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Application of Millimeter-wave Heating to Sintering of α-Alumina and Annealing of γ- Alumina†

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Abstract

Millimeter-wave (MM) heating was applied to the sintering of α-alumina and to the annealing of γ-alumina. Dependence of densification on the sintering temperature and time were studied in α-alumina compacts and the behavior of transformation from γ-phase to α-phase on γ-alumina compacts was evaluated from X-ray diffraction patterns. Nearly fully densified α-alumina was obtained at 1523K in the MM-sintering, indicating a decrease of about 250K in the sintering temperature in comparison with conventional sintering. Transformation to α-phase was observed at 1173K for 7.2ks in the MM-annealing. This temperature was lower by 200K than the conventional annealing method.

KEY WORDS: **(Millimeter-wave heating), (α-Alumina), (γ-Alumina), (Sintering) (Transformation)**

1. Introduction

Aluminum oxide has many polymorphs such as α, γ, θ, κ and so on (1). Among these polymorphs, α-alumina is the stable phase and is widely used as an industrial ceramic on account of its excellent mechanical and anti-corrosive properties (2, 3). On the other hand, γ-alumina is mainly used as a functional material such as the support from catalyst. As is well-known, mechanical properties of ceramics strongly depend on grain size and micro-porosity. Accordingly, a highly densified microstructure consisting of fine grains is required for obtaining strong ceramics. In conventional methods, high-temperature and long-time sintering is necessary for complete densification, while such sintering conditions cause grain growth. Thus, it is very important to densify ceramic powder compacts at a low temperature and for a short time to suppress grain growth. To densify the compact of a metastable ceramic powder without transformation is also important for extensive use of these metastable ceramics.

Recently, millimeter-wave heating shows promise as a new heat source, instead of centimeter-wave (especially 2.45GHz). A decrease of densification temperature and well-controllable heating can be expected from the weak temperature dependence of dielectric loss of ceramics and enhancement of mass transfer due to the high-frequency electromagnetic field. Several successful results have been reported on the sintering of ceramics and their composites (4, 5). In the present study, the sintering of high pure α-alumina and annealing of γ-alumina with millimeter-wave heating were examined to obtain basic knowledge on densification and transformation behavior.

2. Experimental Procedures

2.1 Preparation and evaluation of samples

Commercial α-alumina powder and γ-alumina powders (AKP-20 (average grain size 0.55μm) and AKP-G015 (average grain size <0.1μm), Sumitomo Chemical Industries Co. Ltd.), were used as starting materials. Die pressing, cold isostatic pressing and the slip casting methods were used to prepare powder compacts. The diameter and thickness of α-alumina and γ-alumina of compacts were 52 mm and 6-8mm, 10mm and 1mm, respectively. These bodies were dried at room temperature and calcined for 30 min at 873K for α-alumina and at 1073K for γ-alumina. These calcined bodies were sintered or annealed with MM heating and conventional methods.

MM sintering of α-alumina was done in nitrogen at 0.1 Pa and MM annealing was done in air in the range of 1073K to 1673K. Heating and cooling rates were fixed at 20K/min. A high power 28GHz gyrotron generator combined with multi-mode applicator (Fuji Denpa Kogyo, FSS-10-28) was used for MM-sintering and -annealing. In these MM heatings alumina fiberboard was used for thermal insulation. Temperature of the specimen was

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measured by contacting a thermocouple of W/Re or Type-K with the sample surface. Conventional sintering and annealing of samples were made with the same conditions by mean of an electric-furnace.

Densities of sintered α-alumina compacts were measured by the Archimedean method. Transformation of γ to α-phase in annealed bodies was examined using X-ray diffraction (Rigaku, Mini Flex). X-ray diffraction patterns were measured by the θ-2θ method using Cu-Kα radiation (15mA, 30kV).

3. Results

3.1 Sintering of α-alumina

Dependence of relative density on the sintering temperature and time were examined isochronally and isothermally. **Fig.1** (a) shows the variation of relative density of α-alumina sintered for 3.6ks. In the conventionally sintering of α-alumina, relative density was 65%TD at 1373K and increased with increase of sintering temperature, and then amounted to nearly full density (≤97%TD) at 1773K. In the MM sintering of α-alumina, relative density was 72%TD at 1173K and increased with increase of sintering temperature, and finally amounted to nearly full density at 1523K. The MM heating method enables the sintering of α-alumina compacts at about 250K lower temperatures than in the conventional heating.

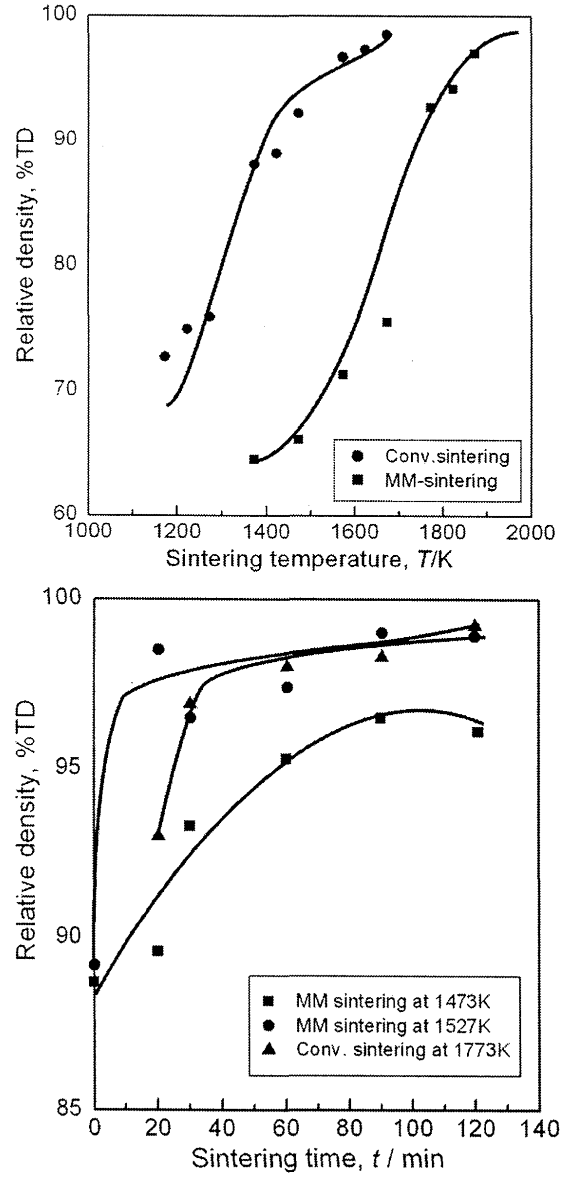

Fig.1 Dependence of relative densities on the sintering temperature and time in α-alumina compacts sintered with millimeter-wave and conventional heating method. (a) Dependence of sintering temperature, (b) Dependence of sintering time.

Fig.1 (b) shows the variation of relative density of α-alumina sintered isothermally at 1573K and 1773K, respectively. Though the difference in the sintering temperature was 200K. It was found that, in MM sintering, a similar densification curve was observed at 200K lower sintering temperature compared with conventional methods. Thus, nearly fully density can be obtained at as low a temperature as 1523K and for short sintering times.

3.2 Annealing of γ- alumina

Dependence of relative density on the annealing temperature was examined. **Figure 2** shows the variation of relative densities of γ-alumina compacts annealed by MM and conventional heating methods for 7.2ks. Relative density of annealed γ-alumina bodies increases with increasing annealing temperature in both heating methods. In the MM heating, a relative density over 50% was obtained at 200K lower annealing temperature compared with the conventional heating method.

Figure 3 shows X-ray diffraction patterns of γ-alumina bodies annealed by conventional and MM heating methods at temperatures from 1073K to 1473K in air. In the conventional heating, γ-Al$_2$O$_3$ phase was kept up to 1273K and changed to mixed phases consisting of α, γ, and θ phase at 1373K, while transformation from γ to α phase occurred at 1373K. Finally, single α-phase was observed at 1473K for 7.2ks.

On the other hand, similar transformation behavior

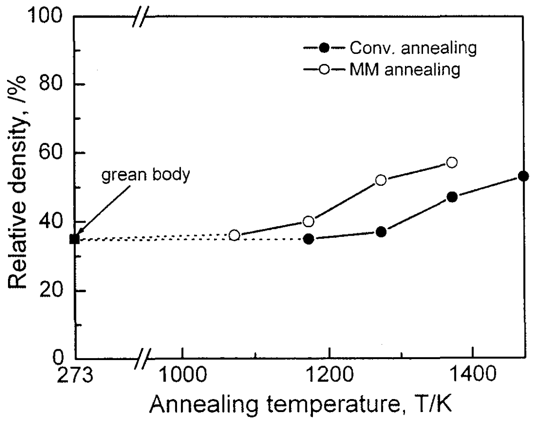

Fig.2 Dependence of relative densities on the temperature in γ-alumina compacts sintered with millimeter-wave and conventional heating method.

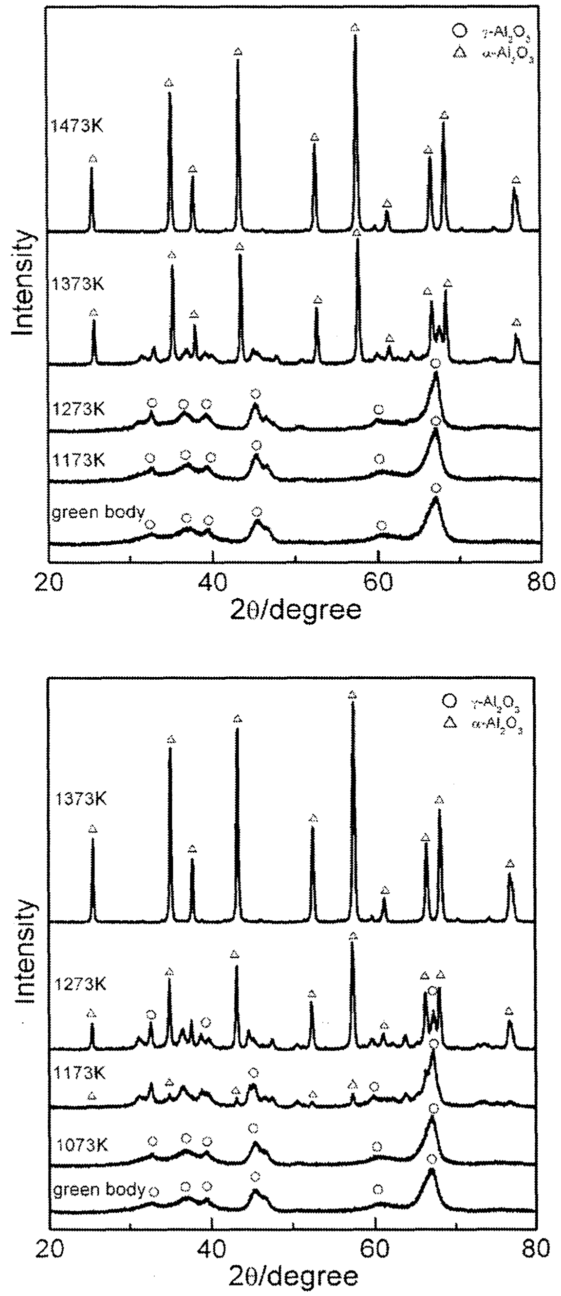

Fig.3 X-ray diffraction patterns of γ-alumina bodies annealed by conventional and MM heating methods. (a) Conventional heating, (b) MM heating.

was also observed in the MM heating method. However, mixed phase was detected at 1173K. The beginning of transformation from γ to θ and/or α phase was 200K lower when compared with conventional a annealing. Thus,MM-heating method enhanced the transformation of metastable aluminas.

4. Conclusions

The conclusions in the present paper are summarized as follows.

(1) Nearly fully densified α-alumina was obtained at 1523K in MM-sintering, indicating a decrease of about 250K in the sintering temperature in comparison with the conventional sintering.

(2) Transformation temperature of γ-alumina in MM annealing was lower than that in conventional annealing. The MM-heating method enhanced the transformation of metastable aluminas to α-alumina.

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