Effect of Sintering Temperature on Flexural Properties of Alumina Fiber-Reinforced, Alumina-Based Ceramics Prepared by Tape Casting Technique

Yasuhiro Tanimoto, PhD[1] and Kimiya Nemoto, DDS, PhD[2]

Purpose: **The purpose of this study was to investigate the effect of sintering temperature on flexural properties of an alumina fiber-reinforced, alumina-based ceramic (alumina-fiber/alumina composite) prepared by a tape casting technique.**

Materials and Methods: **The alumina-based ceramic used a matrix consisting of 60 wt% Al_2O_3 powder and 40 wt% SiO_2-B_2O_3 glass powder with the following composition in terms of wt%: 33 SiO_2, 32 B_2O_3, 20 CaO, and 15 MgO. Prepreg sheets of alumina-fiber/alumina composite in which uniaxial aligned alumina fibers were infiltrated with the alumina-based matrix were fabricated continuously using a tape casting technique employing a doctor blade system. Four sintering temperatures were investigated: 900°C, 1000°C, 1100°C, and 1200°C, all for 4 hours under atmospheric pressure in a furnace. The surface of the alumina-fiber/alumina composite after sintering was observed with a field-emission scanning electron microscope (FE-SEM). A three-point bending test was carried out to measure the flexural strength and modulus of alumina-fiber/alumina composite specimens. In addition, sintered alumina fiber was characterized by X-ray diffraction (XRD).**

Results: **FE-SEM observation showed that alumina-fiber/alumina composite was confirmed to be densely sintered for all sintering temperatures. Three-point bending measurement revealed that alumina-fiber/alumina composite produced at sintering temperatures of 1100°C and 1200°C exhibit flexural strengths lower than those of alumina-fiber/alumina composite produced at sintering temperatures of 900°C and 1000°C; alumina-fiber/alumina composite produced at sintering temperatures of 1100°C and 1200°C exhibit flexural moduli lower than that of alumina-fiber/alumina composite produced at a sintering temperature of 1000°C. Additional XRD pattern of alumina fiber indicated that with increasing sintering temperature, the crystallographic structure of γ-alumina transformed to mullite.**

Conclusions: **There were significant differences in the flexural properties between the alumina-fiber/alumina composite sintered at the four temperatures. This indicates that the choice of optimum sintering temperature is an important factor for successful dental applications of alumina-fiber/alumina composite developed by the tape casting system.**

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INDEX WORDS: fiber-reinforced ceramics, sintering temperature, flexural properties, tape casting, alumina

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Accepted June 23, 2005.

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1059-941X/06

doi: 10.1111/j.1532-849X.2006.00133.x

DENTAL CERAMICS have been commonly used as esthetic restorative materials for crowns, bridges, and fixed partial dentures. Furthermore, increased use of ceramics for restorative procedures and demand for improved clinical performance have led to the development and introduction of several new ceramic restorative materials and techniques.[1-3] For example, various new ceramics, such as castable ceramics and CAD/CAM ceramics, which have been introduced into dentistry since the 1980s, have shown considerable potential as restorative materials. As

compared with conventional porcelain ceramics and metal-ceramics, these new dental ceramic materials offer improved esthetic properties and biocompatibility.[4]

Studies are being conducted on fiber-reinforced composite materials commonly used in restorative dentistry.[5-7] A fiber-reinforced material contains a matrix impregnated with fibers. The material derives its strength from the fiber modulus and strength, which are significantly greater than those of the matrix alone. Further studies, including clinical experiments, have demonstrated the feasibility of employing fiber-reinforced composites in dental appliances such as crowns, bridges, and frameworks.[8,9] These systems of dental materials using fibers are being developed one after another, and are being introduced by many companies.[10,11] These systems use continuously oriented fibers preimpregnated with monomers that are ready for curing with heat or light under pressure.

A previous study by the present authors developed an alumina fiber-reinforced, alumina-based ceramic (alumina-fiber/alumina composite) material fabricated by means of a tape casting technique as a new type of dental ceramic.[12] The alumina-fiber/alumina composite fabricated in the previous study is an all-ceramic material composed of alumina fiber reinforcement and alumina-based ceramic. As a result, the multifilament alumina fibers are well infiltrated into the alumina-based ceramics by using the tape casting method. Moreover, the results of our previous study demonstrate that the shrinkage and flexural properties of alumina-fiber/alumina composite represent an improvement over unreinforced alumina, and the beneficial effects of fiber reinforcement can be seen. However, a detailed study of alumina-fiber/alumina composite has not yet been reported.

Clearly, the strengths of alumina-fiber/alumina composite are dependent on variables such as manufacturing processes and chemical composition.[13-16] Sintering temperature is an especially important factor in fiber-reinforced ceramic systems, because sintering temperature determines fiber degradation, sintering degree, and porosity, all of which can affect the strength of ceramics; sintering temperature has a significant effect on strength.[17,18] Therefore, optimization of the strength and microstructure of fiber-reinforced ceramics by suitable selection of sintering parameters is needed; however, the effect of sintering temperature on strength of alumina-fiber/alumina composite developed by the tape casting technique has not been investigated in detail.

The purpose of this study was to investigate the effect of sintering temperature on the flexural properties of an alumina-fiber/alumina composite produced by the tape casting technique.

Materials and Methods

Fabrication of Alumina-Fiber/Alumina Composite

Figure 1 shows a flowchart of the fabrication process of alumina-fiber/alumina composite. An alumina-based powder, consisting of 60 wt% alumina powder (α-Al_2O_3, AL45-1, Showa Denko Co., Ltd., Tokyo, Japan) and 40 wt% SiO_2-B_2O_3 glass powder (SNK-01, Senyo Glass Co., Ltd., Osaka, Japan) with a composition (wt%) of 33 SiO_2, 32 B_2O_3, 20 CaO, and 15 MgO, was used as a matrix for the alumina-fiber/alumina composite. SiO_2-B_2O_3 glass powder was added to the alumina powder in order to lower the sintering temperature, to avoid degradation of the alumina fibers during sintering of the alumina-fiber/alumina composite.

First, an alumina-based aqueous slurry was prepared by mixing 18 g alumina powder, 12 g SiO_2-B_2O_3 powder, 0.12 g dispersant (AQ-2559, Lion Co., Tokyo, Japan), 6 g distilled water (Wako Pure Chem. Ind. Ltd., Osaka, Japan), and 6 g ethanol (Wako Pure Chem. Ind. Ltd.) in

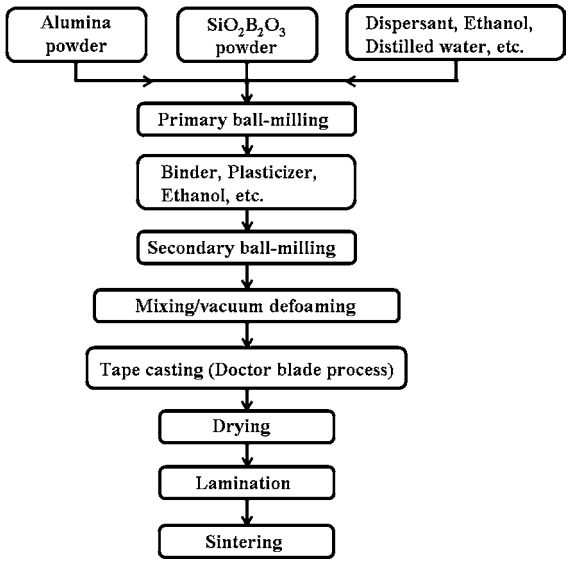

Figure 1. Fabrication process of alumina-fiber/alumina composite.

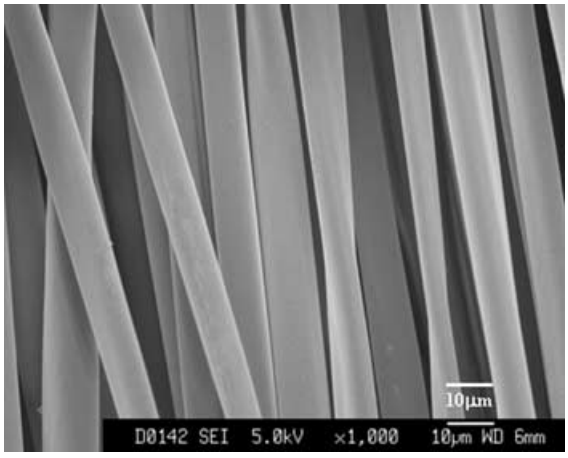

Figure 2. FE-SEM image of alumina fiber used as a reinforcement for the alumina-fiber/alumina composite.

an Al_2O_3 container with two sizes of Al_2O_3 balls (ϕ = 3, 5 mm) for 18 hours with a planetary ball mill (P5/2, Fritsch Japan Co., Ltd., Kanagawa, Japan). Next, 4.8 g binder (HB-500, Lion Co.), 0.6 g plasticizer (PEG #600, Lion Co.), 0.03 g dispersing agent (1020H, Lion Co.), 0.06 g ammonia water (25% ammonia solution, Wako Pure Chem. Ind. Ltd.), and 1.5 g ethanol were added to the initially prepared alumina slurry and further mixed by ball milling for 2 hours. Finally, the alumina slurry was degassed by means of a rotary pump for 30 minutes.

The reinforcement used was alumina fiber (γ-Al_2O_3, Alflex, Taimei Chem. Co., Ltd., Nagano, Japan) having a mean diameter of 10 μm, as shown in Figure 2. The alumina fibers as manufactured are not uniform in cross-section along the length and exhibit slight variation in diameter along the fibers' longitudinal direction, ranging from about 9 to 11 μm. The alumina fibers were preheated at 700°C for 30 minutes, to remove binder before the tape casting. Prepreg sheets of alumina-fiber/alumina composite in which the uniaxial aligned alumina fibers infiltrated the alumina matrix were fabricated continuously by a tape casting technique with a doctor blade system (DP-150, Sayama Riken Corp., Saitama, Japan), as shown in Figure 3. Here, the alumina fibers are fixed to the carrier film, which moves at a speed of 20 cm/min, so that the alumina fibers become impregnated by running through the alumina-based slurry. The heights of blades A and B were adjusted to 1.0 and 1.5 mm, respectively. The width of the alumina-fiber/alumina composite sheet was 150 mm. This width is limited only by the size of the doctor blade machine. The sheet was then dried at room temperature to remove the solvents. Monolayer pieces were then cut to specimen geometry, laminated, and pressed together to prepare multilayer preforms. Then, five plies of the alumina-fiber/alumina composite sheets were stacked and pressed at 100°C under a pressure of 21 MPa for 30 minutes, before sintering. Thus, alumina fibers can be aligned in parallel not only to the long axis but also to the thickness direction, because the alumina-fiber/alumina composite sheets that the alumina fibers were previously impregnated with were laminated later, after the drying process.[12,19] The multilayer preforms of the alumina-fiber/alumina composite sheets were sintered at a maximum temperature of 900°C, 1000°C, 1100°C, or 1200°C under atmospheric pressure for 4 hours in a furnace (MSFT-1520-P, Nikkato Corp., Tokyo, Japan). Consequently, after sintering, alumina-fiber/alumina composite samples having a fiber content of about 6 vol% were obtained. This fiber content was chosen for the present work to prepare a green sheet with high flexibility that allowed for ease of handling and free shaping for the actual clinical application, according to the previous study.[12]

Observation by Field-Emission Scanning Electron Microscopy

After vacuum drying and platinum sputtering of the specimen surface, the surface appearance of the alumina-fiber/alumina composite was observed with a field-emission scanning electron microscope (FE-SEM, JSM-6340F, JEOL, Tokyo, Japan) at an acceleration voltage of 5 kV.

Three-Point Bending Test

The specimens used for the bending test were rectangular bars measuring 20 mm long, 4.5 mm wide, and 1.2 mm thick. Three-point bending tests were

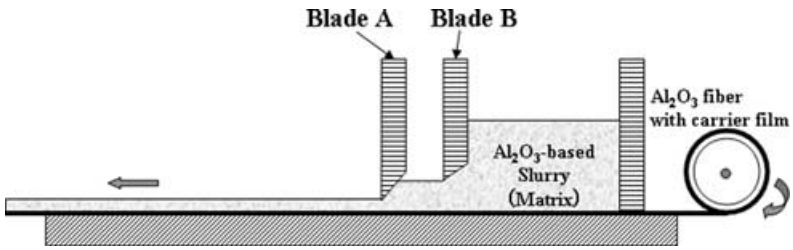

Figure 3. Tape casting system for alumina-fiber/alumina composite sheet fabrication.

performed at a constant loading speed of 0.5 mm/min at a span length of 16 mm, by use of a computer-controlled Instron testing machine (TG-5kN, Minebea, Tokyo, Japan). The flexural strength F and the flexural modulus E were calculated from the following formulae:

$$F = (3/2)(PL/bh^2) \quad (1)$$

$$E = (1/4)(L^3/bh^3)k \quad (2)$$

where P is maximum load, L is span length, b is specimen width, h is specimen thickness, and k is the slope at the initial stage in the load–deflection curve. The experimental values are the average of 12 measurements ($n = 12$). In addition, the orientation of the continuous fiber is along the longitudinal direction.

The results of flexural strength and flexural modulus were examined with an analysis of variance and were tested by the Scheffé multiple comparisons test among the means at $p = 0.05$.

X-Ray Diffraction

The used alumina fiber was characterized by X-ray diffraction (XRD, θ-2θ diffractometer, Geigerflex, Rigaku Corp., Tokyo, Japan), which had an X-ray source of Cu K_α and a power of 40 kV × 30 mA. Samples were placed in an aluminum holder. All collected X-ray spectra were corrected with reference to pure silicon (99.95%) as an external standard.

Results

Figure 4 shows the FE-SEM images of the alumina-fiber/alumina composite surfaces after sintering at four sintering temperatures: 900°C, 1000°C, 1100°C, and 1200°C. At all sintering temperatures, the alumina-fiber/alumina composite was confirmed to be densely sintered.

Figure 5 shows the flexural strength of alumina-fiber/alumina composites plotted against sintering temperature, as obtained from the three-point bending test. Alumina-fiber/alumina composites with sintering temperatures of 900°C, 1000°C, 1100°C, and 1200°C had flexural strengths of 205.4, 185.1, 132.3, and 130.0 MPa, respectively; flexural strength decreases with increasing sintering temperature. The specimen groups with sintering temperatures at 900°C and 1000°C were significantly higher than groups at 1100°C and 1200°C, in flexural strength ($p < 0.05$). Also, the specimens sintered at 1100°C and 1200°C showed no significant difference in flexural strength ($p > 0.05$) between each other.

Figure 6 shows the flexural modulus of alumina-fiber/alumina composites plotted against sintering temperature. Alumina-fiber/alumina composites with sintering temperatures of 900°C, 1000°C, 1100°C, and 1200°C had flexural moduli of 109.4, 118.5, 102.4, and 101.6 MPa, respectively. The

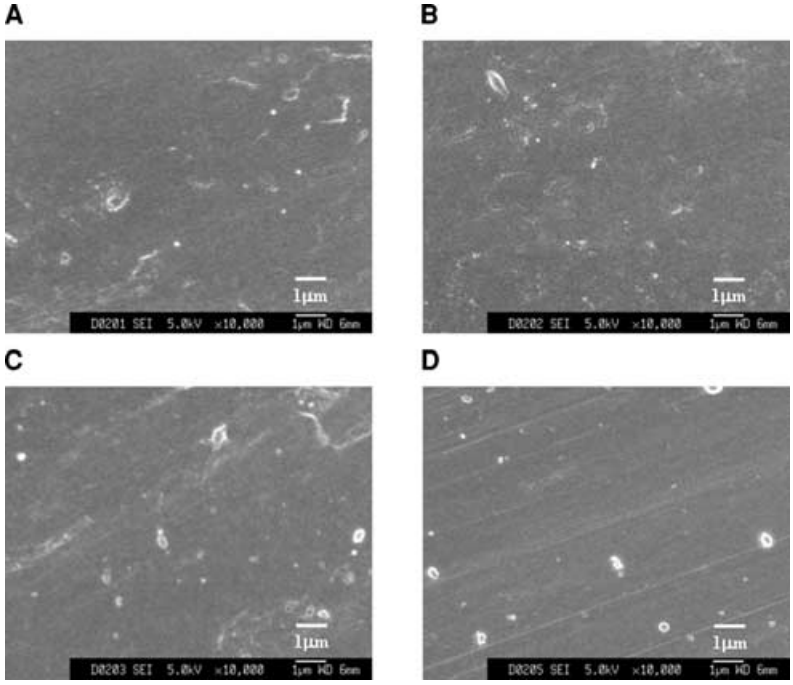

Figure 4. (*A*) FE-SEM image of surface of alumina-fiber/alumina composite after sintering at 900°C. (*B*) FE-SEM image of surface of alumina-fiber/alumina composite after sintering at 1000°C. (*C*) FE-SEM image of surface of alumina-fiber/alumina composite after sintering at 1100°C. (*D*) FE-SEM image of surface of alumina-fiber/alumina composite after sintering at 1200°C.

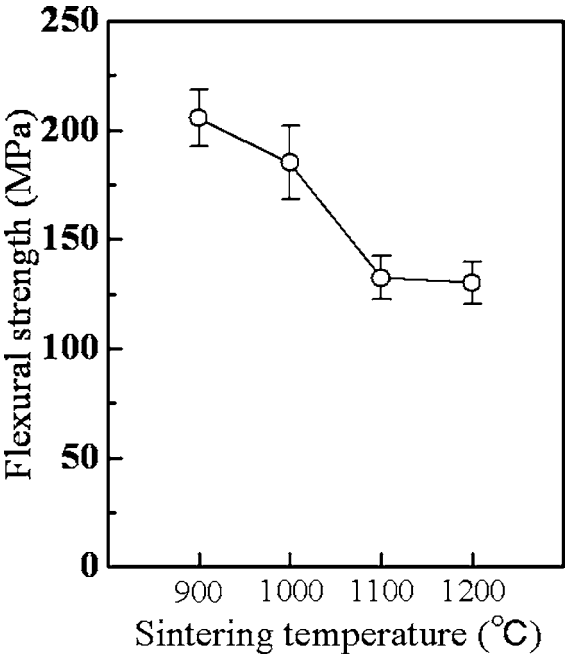

Figure 5. Relationship between flexural strength and sintering temperature.

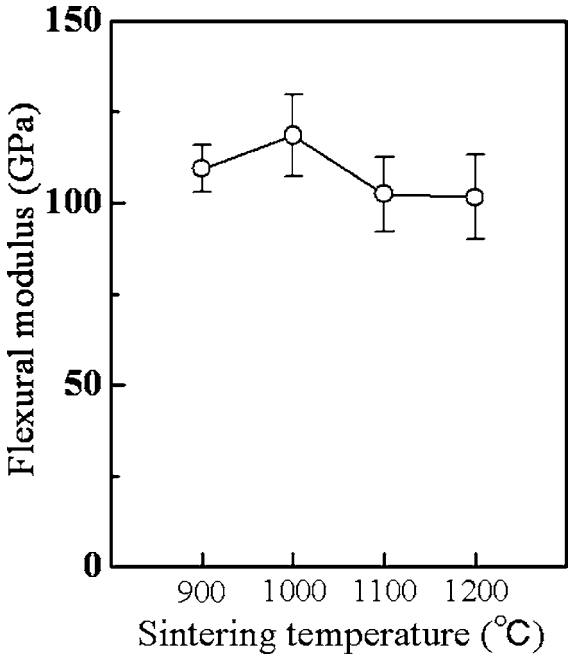

Figure 6. Relationship between flexural modulus and sintering temperature.

flexural modulus increased with increasing sintering temperature up to 1000°C, and decreased with further increase in sintering temperature. Specimens sintered at 1000°C showed significant differences in flexural modulus from samples sintered at 1100°C or 1200°C ($p < 0.05$).

Figure 7 shows the XRD spectra of the alumina fiber after sintering at 900°C, 1000°C, 1100°C, and 1200°C. The spectra of the alumina fiber after sintering at 900°C, 1000°C, and 1100°C confirmed the γ-alumina peaks at 2-theta values of 19.83°, 32.12°, 37.42°, 39.55°, 45.52°, 59.90°, and 67.15°. In contrast, the spectra of the alumina fiber after sintering at 1200°C had mullite peaks at 2-theta values of 16.50°, 26.03°, 26.30°, 31.02°, 33.27°, 35.30°, 37.08°, 39.28°, 40.88°, 42.60°, 48.05°, 49.43°, 53.47°, 53.98°, 57.60°, 58.32°, 60.68°, 63.57°, 64.57°, 65.52°, 66.33°, 69.73°, 70.45°, 70.93°, 74.18°, 74.98°, and 76.77°.

Discussion

We have developed the alumina-fiber/alumina composite by a tape casting technique. The major advantage of the tape casting technique is that the thickness of the ceramic sheet can be adjusted precisely by varying the gap between the blade and the glass surface.[20,21] Mistler et al reported that a tape casting process can be used to produce thin sheets of essentially fully dense iron aluminide.[22] Yeo et al fabricated zirconia-stainless steel functionally graded material by tape casting.[23] Therefore, fiber-reinforced ceramic sheets prepared by the tape casting technique have potential uses in a wide range of dental applications, such as crowns, bridges, laminate veneers, and dental ceramics for many types of restorations in fixed prosthodontics.

Meanwhile, the mechanical properties of fiber-reinforced ceramics are related to factors such as sintering temperature, holding time, and manufacturing processes.[17,18,24,25] Hirata et al[18] and Tanimoto[25] reported that the mechanical properties of fiber-reinforced ceramics are influenced by sintering temperature. In other words, high temperature could produce fiber damage or an inhomogeneous fiber distribution along the infiltration direction. Thus, in the present study, we investigated the effect of sintering temperature on flexural properties of an alumina-fiber/alumina composite prepared by a tape casting technique. Sintering temperature was varied from 900°C to 1200°C in steps of 100°C. Figure 4 confirmed that no alumina powder or pores remained on the surface, and that the surface was smooth for

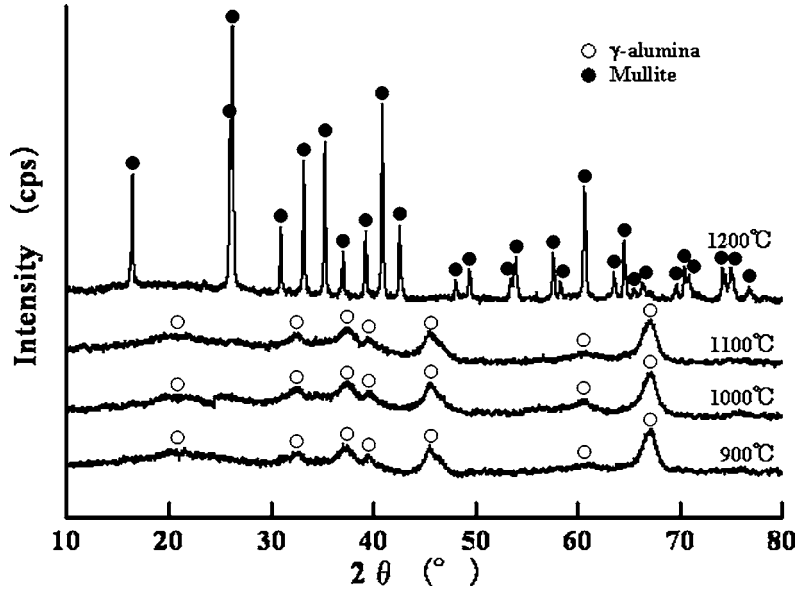

Figure 7. XRD patterns of alumina-fiber after sintering.

all sintering temperatures. This indicates that the alumina-fiber/alumina composite was densely sintered at temperatures of 900°C to 1200°C. In order to promote crystallization of the matrix phase and lower the sintering temperature to avoid the thermal degradation of alumina fibers, in the present study, SiO_2-B_2O_3 glass powder was added to alumina powder. The effect of glasses as additives on the crystallization of ceramics has been investigated.[26-28] Duan et al reported that adding B_2O_3 to the glasses makes the crystal grains more uniform than those in Na_2O-CaO-MgO-Al_2O_3-SiO_2-TiO_2 system glasses.[29] Yang et al reported that the softening temperature of MgO-CaO-Al_2O_3-SiO_2 system glasses decreases with increasing content of B_2O_3 additive.[30] In a detailed study, Tanimoto reported that the optimum Al_2O_3/SiO_2-B_2O_3 weight ratio in a fiber-reinforced ceramics system is 6/4.[25] Thus, applying the same alumina/SiO_2-B_2O_3 weight ratio as used by Tanimoto, we have fabricated alumina-fiber/alumina composites.

The mean values of the flexural strength range from 205.4 to 130.0 MPa (Fig 5). Flexural strength decreases with increasing sintering temperature. The mean values of flexural modulus range from 101.6 to 118.5 MPa (Fig 6). Flexural modulus increases with sintering temperature up to 1000°C, then decreases with further increase in sintering temperature. These results confirm that alumina-fiber/alumina composites produced at sintering temperatures of 1100°C and 1200°C are significantly lower in flexural strength and flexural modulus than alumina-fiber/alumina composites sintered at temperatures below 1000°C. Yoshida et al investigated the effect of sintering temperature on mechanical properties of the SiC fiber-reinforced SiC matrix (SiC/SiC) composite.[17] They reported that sintering temperature affects the characteristics of interfacial bonding between fiber and matrix in the SiC/SiC composite. Thus, our experimental results may be attributed to differences in the interface between individual fibers and the matrix, which is caused by differences in sintering temperature.

XRD spectra of the alumina fiber reinforcement after sintering at 900°C to 1100°C exhibit mainly the peaks of γ-alumina (Fig 7). As sintering temperature was increased (above 1200°C), mullite phase was observed in the XRD pattern. This indicates that the γ-alumina fiber had been fully transformed to mullite after sintering at 1200°C. Actually, an alumina-fiber/alumina composite fabricated in this study is a composite material, composed of alumina-fiber reinforcement and alumina-based ceramic; an alumina-fiber is covered with alumina-based ceramic. In a previous study, we confirmed that the XRD spectra of alumina-based ceramic in alumina-fiber/alumina composite after sintering at 1000°C, had mullite peaks.[12] Therefore, we speculate that the interface between fiber–matrix in alumina-fiber/alumina composite is transformed at approximately 1000°C.

Generally, the reduction in strength of fiber-reinforced ceramic composites is related to the degradation of fiber-reinforcement by extensive transformations. Deléglise et al investigated the tensile strengths of single filaments from room temperature to 1200°C.[31] They reported that fiber strength was almost constant up to 1000°C, and the failure stress decreased rapidly from 1100°C because of fiber degradation. Our experimental results show general agreement with those of Deléglise et al.

The most important feature in flexural properties of alumina-fiber/alumina composite is good stress-transfer ability at the fiber–matrix interface; such stress-transfer can be enhanced by either improving the bond between the fiber and the matrix, or by controlling the fiber surface. Therefore, currently desired interfacial properties in ceramic fiber-reinforced ceramic materials are usually achieved by a coating layer between the fiber and the matrix, which is applied to the fiber deliberately prior to composite consolidation. Hwang et al prepared fiber-reinforced ceramics with $LaPO_4$-coated fiber by the combustion chemical vapor deposition method.[32] Koopman et al fabricated $BaZrO_3$-coated alumina fibers using sol-gel techniques.[13] Bo et al fabricated colloidal $CePO_4$ particles coating short alumina fibers using the layer-by-layer assembly technique.[16] These reports indicate that the fiber–matrix interface plays a key role in determining the mechanical behavior of alumina-fiber/alumina composite. From the above-mentioned point, although the results of the present study indicate the influence of sintering temperature on flexural properties, as well as the relationship between the sintering temperature and flexural properties, more research on the optimization of the fiber–matrix interface is required.

Finally, alumina-fiber/alumina composites fabricated by tape casting technique in the present study have potential for clinical applications, such as crowns, bridges, and dental ceramics for many types of dental prostheses; however, the clinical applications of alumina-fiber/alumina composites have not been investigated in detail. Therefore, the clinical applications of alumina-fiber/alumina composites to dental prostheses such as crowns and bridges should be further investigated.

Conclusions

The present study revealed that both flexural strength and flexural modulus decreased significantly when the crystallographic structure of γ-alumina fiber was transformed. In other words, the strengthening effect of alumina fibers on alumina-fiber/alumina composite could not be fully assessed in case of sintering temperatures of 1100°C and 1200°C, because degradation of alumina fiber could not be completely avoided. To obtain the most effective function of fiber-reinforcement, lowered sintering temperatures are important factors for successful dental applications of alumina-fiber/alumina composite developed by the tape casting method.

Acknowledgments

This work was supported in part by a Grant-in-Aid for Encouragement of Young Scientists (B) (No. 15791143) from the Japan Society for the Promotion of Science and by a grant from the Ministry of Education, Culture, Sports, Science and Technology to promote 2001-Multidisciplinary Research Projects (in 2001 to 2005).

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