM photograph of alumina/YAG composite fiber (YAG : 40 vol%).