Sintering of the Cold Isostatic Pressed (CIP) Alumina Ceramics Using a Submillimeter Waves Gyrotron

サブミリ波ジャイロトロンを用いて焼結したCIPアルミナセラミックス

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Due to obtain better characteristics of alumina ceramics we have investigated submillimeter waves sintering effect to alumina. Sintering was done by using a submillimeter waves (SMMW) gyrotron. Alumina samples were pressed by using cold isostatic pressing before sintering. Density of sintered samples were measured by using Archimedes method. The densification of SMMW sintered samples then compared to samples sintered by millimeter waves (MMW) from previous results. The CIP treated samples sintered by SMMW show a higher densification than MMW ones. In other hand the samples without CIP treatment show that the samples sintered by SMMW have a lower densification than MM ones. To explain the results, a microstructure evaluation using SEM was performed.

1. Introduction

Frequency of electromagnetic radiation is one of the main roles for determining material properties after heating[1-3]. To investigate effect of highly frequency waves to alumina properties, a high purity alumina was prepared and sintered. Alumina samples sintered by millimeter waves (MMW) for samples without and with CIP treatment show a significant difference both on their density and hardness [1]. Preliminary research using alumina ceramics without CIP treated samples reported that densification of samples alumina sintered using MMW is higher than sintered by using submillimeter waves (SMMW)[2]. In this study, densification of the CIP treated alumina sintered by using SMMW are reported.

2. Experiment

The raw material for the experiment was an α-alumina powder (AES-11C; Sumitomo Chemical Co. Ltd., 99.8 % purity, 400 nm in average particle size, and 7-8 m^2/g of specific surface area) as a starting samples. Powder compacts were prepared by slip casting method. Ammonium salt of poly(methacrylic) acid (NH_4^+ -PMAA; Aron A6114, Molecular Weight 6000, Toagousei Co. Ltd., Japan) is added into the slurry to prevent agglomeration of suspended alumina particles. To remove hard agglomerates prior to compaction, the powders were ball mixed in an alumina jar for 22 hours. A cylindrical samples of 20 mm x 5 mm were obtained then followed by cold isostatic pressing (CIP) at 150 MPa. Pre sintering was done for all samples by heating at 600 ^{0}C in electric furnace (FUA-112DB; Johnson Scie. Equipment Corp.) after dried at room temperature about 12 hours in air. After pre sintering, samples then sintered by using a submillimeter waves gyrotron material processing system (SMMW-MPS) in Research Center for Development of Far Infrared Region, University of Fukui, Japan. The SMMW-MPS are consisted of a 300 GHz, 3.5 kW, CW gyrotron. The whole system is shown in fig.1.

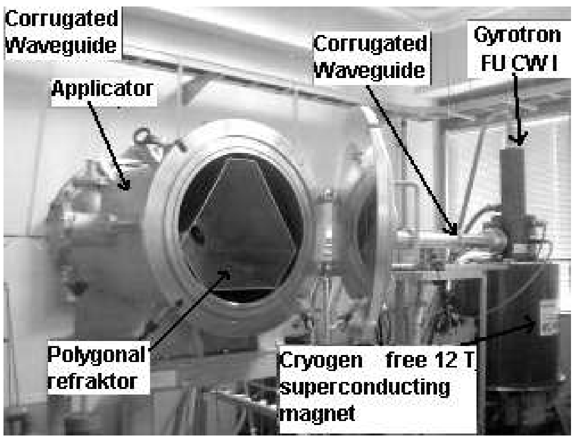

Fig. 1 Whole system of submillimeter wave gyrotron material processing system

The sintering was done starting from final temperature 1100 ^{o}C to 1700 ^{o}C. After reaching final temperature, the temperature was kept about 20 minutes. Densities of the final sintered samples were measured by using the Archimedes method. To characterized the microstructure of samples a scanning electron microscope(SEM) (Hitachi S-2600HS, Hitachi High Tech. Corp., Japan) was performed. Before SEM, the specimens are coated with Pt using ion sputtering Hitachi C-1030.

3. Result and Discussion

Density dependence to final sintering temperature was evaluated and plotted. The density of CIP treatment samples sintered by MMW from previous result in [1] are also plotted in the graph as shown in fig. 2. The graph shows that samples with submillimeter waves heating have a better densification than millimeter waves ones. The highest relative density achieved for SMMW heating is about 99.1 % of theoretical density (TD). From that result we can conclude that for CIP samples the submillimeter waves heating is more effective for densification than millimeter waves ones.

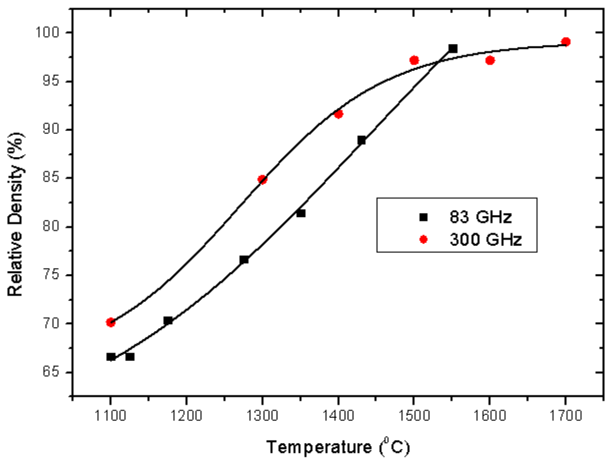

Fig. 2 Relative density of CIP alumina sintered by millimeter and submillimeter waves

This is agreed to previous results by Sano,*et al.* on [1], that CIP alumina sintered by 83 GHz showing a significant higher densities and bending strengths than that sintered by the lower frequency, 30 GHz. To understanding the results a microstructure evaluation for the green samples after pre sintering was done. The relative density of green alumina sample for the CIP treated samples is 58.2 %, with the density difference to without CIP green sample is 2.7% of theoretical density. The differences of microstructure of CIP and without CIP treated green samples may be the one reason that the submillimeter waves heating have better densification than millimeter ones for CIP treated ones as shown in fig. 3 and 4.

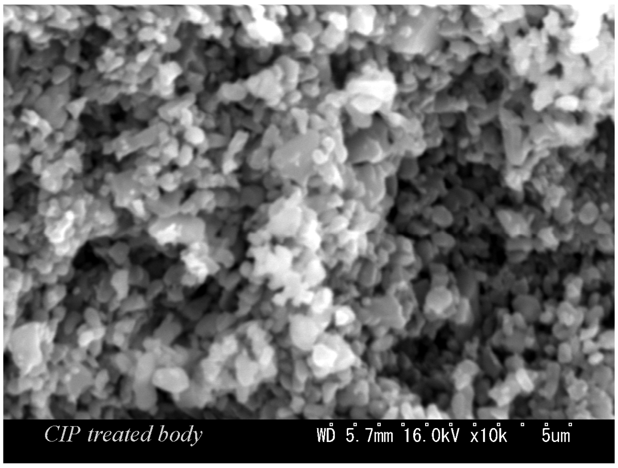

Fig. 3 SEM photograph of CIP treated alumina green sample (D=58.2 % TD).

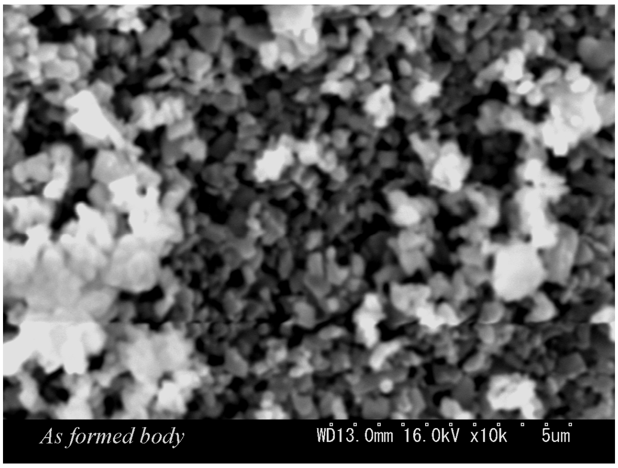

Fig. 4 SEM photograph of not CIP treated alumina green sample (D=55.5 % TD)

4. Conclusion

Densification of CIP treated alumina sintered by submillimeter waves shown higher densification than millimeter ones. The differences of microstructures of the green samples as shown on SEM photograph results may be the one reason of that densification behavior.

References

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[2] S. Mitsudo, *et al.*, FIR Center Report-**105**, (2010).
[3] Y. Makino, *et al.*, J. Advances in Microwave & Radio Frequency Processing,**8,** (2006),570–576.