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Heating and Sintering of Alumina using High-Power Pulsed 60-GHz Gyrotron†

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Abstract

The fine focusing of millimeter-wave radiation from a high-power pulsed 60 GHz gyrotron has been performed using a two-dimensional ellipso-parabolic focusing antenna system and the feasibility to achieve the power density of 100 kW/cm² has been demonstrated. The focused beam profiles have shown excellent agreements with theoretical calculations. In the ceramics heating experiments performed with this focusing system, it has been demonstrated that the green body of alumina (5 wt% SiC) can be heated to 1200 °C in 150 s using millimeter-wave radiation with a 10-ms pulse duration fired at a frequency of 0.5 Hz. Confinement of the incident radiation in the cavity has given an efficient ceramics heating. A preliminary sintering experiment resulted in densification of high-purity alumina from its green-body density of 54% theoretical density (TD) to › 99% TD.

KEY WORDS :(Millimeter-wave radiation) (Gyrotron) (Ceramics heating) (Alumina sintering)

1. Introduction

We have carried out focusing of millimeter-wave radiation from a 60-GHz gyrotron (VGE 8060, Varian). The millimeter-wave radiation from the gyrotron with 0.5-cm wavelength and 200-kW peak power may be focused to obtain a high energy density beam (› 100 kW/cm²). In a series of experiments using quasi optical antenna system, the focused beam profiles have shown excellent agreements with theoretical predictions.[1-2]

The finely focused millimeter-wave radiation has been used for heating and sintering of alumina. Ceramics heating with microwave or millimeter wave is fundamentally different from conventional processes. When microwave radiation propagates through a dielectric material, the alternating electric fields generated internally within the medium induce translational vibrations and rotations of electric dipoles. In the microwave processes, therefore, heat is generated internally within the material in contrast to the conventional processes where the material is heated by the external sources. Due to this volumetric and internal heating, it may be possible to heat the material very rapidly while ensuring sufficient uniformity.

The microwave power absorbed per unit volume P is given by

$$P=2\pi f \varepsilon_0 \varepsilon_r \tan\delta |E|^2 \qquad (1)$$

where f is the frequency of the microwave, ε_0 is the permittivity of free space, ε_r is the relative dielectric constant, $\tan\delta$ is the loss tangent, and E is the magnitude of the internal field. Equation (1) provides the basis for microwave heating ; i.e. the absorbed microwave power varies linearly with the frequency, the dielectric constant, the loss tangent, and the square of the internal electric field.

The attenuation of the incident microwave in the materials can be described by the penetration depth[3] D at which the incident power is reduced by one half.

$$D=\frac{3\lambda_0}{8.686\pi\tan\delta(\varepsilon_r/\varepsilon_0)^{1/2}} \qquad (2)$$

where λ_0 is the free space wavelength. For the heating of materials with smaller dielectric losses, the penetration depth is greater; i.e. uniform heating with smaller thermal gradient is possible. On the other hand, selective and localized heating may be possible by partially doping the materials with higher dielectric losses, which may be applicable to joining of ceramics.

From these points of view, 60-GHz millimeter radiation is one of the appropriate heat sources for processing such ceramics as pure alumina, which are not easily heated by widely used 2.45-GHz microwaves. The major objectives of the present study include (i) to investigate the heating characteristics of alumina using the finely focused millimeter-wave radiation from the 60-GHz gyrotron, (ii) to control the heating rate by appropriately selecting the

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pulse duration and the incident power, and (iii) to efficiently sintere alumina green bodies without suffering appreciable grain growth.

2. Focusing of Millimeter-Wave Radiation

The quasi-optical antenna system used in the present study is schematically shown in **Fig. 1.**
This system, a two-dimensional ellipso-parabolic focusing antenna, consists of a cut-in-half circular waveguide and two cylindrical reflectors. Millimeter waves with the cylindrical TE_{02} mode radiated upwards from the output window of the 60-GHz gyrotron are transmitted through an arc detector, a mode filter with an inner-diameter of 63.5 mm and a circular taper waveguide to reduce the diameter to 25.6 mm. A cut-in-half circular waveguide is directly coupled to the taper to emit the beams obliquely with their electric field almost linearly polarized in the y-direction and to an direction of 25.8 degrees from the z-axis.

First the beams from the cut-in-half antenna are focused to the point **O'** on the **O_2**-axis using the elliptic mirror made of aluminum plate (see Fig. 1 (A)). Second the beams are further focused to the point **F** using the parabolic aluminum-plate reflector (see Fig. 1 (B)). The equation for the elliptic reflector is given by

$$\frac{(x-f_e)^2}{A^2}+\frac{y^2}{B^2}=1 \tag{3}$$

Where A and B are the lengths of the minor axis (86.6 mm) and the major axis (100 mm), respectively, and f_e is the focal length of the ellipse defined as $f_e = (B_2\text{-}A_2)^{1/2}$ = 50 mm. Furthermore the equation for the parabolic reflector is given by

$$z' = -\frac{1}{4f_p}(x'+x'_f)^2 - f_p \tag{4}$$

where $\mathbf{f_p}$ is the focal length of the parabola (74 mm) and $\mathbf{x'_f}$ was given as 122.5 mm. As a result, the focusing point F of the two-dimensional ellipso-parabolic antenna is located at x = -74.7 mm.

The focused beam profiles with the two-dimensional

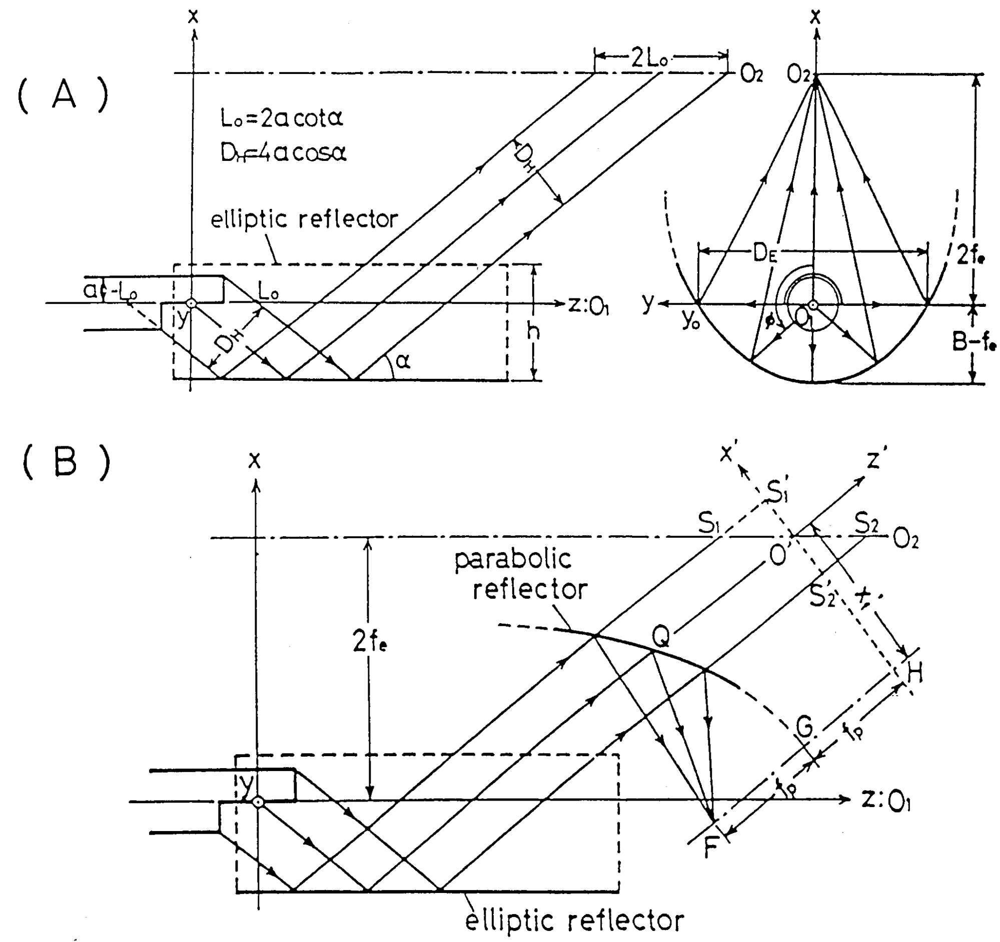

Fig. 1. Schematic diagram of the two-dimensional ellipso-parabolic focusing antenna system.

ellipso-parabolic antenna system were observed at various x-positions. The observation of the profiles were performed by injecting the beams onto a microwave absorber sheet (Eccosorb AN, Emerson and Coming) and measuring the temperature distribution on the sheet corresponding to the profile of the absorbed beam energy. The temperature distributions were measured using a thermal video recorder (TVS-3000, Nippon Avionics).

Shown in **Fig. 2** are the observed profiles of the beams with a pulse duration of 0.5 ms and an output power of 10 kW. Here it should be noted that the focusing position F of the two-dimensional ellipso-parabolic antenna has been designed to be at x = -74.7 mm. As may be clearly seen, at a position of x = -70 mm a finely focused and nearly gaussian beam profile has been obtained with a full width at half maximum of ~10 mm. Furthermore this shows an excellent agreement with theoretical predictions based on a method developed by Wada and Nakajima.[2)] These results ensure that a high energy density beam with 100 kW/cm^2 is easily obtained when the beam power is increased to 100 kW.

3. Heating of Alumina Using High-Power Pulsed 60-GHz Radiation

A series of experiments were performed to investigate the heating characteristics of alumina (100-% Al_2O_3 and Al_2O_3-SiC mixture with 5 wt% SiC) irradiated with the high-power pulsed 60-GHz millimeter-wave radiation. Here the additive (5 wt% SiC) behaves as an assistance to enhance sample heating since its dielectric loss is higher than Al_2O_3. The heating characteristics have been studied in terms of the pulse duration and the power of the irradiated beams since they are directly related to the absorbed radiation energy. The effect of the radiation confinement in a cavity furnace has also been investigated.

First the dependence of increased temperature per one-pulse irradiation on the pulse duration was measured using the experimental setup schematically illustrated in **Fig. 3**. The finely focused beams with a output power of 30 kW were irradiated onto the samples placed in the center of a cylindrical metal cavity (74 mm in diameter and 80 mm in length), which had a beam entrance window with a 39-mm diameter and a temperature measurement window with a 40-mm diameter. Here the cavity was located so that the sample was positioned at the focusing point F (x = -74 mm) of the two-dimensional ellipso-parabolic focusing antenna system, and the pulse irradiation was performed in air at a room temperature. The increased temperatures were measured using the thermal video recorder.

The result is plotted in **Fig. 4** as a function of the pulse duration. For both of the samples, it can be clearly seen that the increased temperature per one-pulse irradiation varies almost linearly with the pulse duration, i.e., the

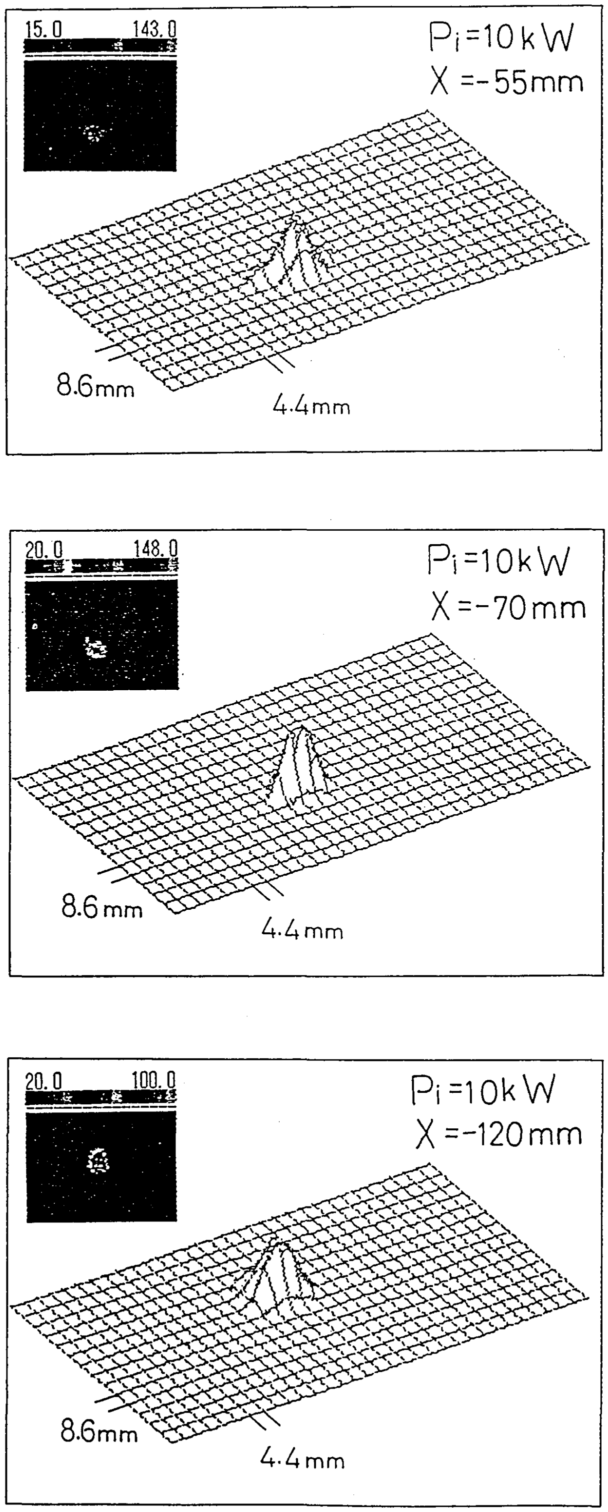

Fig. 2.Focused beam profiles with the two-dimensional ellipso-parabolic focusing antenna system.

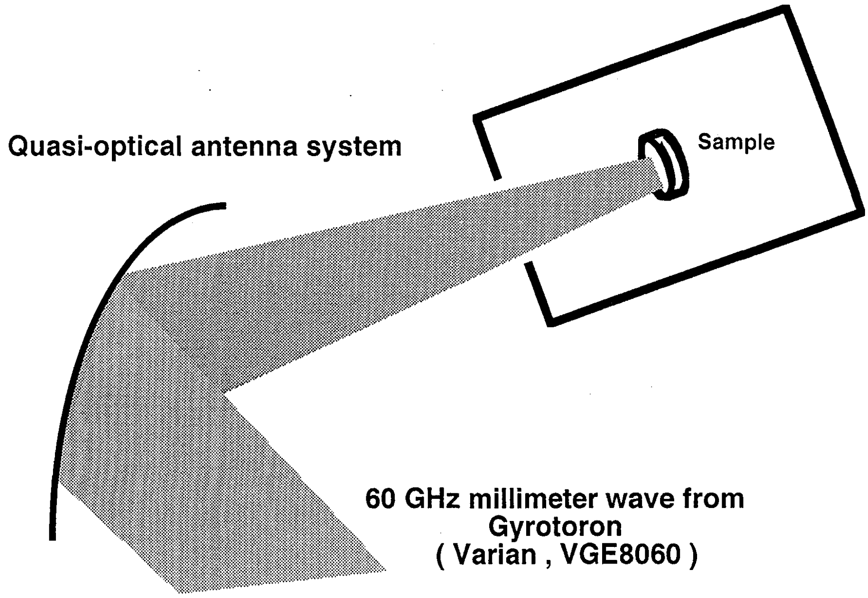

Fig. 3.Experimental setup for the measurement of alumina heating.

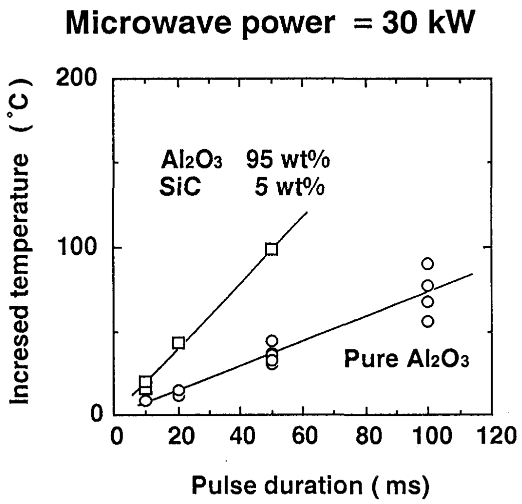

Fig. 4.Increased temperature of alumina as a function of the pulse duration.

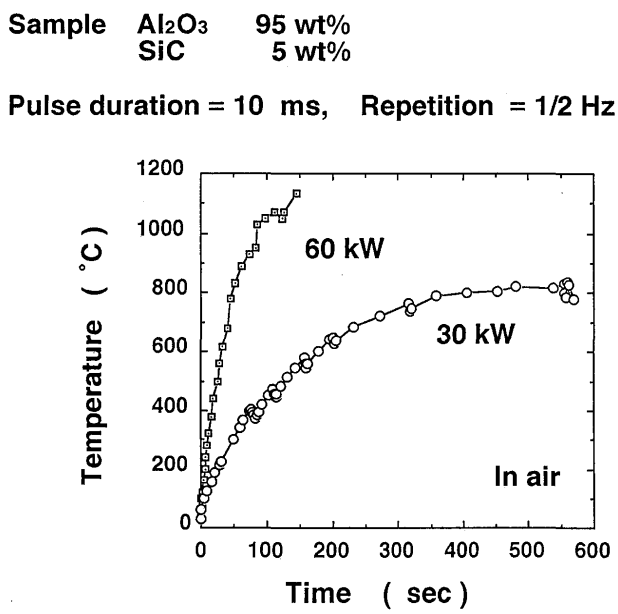

Fig. 5.Power dependence of alumina heating.

absorbed energy. Furthermore doping of the small amount of SiC additive (5 wt%) has resulted in higher temperature increase than that for pure alumina by 2.5 times. This suggests that at a room temperature the dielectric loss of SiC for the 60-GHz radiation is approximately 50 times higher than that of Al_2O_3.

Second the power dependence of the alumina heating was obtained using the experimental setup schematically illustrated in Fig. 3. In this experiment, the focused beams with output powers of 30 kW and 60 kW with a 10-ms pulse duration at a frequency of 0.5 Hz were irradiated onto the samples (5 wt% SiC) placed in the center of a cylindrical metal cavity (100 mm in diameter and 140 mm in length). Here the cavity was located so that the sample was positioned at 70 mm in the rear side of the focusing point F (x = -140 mm), and the pulse irradiation was performed in air at a room temperature.

Figure 5 shows the temporal change of the sample temperature with radiation power as a parameter. Here it should be pointed out that the horizontal axis denotes the real time instead of the sum of the irradiated pulse durations, which is 1/200 of the real time; i.e., the temporally averaged incident powers for 30-kW and 60-kW operations are 150 W and 300 W, respectively. Within the region where the temperature varies linearly with time, the heating rate has been shown to have a linear dependence on the incident radiation power. The reason for the temperature saturation in the case of the 30-kW incident power after 500 s may be due to the balance between the heating rate (the absorbed radiation power) and the cooling rate (the conduction loss and the radiation cooling). Furthermore it has been observed that, even with such a small averaged power as 300 W, the sample can be heated quite rapidly (within 150 s) to ~1200 °C, which is typically required to sintere alumina green body.

From the experimental results obtained so far, we have demonstrated the controllability of the heating rate in terms of the pulse duration and the incident radiation power. Thus, in the next step, the cavity design may become important to efficiently utilize the incident radiation energy for the sample heating. For this purpose, additional heating of the sample by the multiply scattered radiation confined in a cavity may be effective.

The effect of the radiation confinement on the heating characteristics was investigated using a cylindrical cavity with various lengths along the axis of the incident beam. The metal cavity used in the experiment had a dimension of 109 mm in diameter and 420 mm in length. The alumina sample (5 wt% SiC) located at a distance of 140 mm from the incident window (50 mm in diameter) was irradiated with the focused beams with a 29-kW output power and a 10-ms pulse duration fired at a frequency 0.5 Hz. Here the cavity was placed so that the sample was positioned at 215 mm on the rear side of the focusing point F (x = -290 mm), and the pulse irradiation was performed in air at a room temperature. The cavity length was varied by changing the position of the metal reflector located at the rear side of the sample. For comparison, the sample was irradiated with the focused beams in absence of the reflector (W/O reflector).

The dependence of the sample heating rate on the cavity size is summarized in **Fig. 6**. As a result of the experiment, the heating rate has been found to be improved by 1.5-2 times by the confinement of the incident radiation using the metal reflector. This result suggests that approximately the same amount of the absorbed radiation energy directly incident onto the sample can be supplied by the multiply scattered radiations confined in the cavity.

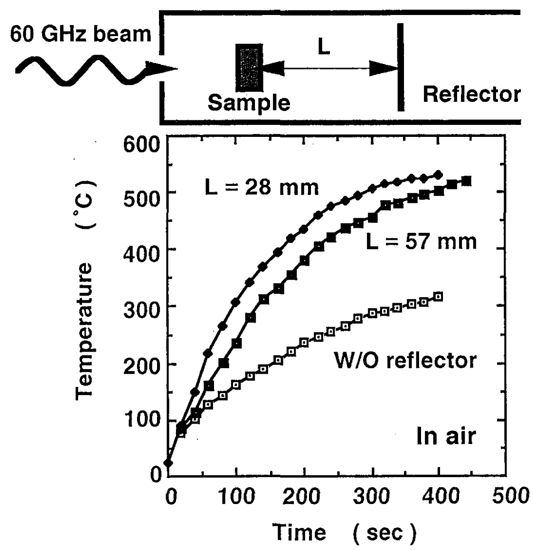

Fig. 6.Dependence of alumina heating on cavity size.

4. Millimeter-Wave Sintering of Alumina

Based on the heating characteristics obtained above, a series of sintering experiments has been performed using the finely focused 60-GHz beams. In the present study alumina has been selected for the initial research. In projecting to higher radiation frequencies, alumina demonstrates considerable merit because high-purity alumina cannot be easily heated using microwaves as low as 2.45-GHz frequencies.

The green-body samples of alumina were prepared by pressing high-purity alumina powder (99.995 wt% Al_2O_3) with an average grain size of 0.2 μm and had a 54-% theoretical density (TD). The samples covered with fiber-ceramics insulator were placed in a metal cavity with a radiation reflector located at a distance of 85 mm from the sample holder. To prevent the conduction heat losses, the cavity was evacuated to 5×10^{-6} Torr using a turbo-molecular pump.

The heating rate and the cooling rate of the samples were controlled by appropriately selecting the pulse duration and the output power from the gyrotron. Typical temperature history involved in the process is shown **Fig.7**. In this example the sample was heated with a rate of ~1 °C/s and was kept at a temperature of 1400 °C for sintering. For characterizing the sintered alumina, the grain size was observed with scanning electron micrographs of the fracture surfaces.

The fracture surfaces of the green body and the sintered alumina densified to 99 % TD are compared in **Fig. 8.** These micrographs show that the densification of the high-purity alumina has been achieved without suffering appreciable grain growth, which is in contrast to the

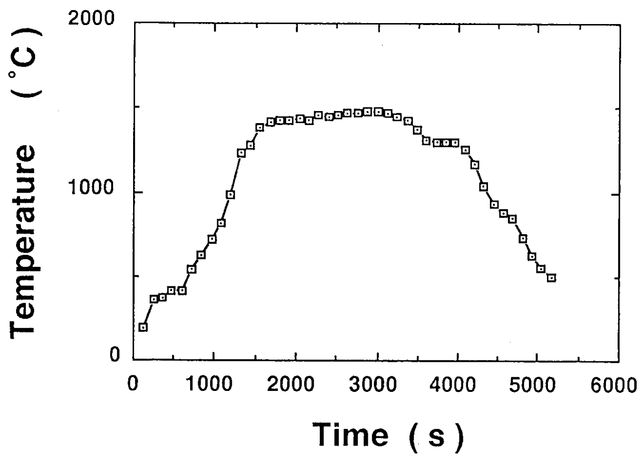

Fig. 7. Typical firing schedule for alumina sintering using the finely focused 60-GHz radiation.

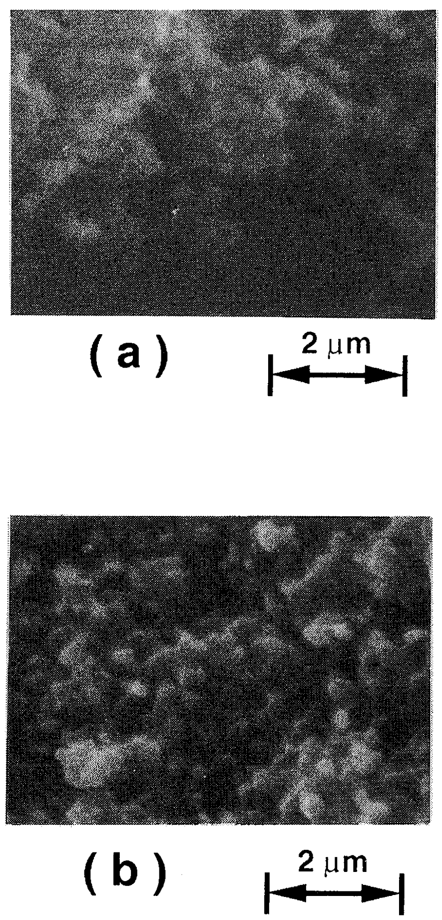

Fig. 8. Fracture surfaces of high-purity alumina; (a) green body, 54 % theoretical density (TD), and (b) sintered alumina, 99 % TD.

conventional sintering process. This feature may be due to the fact that densification in the microwave sintering progresses at lower temperature than in the conventional sintering method.[4-5]

5. Summary

The fine focusing of the millimeter-wave radiation has been performed using the two-dimensional ellipso-parabolic antenna system. The focused beam profiles have shown excellent agreements with the theoretical predictions. Furthermore the feasibility of the high energy-density millimeter-wave beams with › 100 kW/cm^2 has also been shown.

The high-power pulsed 60-GHz radiation beam has been applied to the alumina heating and its characteristics have been investigated. As a result, the controllability of the sample heating has been demonstrated in terms of the pulse duration and the incident beam power. Furthermore radiation confinement in the metal cavity has given an efficient sample heating. A preliminary sintering experiment resulted in densification of high-purity alumina from its green-body density of 54% theoretical density (TD) to › 99% TD, without suffering the appreciable grain growth.

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