Effect of mechanical processing on thermal and mechanical properties of fibrous fumed alumina compacts

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ABSTRACT
In the present study, the fibrous fumed alumina compact with low thermal conductivity is prepared with a mechanical processing technique. The thermal conductivity of the compact remains low up to a temperature of 350°C. For the fibrous compact, the mechanical processing parameters play important role on controlling the thermal insulation capability. The thermal conductivity of compact increases from 0.036 to 0.042 W/m·K by increasing the rotating speed of rotor. On the other hand, the value remains low by increasing processing time. The strength is decreased due to the increase of processing time. Thus, a lower rotating speed and a shorter processing time could result in a compact with favorable thermal insulation and strength. The results can be related to the surface structure of the coated alumina fibers and granulated structure within the compacts.

KEYWORDS
Fumed alumina; thermal conductivity; mechanical processing; thermal insulation; strength

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1. Introduction
Recently, fast industrial growth and large energy usage induces harsh environmental problems [1–3]. In particular, the problems can be related to the waste heat generated from industrial furnaces, factories, buildings and houses. Therefore, to reduce the waste heat more efficiently is an important subject.

To develop a high-performance thermal insulation material is one of the solutions. The ceramic compacts with porous structure, especially nanoporous structure, are preferred for thermal insulation [4–6]. To control the porous structure within the compact, a novel mechanical processing technique has been developed [7]. For the technique, an attrition type mill has been employed. Though the technique is conducted at room temperature without any media (balls and solvent), the powder mixtures receive various stresses, such as compressive and shear stresses, during the processing [8–10].

The glass fiber reinforced nano-silica compacts have been successfully prepared using this technique [9–13]. By applying the mechanical stresses, the fiber’s surface was coated with porous silica nanoparticles. The surface layer composes of pores smaller than 100 nm [9,10]. Along with some silicon carbide (SiC) particles as opacifier [9], an extremely low thermal conductivity around 0.026 W/m·K (at 100°C) is obtained [9,10] by combining cyclic heat method [14–16] and drop calorimeter method [17]. The compacts can thus be used as the advanced thermal insulation materials. However, due to the silica nanoparticles and glass fibers which are unstable at high temperature [18,19], the applications of such silica compacts above 600°C are difficult. For high-temperature applications, alternative fibrous ceramic compacts are needed.

The use of alumina nanoparticles may improve the high-temperature insulation capability. Though a lot of attention has been given to the use of alumina nanoparticles for engineering purposes, its application for thermal insulation is relatively lacking [20]. The present study aims to develop a compact composing of ceramic fiber and fumed alumina particles. The starting materials are fumed alumina nanoparticles, ceramic fibers and SiC powder. The length of ceramic fiber is in the range of several centimeters; the alumina content is as high as 80%. The potential of the fibrous fumed alumina compact for high-temperature application is also evaluated.

The previous study [10] pointed out that the property of the porous ceramic compacts is affected by the processing parameters. Therefore, the present study focuses on the evaluation of processing parameters on the thermal conductivity and strength of the fibrous alumina compacts. Two important processing parameters, rotating speed and processing time, are evaluated. The thermal conductivity measurement was conducted in the temperature range from 100°C to 350°C. The strength of the compacts was also measured. The microstructure characteristics of porous thermal insulation compact are also evaluated.
2. Experimental procedures

In the present study, the starting materials were fumed alumina powder (AEROXIDE®Alu130; Nippon Aerosil, Tokyo, Japan), ceramic fibers (B80, DENKA ALCEN; 80% Al₂O₃ and 20% SiO₂, Denka, Tokyo, Japan) and SiC powder (GMF-6S; Pacific Rundum, Toyama, Japan). The specific surface area of the fumed alumina was 128 m²/g, as determined by a BET method (3Flex Surface Characterization Analyzer, Micromeritics Japan, Chiba, Japan). The equivalent size of alumina nanoparticle was 12 nm, as estimated from the surface area.

The micrograph of the starting fibers is shown in Figure 1. The addition of ceramic fibers might enhance the strength and thermal resistance of the resulting insulation compact [20]. The diameter and length of the fibers were around 4 μm and several centimeters, respectively. The SiC powder was added to manipulate the radiation heat transfer of the compact. The specific surface area of the SiC powder was 5.4 m²/g. The average diameter of SiC was 1.9 μm, as determined by a particle size analyzer (Microtrac MT3300 EXII, NIKKISO, Tokyo, Japan). Figure 2 shows the morphology of starting SiC particles observed by scanning electron microscopy (SEM, JSM-6010LA, JEOL Ltd., Tokyo, Japan).

The composition of the starting powder mixtures is shown in Table 1. The compounding ratio (%) of fumed alumina: ceramic fibers: SiC powder in the powder mixture was 65: 20: 15. For each batch, 100 g powder mixture was used. The starting mixtures were treated by the mechanical processing method, using an attrition type mill (Mechanofusion System, Hosokawa Micron Corp., Osaka, Japan). The apparatus includes a main chamber, 12 stainless blades on a rotor and motor. The clearance between the rotor and chamber was 3 mm [7]. Though no media (balls and liquid) was used, compressive and shear forces were applied on the powder mixtures during the mechanical processing. The effect of following processing parameters was evaluated: (1) the rotating speed and (2) the processing time. The rotating speed was 1000, 1500 and 2000 rpm; the processing time was 1, 5 and 10 min. The temperature and humidity of atmosphere during the processing were set around 300 K and 50%, respectively. The processed powder mixture (fumed alumina/ceramic fiber/SiC) was obtained after the mechanical processing. The morphology of the processed powders was observed by Scanning Electron Microscope (SEM). And the specific surface area of the processed powders was determined by the Brunauer-Emmett-Teller (BET) method. Then, the processed powder was put in a square die and pressed uniaxially to form the board (100 mm × 150 mm × 10 mm), by using a uniaxial pressure of 2 MPa. The apparent density and porosity of the compact board were determined by measuring its dimensions and weight.

In the present study, the thermal conductivity was measured by a method proposed by Ohmura et al. [21]. The measuring temperatures varied from 100°C to 350°C. The details for the method and measuring apparatus can be found elsewhere [21]. The strength measurement was conducted by a three-point bending test, using a universal testing machine. The dimensions of the specimens were 100 mm × 30 mm × 10 mm, cutting from the compact. The span was 80 mm; the crosshead speed was 1 mm/min. For the specimens prepared by each processing condition, three specimens were measured. After measuring the fracture load, the flexural strength was determined.

3. Results

3.1 Characterization of processed powder mixtures

Figure 3(a–e) shows the morphology of the ceramic fibers after the mechanical processing with different
processing parameters. The fibers are coated with a surface layer composed of alumina nanoparticles. With the processing time of 1 min, the fiber prepared with the highest rotating speed (2000 rpm) exhibits a thick surface layer, as demonstrated in Figure 3(c). From Figure 3(a–c), the thickness of surface layer increases roughly with increasing rotating speed. On the other hand, at a constant rotating speed of 1000 rpm, the thickness of surface layer on fibers is more or less the same, as shown in Figure 3(a, d, e).

Figure 4(a–c) shows the SEM images of the alumina particles dispersed in the processed powder with different processing parameters. The images were obtained by observing the processed powder set on the sample holder, and controlling the field of view on the area except fibers. From Figure 4(c), large and porous granules of alumina nanoparticles around several tens of micrometers are observed with the processing parameter of 10 min/1000 rpm. The morphology of the particles is similar as the powder mixture treated with 1 min/1000 rpm and 1 min/2000 rpm, as shown in Figure 4(a) and (b). From the SEM micrographs, it implies that the alumina granule size is affected by the processing time, which may underline the properties of the compact.

Figure 5(a, b) shows the relationships between the specific surface area (SSA) of processed powder as a function of the processing parameters. With the increase of rotating speed, the SSA decreases from 95 m$^2$/g to around 60 m$^2$/g. With the increase of processing time, the SSA also decreases slightly from 95 m$^2$/g to around 80 m$^2$/g. The decrease of SSA in both figures referred to the forming of composite particles after the processing. The large decrease of
SSA in higher rotating speed may be due to the densely packed structure of nanoparticles on fiber surface as shown in Figure 3(c).

### 3.2 Characterization of fibrous alumina compact

Table 2 shows the values of apparent density and porosity of the fibrous alumina compacts with different processing parameters. The table indicates that the fibrous fumed alumina compacts with high porosity and light weight are successfully prepared in the experiment.

Figure 6 shows the fracture surface of the compact with the processing parameter of 1 min/1000 rpm. It is observed that ceramic fibers are dispersed in the fumed alumina compact. The microstructure was almost similar to that obtained by the other processing conditions.

The thermal conductivity of the compacts processed with three different parameters is shown in Figure 7. Within the temperature range measured (100°C–350°C), the fibrous compact processed at
1000 rpm for 1 min exhibits the lowest thermal conductivity, around 0.036 W/m-K. However, the thermal conductivity increases to 0.042 W/m-K as the rotating speed increases to 2000 rpm. The compact treated at 1000 rpm for 10 min also shows a low thermal conductivity of 0.037 W/m-K. The values are close to those of another fibrous alumina as determined by the cyclic heat method [20]. Figure 7 suggests that the rotating speed is an important parameter in terms of thermal conductivity.

Figure 8 shows the strength for the fibrous compact. Error bar in the figure indicates one standard deviation. The strength of the compact treated at 1000 rpm for 1 min is 0.58 MPa. The value is high enough to allow the machining on the fibrous compact. From the figures, it is observed that the strength drops with the increase of processing time.

For investigating the thermal stability at high temperature, the fibrous alumina compact with the processing parameter of 1 min/1000 rpm was heat-treated. The dwelling time was 1 h. Figure 9 shows the relationship between the volume shrinkage and heat treatment temperature. The volume shrinkage of the compact was around −0.5% to +0.5% at the heat treated temperature range from 800°C to 1100°C. The change of volume was very slightly and was almost in the range of measuring error. Therefore, the fibrous alumina compacts prepared in present study have a possibility to be applied as the thermal insulation material for high temperature.

4. Discussion

After mechanical processing, all fibers are covered with alumina nanoparticles (Figure 3). It indicates that the direct contacts between fiber and fiber in the compact are hindered by the presence of alumina surface layer. In addition, since the nanoparticles are attached on the surface of fibers, a uniform dispersion of ceramic fibers within alumina nanoparticles can be achieved. It is therefore possible to manipulate the heat transfer within the fibrous compact with a high loading amount of fibers. The thermal conductivity of the fibrous compact is thus controlled by the alumina nanoparticle layer, despite the fiber content is high. The thermal conductivity of the fibrous alumina compact remains low till a
temperature of 350°C. It will be of interest to investigate its thermal stability at a temperature higher than 350°C.

The compact shows a relatively high thermal conductivity as the powder is processed at 2000 rpm for 1 min, as shown in Figure 7. According to Figure 3(c), the coated alumina layer on the fiber is thick. Such particle morphology may increase the contact areas between coated fiber and other particles, adding to the increase of apparent density of the compact (Table 2). It leads to the increasing solid conduction of heat transfer. On the other hand, the thermal conductivity of the compact with the fiber treated with 1 min/1000 rpm and 10 min/1000 rpm is low. For these two compacts, the coated alumina nanoparticle layer is thin. Their morphology is similar, as shown in Figure 3 (a, d, e). It suggests that the surface structure of fibers affects directly the thermal conductivity of the compact.

In Figure 8, the strength almost not changes with the increase of rotating speed. However, the strength drops with the increase of processing time. The increase of processing time affects the size of alumina granules, as demonstrated in Figure 4. The larger size of granules induces less contact point between particles in resulted compact. A longer processing time thus leads to a lower strength.

5. Conclusions

The effect of mechanical processing conditions on the thermal and mechanical properties of fibrous alumina compact was investigated. The thermal conductivity of the fibrous compact was as low as 0.036 W/m-K. The thermal conductivity remained low with the increase of measurement temperature to 350°C. With the increase of rotating speed, the thermal conductivity increased to around 0.042 W/m-K. The fibrous alumina compact was machineable. The strength decreased with the increase of processing time. Therefore, a lower rotating speed and a shorter processing time are favorable. The thermal and mechanical properties can be related to the surface structure of coated alumina fibers and formation of granules of alumina nanoparticles.

Disclosure statement

No potential conflict of interest was reported by the authors.

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