Fabrication and Microstructure of Al_2O_3 Matrix Composites by In-situ Reaction in the Al_2O_3 – La_2O_3 System

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Al_2O_3 – La_2O_3 系からのその場反応によるアルミナ基複合材料の製造と微細構造

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Al_2O_3 /aluminate composites were fabricated by a solid reaction in the La_2O_3 – Al_2O_3 system. The formed phase showed elongated morphology that could be controlled by varying the hot-pressing temperature and time. Dense Al_2O_3 /LaAl₁₁O₁₈ composites were fabricated by hot-pressing above 1500°C. When the hot-pressing temperature was 1400°C, Al_2O_3 /LaAlO₃ composites were formed from starting composition. The grain size of monolithic alumina ceramics increased with increasing hot-pressing temperature, and marked grain growth occurred above 1600°C. However, the elongated LaAl₁₁O₁₈ phase suppressed the growth of alumina grains in the Al_2O_3 /LaAl₁₁O₁₈ composites. Small LaAl₁₁O₁₈ grains existed in the alumina grains as an intragranular phase, while needlelike grains with a large aspect ratio existed at Al_2O_3 grain boundaries. The amount of in-situ formed LaAl₁₁O₁₈ phase increased with increasing amount of La_2O_3 , and the aspect ratio of LaAl₁₁O₁₈ grains increased with increasing hot-pressing time.

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1. Introduction

Ceramic composites that reinforce grains with a high aspect ratio second phase can be fabricated by the addition of whiskers, platelets and fibers. However, these composites have some problems during processing, i.e., difficulties in homogeneous mixing of powders and densifying the composites.^{1,2)} Alumina-based composites with elongated structure can nevertheless be obtained easily through in-situ formation by a solid reaction. Such composites show almost maximum density value due to an in-situ reaction during sintering. Recently, the in-situ fabrication of elongated composites such as corundum-rutile, SrAl₁₂O₁₉/Ce-TZP, LaAl₁₁O₁₈/Ce-TZP, and Al_2O_3 /aluminate platelet composites has been reported.^{3)–6)} In those studies, calcination was used for the in-situ formation of LaAl₁₁O₁₈⁵⁾ and dopants for the in-situ formation of various aluminate platelets.⁶⁾ These processing methods are complicated, and the grain morphology of the in-situ formed second phases exhibit a nearly plateletlike grain. The possibility exists, however, that in-situ second grains with an elongated morphology can be obtained by controlling the composition as well as the sintering conditions. The phase diagram of the Al_2O_3 – La_2O_3 system⁷⁾ indicates an LaAl₁₁O₁₈ phase (lanthanum- β -alumina) that exists preferentially within Al_2O_3 with a molar ratio of 1 : 11 for La_2O_3 : Al_2O_3 . The LaAl₁₁O₁₈ phase can be fabricated in-situ from the solid-state reaction between Al_2O_3 and La_2O_3 . In the case of sintering from a mixture of Al_2O_3 and La_2O_3 powders, an Al_2O_3 matrix is compatible with the formation of aluminate compounds such as LaAl₁₁O₁₈ and LaAlO₃.

The objectives of this study were to fabricate Al_2O_3 /LaAlO₃ or Al_2O_3 /LaAl₁₁O₁₈ composites with an elongated second phase by the in-situ reaction between Al_2O_3 and La_2O_3 and to evaluate their microstructural characteristics. The characteristics of the composites were examined by X-ray diffraction (XRD) analysis, and the microstructural properties were observed by scanning electron microscopy (SEM) and transmission electron microscopy (TEM).

2. Experimental procedure

2.1 Fabrication of composites

In order to fabricate alumina-based composites, a high-purity α - Al_2O_3 powder (TM-DAR, Taimei Chemical Co., Japan) with an average particle size of 0.2 μm and an ultrafine La_2O_3 powder (Nihon Yttrium Co., Japan) with an average particle size of 0.3 μm were used as starting materials. Al_2O_3 /aluminate composites were formed by using a controlled composition with a molar ratio of 1 : 0.005 to 1 : 0.02 (Al_2O_3 : La_2O_3). Our study encompassed compositions from 5 to 20 vol% of LaAl₁₁O₁₈ in the second phase for Al_2O_3 /LaAl₁₁O₁₈ composites. The blended powders were homogenized by ball milling for 24h in methanol solution using a polyethylene jar and Al_2O_3 balls. The resultant slurry was dried and then sieved through a 200 mesh screen. Sintering was carried out by hot-pressing. The mixed powders were uniaxially hot-pressed in graphite dies spray-coated with BN powder from 1400 to 1800°C for 0.5 to 5h under 30 MPa pressure in Ar atmosphere. The hot-pressed specimens were nominally cut with a diamond blade to rectangles measuring 3 × 3 × 5 mm. The cut pieces were polished initially with 3 μm diamond paste and finally with 1 μm diamond paste to obtain a mirrorlike surface finish.

2.2 Evaluation of properties

The bulk densities were determined by the Archimedes technique, using water at room temperature. Phases in the hot-pressed bodies were identified from XRD patterns using Ni-filtered Cu K α radiation. Thermal etching was performed at 1500°C in Ar atmosphere to evaluate grain structure. Grain sizes of thermally etched specimens were measured using the linear intercept method.⁸⁾ For microstructural analysis, polished and thermally etched surfaces of specimens were observed using SEM (model ABT 150 F, Topcon Co., Ltd., Japan) and TEM (model EM 420 T, Philips Electronic Instruments, Inc., NJ) with an X-ray energy-dispersive spectroscopy (EDS) analyzer attachment. TEM samples 3 mm in diameter were prepared using

a conventional thinning procedure, including dimple grinding and final thinning by Ar ion milling.

3. Results and discussion

3.1 Fabrication of Al_2O_3 /aluminate composites by in-situ reaction

The XRD results obtained from a specimen prepared using a molar ratio of 1 : 0.02 (Al_2O_3 : La_2O_3) and hot-pressed at various temperatures for 2h are shown in Fig. 1. For the samples hot-pressed at 1400°C, the LaAlO_3 phase was detected in the second phase, as shown in Fig. 1(A). Specimens hot-pressed at or above 1500°C exhibited the $\text{LaAl}_{11}\text{O}_{18}$ phase, as shown in Figs. 1(B) and (C). This means that LaAlO_3 was converted to $\text{LaAl}_{11}\text{O}_{18}$. Hence, the differences in X-ray patterns indicate that the LaAlO_3 phase prepared at 1400°C is an intermediate phase for $\text{LaAl}_{11}\text{O}_{18}$ formation. If sintering took place at or above 1500°C, the LaAlO_3 phase disappears, whereas the $\text{LaAl}_{11}\text{O}_{18}$ phase appears as shown in Figs. 1(B) and (C). It is suggested that the mechanism of the solid-state reaction involved in $\text{LaAl}_{11}\text{O}_{18}$ formation in the Al_2O_3 - La_2O_3 system occurs as follows:⁹⁾

The crystal structure of LaAlO_3 and $\text{LaAl}_{11}\text{O}_{18}$ have a perovskite structure⁹⁾ and a β -alumina structure,¹⁰⁾ respectively. $\text{LaAl}_{11}\text{O}_{18}$ would be formed if the in-situ reaction take place according to Eqs. (1) and (2). The formation of LaAlO_3 and $\text{LaAl}_{11}\text{O}_{18}$ is associated with the sintering conditions. $\text{LaAl}_{11}\text{O}_{18}$ has a stoichiometric alumina-rich composition in which the La to Al ratio is 1 : 11. However, when an La_2O_3 and Al_2O_3 mixture of 1 : 11 ratio was sintered, it did not react completely to form the stoichiometric $\text{LaAl}_{11}\text{O}_{18}$ when sintering temperature was below 1500°C. There is a possibility that La_2O_3 reacts with the Al_2O_3 matrix to form the LaAlO_3 phase due to the reaction shown in Eq. (1). However, its reaction rate depends mainly on the sintering temperature. At 1400°C, the reaction of Eq. (1) is rapid whereas that shown in Eq. (2) is relatively slow and represents the limiting factor in the overall formation of stoichiometric $\text{LaAl}_{11}\text{O}_{18}$.⁹⁾ Thus, the formation of LaAlO_3 appears to be complete below 1400°C, whereas temperatures higher than 1500°C are required for the complete formation of $\text{LaAl}_{11}\text{O}_{18}$. Equation (2) is important for the

development of elongated $\text{LaAl}_{11}\text{O}_{18}$ grains. Above 1500°C, the reaction shown in Eq. (2) is predominant, because it is known that LaAlO_3 is the intermediate phase during the solid-state formation of $\text{LaAl}_{11}\text{O}_{18}$. Ropp and Carroll⁹⁾ reported that the solid-state formation of $\text{LaAl}_{11}\text{O}_{18}$ occurs via the reaction between LaAlO_3 and Al_2O_3 from the Al_2O_3 - La_2O_3 system and the formation of LaAlO_3 appears to be completed at 800°C. The $\text{LaAl}_{11}\text{O}_{18}$ phase should be formed internally if the in-situ reaction takes place according to the reaction between either 1 La_2O_3 and 11 Al_2O_3 or LaAlO_3 and 5 Al_2O_3 . The XRD results in Fig. 1 indicates that the formation of an $\text{LaAl}_{11}\text{O}_{18}$ phase is influenced by the hot-pressing temperature and a temperature above 1500°C is required to fabricate the Al_2O_3 / $\text{LaAl}_{11}\text{O}_{18}$ composite. The $\text{LaAl}_{11}\text{O}_{18}$ phase, which has a hexagonal structure, resembles the structure of sodium beta(β)-alumina, and their phase diagram is similar to that of sodium beta alumina.¹¹⁾ Thus, for $\text{LaAl}_{11}\text{O}_{18}$, two oxygens per La would be expected in the cation plane. The powder X-ray diffraction data for $\text{LaAl}_{11}\text{O}_{18}$ on the basis of hexagonal indexing with lattice parameters of $a_0 = 0.5561 \pm 0.0007$ nm and $c_0 = 2.2041 \pm 0.0004$ nm were obtained.¹²⁾ The morphology of the matrix and the second phase is also affected by hot-pressing temperature.

Figure 2 shows the microstructure of Al_2O_3 /20 vol% aluminate composites hot-pressed at various temperatures for 2h and thermally etched. The grain boundary phase is dissolved by thermal etching. Figure 2(A) shows the needlelike shape of LaAlO_3 which has an aspect ratio of about 4–5, though its samples hot-pressed at 1400°C. For Al_2O_3 /20 vol% $\text{LaAl}_{11}\text{O}_{18}$ composite, the Al_2O_3 matrix grains are equiaxed, while the second phase $\text{LaAl}_{11}\text{O}_{18}$ grains are elongated, as shown in Figs. 2(B) to (D). The elongated $\text{LaAl}_{11}\text{O}_{18}$ grains having an aspect ratio of 5–9 are dispersed in the Al_2O_3 matrix. The high aspect ratio of this sample could be explained by the anisotropic grain growth of $\text{LaAl}_{11}\text{O}_{18}$, because the lattice parameters of a_0 and c_0 are markedly different. Hence, it is possible that grain growth can occur in one direction. The elongated $\text{LaAl}_{11}\text{O}_{18}$ grains exist within the Al_2O_3 matrix grains and at grain boundaries. Some $\text{LaAl}_{11}\text{O}_{18}$ grains with small aspect ratios exist as an intragranular phase between Al_2O_3 matrix grains. Other elongated $\text{LaAl}_{11}\text{O}_{18}$ grains exist at alumina grain boundaries as an intergranular phase. Figures 2(C) and (D) conform that grain growth of in-situ formed $\text{LaAl}_{11}\text{O}_{18}$

Fig. 1. XRD results of hot-pressed specimens with Al_2O_3 : La_2O_3 = 1 : 0.02 as a function of hot-pressing temperature.

Fig. 2. SEM micrographs of thermally etched surfaces of Al_2O_3 /20 vol% aluminate composites hot-pressed at various temperatures for 2h: (A) 1400°C, (B) 1500°C, (C) 1700°C and (D) 1800°C.

occurred with increasing hot-pressing temperature. In particular, the size of the elongated grains increased with increasing temperature, resulting in the formation of plateletlike grains at temperature above 1700°C. Plateletlike grains could be grown by increasing the sintering temperature of composites. The morphology of the second phase depends on the dopant as well as the sintering conditions. It is well established that in-situ doping led to the development of plateletlike grains. Yasuoka et al.¹³⁾ reported that SiO₂ addition (ppm order) is effective for the development of plateletlike LaAl₁₁O₁₈ grains via an in-situ reaction. In addition, the Al₂O₃ matrix grains in the composites of Fig. 2 remained relatively small in size in comparison with monolithic Al₂O₃ grains of Fig. 3, under the same sintering condition. The surface structure of a thermally etched monolithic alumina hot-pressed at 1500°C for 2h and 1800°C for 2h are shown in Fig. 3. The alumina grains are larger and equiaxial. The morphology of alumina matrix is dependent on the dopants used. It was shown that the morphology of alumina grains changed from equiaxial to plateletlike with the addition of a small amount of SiO₂.¹³⁾ Such plateletlike grains are related to liquid-phase sintering due to the existence of dopants. However, no plateletlike grains of alumina were developed in the hot-pressed specimens used in this study. Accordingly, the existence of equiaxial grains in the Al₂O₃ matrix demonstrates that both Eqs. (1) and (2) represent reactions that occur during solid-state sintering. From Figs. 2 and 3, grain growth of Al₂O₃ was proven to be suppressed by the existence of an elongated second phase such as LaAlO₃ and/or LaAl₁₁O₁₈.

Figure 4 shows the effect of hot-pressing temperature on the bulk density of Al₂O₃/20 vol% aluminate composites. Bulk density increase with increasing in hot-pressing temperature, and ranges from 3.74–4.05. The bulk density of specimens hot-pressed above 1500°C indicates nearly full density. The theoretical density of Al₂O₃/20 vol%LaAl₁₁O₁₈ composite (4.06 g/cm³) was estimated by the lever rule from the true density of each compound (Al₂O₃: 3.98 g/cm³, LaAl₁₁O₁₈: 4.36 g/cm³). In ceramic composites that have an elongated second phase, it is generally difficult to obtain specimens with full density

Fig. 4. Bulk density of hot-pressed specimens with Al₂O₃:La₂O₃ = 1 : 0.02 as a function of hot-pressing temperature.

because of the constraint of the second phase network. Ideally, if the microstructural features are developed after densification is completed, via in-situ formation of a second phase that has a highly anisotropic growth habit, then a composite that have an elongated shape can be obtained without difficulty in sintering.¹²⁾ Thus, ZrO₂/LaAl₁₁O₁₈ composites exhibit nearly full density, even when the LaAl₁₁O₁₈ content is increased up to 40 vol%.¹⁴⁾

3.2 Microstructural characteristics

Figure 5 shows a transmission electron micrograph of in-situ formed LaAl₁₁O₁₈ grains in the Al₂O₃/20 vol% LaAl₁₁O₁₈ composite hot-pressed at 1500°C for 2h. LaAl₁₁O₁₈ grains show a dark contrast, but can be easily recognized by its elongated shape (arrows) like as interlocking form of grains. They exist mainly mostly at Al₂O₃ grain boundaries, however, some small LaAl₁₁O₁₈ grains exist within the Al₂O₃ matrix grains as an intragranular phase. In addition, the morphology of LaAl₁₁O₁₈ grains depends on the sintering temperature as well as cooling schedule. When this LaAl₁₁O₁₈ melt was rapidly cooled after sintering, hexagonal platelets were obtained. The direction of rapid growth was parallel to the *a*₀ direction of the hexagonal lattice.¹²⁾

Figure 6 shows that SEM images of morphology and location of LaAl₁₁O₁₈ grains in the Al₂O₃ matrix of Al₂O₃/20 vol%LaAl₁₁O₁₈ composites, at various hot-pressing times. Figure 6(A) shows that short elongated LaAl₁₁O₁₈ grains exist within the Al₂O₃ matrix grains of the Al₂O₃/20 vol% LaAl₁₁O₁₈ composite hot-pressed at 1600°C for 0.5h, as an intragranular phase. Figure 6(B) shows the elongated LaAl₁₁O₁₈ grains in the Al₂O₃/20 vol%LaAl₁₁O₁₈ composite hot-pressed at 1600°C for 5h. Elongated LaAl₁₁O₁₈ grains with a large aspect ratio are needlelike and exist at Al₂O₃ grain boundaries, as an intergranular phase. It was reported that the aspect ratio and the particle size of aluminate grains were dopant-dependent.⁶⁾ For example, when Mg,

Fig. 3. SEM micrographs of thermally etched surfaces of monolithic Al₂O₃ hot-pressed at various temperatures for 2h: (A) 1500°C and (B) 1800°C.

Fig. 5. TEM micrograph of elongated LaAl₁₁O₁₈ in Al₂O₃/20 vol%LaAl₁₁O₁₈ composites hot-pressed at 1500°C for 2h.

Fig. 6. SEM micrographs of thermally etched surfaces of Al_2O_3 /20 vol% $\text{LaAl}_{11}\text{O}_{18}$ composites hot-pressed at 1600°C for (A) 0.5h and (B) 5h.

Fig. 7. Average grain size of hot-pressed specimens as a function of hot-pressing temperature; (A) monolithic alumina, (B) matrix alumina of Al_2O_3 /20 vol% aluminate composites and (C) aluminate grains (LaAlO_3 or $\text{LaAl}_{11}\text{O}_{18}$) of Al_2O_3 /20 vol% aluminate composites.

Sr-Mg and Y-Mg were used as dopants, elongated aluminate grains were found.

Figure 7 illustrates the effects of hot-pressing temperature on the average size of grains in the monolithic alumina and Al_2O_3 /20 vol% aluminate composites hot-pressed at various temperatures for 2h. Monolithic alumina was hot-pressed in a similar manner in order to compare its microstructural characteristics with those of Al_2O_3 /20 vol% aluminate composites. When hot-pressing temperature is increase, Al_2O_3 grain growth takes place in monolithic Al_2O_3 . However, the presence of a second phase is known to hinder matrix grain growth. As shown in Fig. 7, monolithic alumina grains grow sharply as temperature increases, reaching approximately $30\text{ }\mu\text{m}$ at 1800°C . In monolithic Al_2O_3 , grain growth and densification occur simultaneously as intergranular pores are removed via

grain boundary diffusion until a few grains start growing markedly. Figures 7(B) and (C) indicate that the matrix alumina grains and width of the elongated aluminate grains are about 2 to $8\text{ }\mu\text{m}$ and 0.3 to $2.5\text{ }\mu\text{m}$, respectively, in the Al_2O_3 /20 vol% aluminate composites. It was also verified that the aspect ratio of $\text{LaAl}_{11}\text{O}_{18}$ grains increases with increasing hot-pressing time. In general, as the sintering temperature increase, grain size (matrix and/or second phase) and aspect ratio increase. This microstructure refinement is more drastic for alumina matrix than for aluminate grains in the composite. No marked grain growth in the alumina matrix is observed, even at temperature above 1600°C , in Al_2O_3 /20 vol% aluminate composites. Grain growth in the composites, is largely controlled even at high temperature. Grain size distribution is another important parameter of the microstructure, together with the average size of the matrix grains. The size and the distribution of grains in the microstructure are influenced by sintering conditions related to the second phase.

Figure 8 shows the grain size distribution of Al_2O_3 in the matrix of monolithic Al_2O_3 and Al_2O_3 / $\text{LaAl}_{11}\text{O}_{18}$ composites hot-pressed at 1600°C for 1h as a function of $\text{LaAl}_{11}\text{O}_{18}$ content (5 to 20 vol%). All of the Al_2O_3 (5 to 20 vol%) $\text{LaAl}_{11}\text{O}_{18}$ composites have elongated grains, regardless of their $\text{LaAl}_{11}\text{O}_{18}$ content, and the aspect ratio increases with increasing $\text{LaAl}_{11}\text{O}_{18}$ content. All of the composites

Fig. 8. Grain size distribution of alumina matrix for monolithic Al_2O_3 and Al_2O_3 / $\text{LaAl}_{11}\text{O}_{18}$ composites hot-pressed at 1600°C for 1h: (A) monolithic Al_2O_3 , (B) Al_2O_3 /5 vol% $\text{LaAl}_{11}\text{O}_{18}$ composite, (C) Al_2O_3 /10 vol% $\text{LaAl}_{11}\text{O}_{18}$ composite and (D) Al_2O_3 /20 vol% $\text{LaAl}_{11}\text{O}_{18}$ composite.

fabricated in the present work attained >99% of their theoretical density. From the quantitative results of grain-size distribution, the mean grain size of monolithic alumina was determined to be approximately 5.2 μm and it has wide grain size distribution (up to 10 μm) as shown in Fig. 8(A). It is well known that alumina ceramics exhibit grain growth at high sintering temperature, resulting in coarse-grained microstructure. In contrast, the grain size of distribution of Al_2O_3 in the composites became narrow, and the average grain size became small, with increasing $\text{LaAl}_{11}\text{O}_{18}$ content (from 5 to 20 vol%). Thus, the in-situ formed $\text{LaAl}_{11}\text{O}_{18}$ grains act to reduce the matrix grain size and narrow the grain size distribution, making composite matrix more uniformly structured. For $\text{LaAl}_{11}\text{O}_{18}$, a minimum amount of about 5 vol% is required for the development of an elongated morphology in the $\text{Al}_2\text{O}_3/\text{LaAl}_{11}\text{O}_{18}$ composites. Also, a dispersion of 5 vol% of the $\text{LaAl}_{11}\text{O}_{18}$ phase is probably the minimum amount for suppression of grain growth in the matrix due to the existence of a second phase. The in-situ formed $\text{LaAl}_{11}\text{O}_{18}$ phase therefore served as a barrier for typical grain growth in the Al_2O_3 matrix by prohibiting grain boundary migration. It can also be said that the Al_2O_3 matrix grain size depends strongly on the amount of the second phase. Abnormal grain growth of Al_2O_3 could be suppressed by incorporating an equiaxed second phase such as one consisting of SiC and YAG grains.¹⁵⁾ The results of grain size distribution, as shown in Fig. 8, confirmed that grain growth in the Al_2O_3 matrix can also be suppressed by the presence of an elongated $\text{LaAl}_{11}\text{O}_{18}$ second phase. Therefore, the grain size of Al_2O_3 matrix in the composites is smaller than that in monolithic alumina.

4. Conclusions

The fabrication and microstructural properties of in-situ formed Al_2O_3 /aluminate composites with elongated grains in the La_2O_3 - Al_2O_3 system were investigated, and the following conclusions made.

(1) A dense $\text{Al}_2\text{O}_3/\text{LaAl}_{11}\text{O}_{18}$ composite with elongated grains was successfully fabricated by an in-situ reaction. $\text{LaAl}_{11}\text{O}_{18}$ was formed as the second phase by the reaction between Al_2O_3 and La_2O_3 at hot-pressing temperatures above 1500°C. $\text{Al}_2\text{O}_3/\text{LaAlO}_3$ composites, however, existed below 1400°C, indicating that the formation of the second phases is dependent on the hot-pressing temperature. In-

situ formed LaAlO_3 and/or $\text{LaAl}_{11}\text{O}_{18}$ second phases exhibited elongated shapes. The $\text{LaAl}_{11}\text{O}_{18}$ phase with an elongated morphology, developed in-situ in the $\text{Al}_2\text{O}_3/\text{LaAl}_{11}\text{O}_{18}$ composites and its aspect ratio was dependent on the hot-pressing temperature and time.

(2) The morphology and the location of $\text{LaAl}_{11}\text{O}_{18}$ grains in the matrix, depended on the hot-pressing time. Short elongated $\text{LaAl}_{11}\text{O}_{18}$ grains existed within alumina grains as an intragranular phase. The $\text{LaAl}_{11}\text{O}_{18}$ grains with a large aspect ratio existed at Al_2O_3 grain boundaries, as an intergranular phase. The aspect ratio of $\text{LaAl}_{11}\text{O}_{18}$ grains increases with increasing hot-pressing time.

(3) The Al_2O_3 matrix grains of $\text{Al}_2\text{O}_3/\text{LaAl}_{11}\text{O}_{18}$ composites are smaller than those in monolithic Al_2O_3 . Grain growth of Al_2O_3 is suppressed due to the existence of an elongated second phase, such as LaAlO_3 and/or $\text{LaAl}_{11}\text{O}_{18}$.

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