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of Alumina Cylinders

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SUBMITTED TO: 1994 Spring Materials Research Society Meeting
April 4-8, San Francisco, CA

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TEMPERATURE DISTRIBUTION IN MICROWAVE SINTERING OF ALUMINA CYLINDERS

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ABSTRACT

Small cylinders of high-purity alumina were encased in a "casket" of low-density zirconia insulation and heated to sintering temperature in a large multi-mode microwave oven. Optical fiber sensors were used to monitor the temperature at several locations in the system. It was found that the alumina samples heat faster than the zirconia insulation at temperatures above 1000°C, and that the temperature distribution in the sample is essentially uniform during the heating process.

A two-dimensional mathematical model of the heat transfer process was developed which reproduces the essential features of the observed phenomena. Literature data for all temperature-dependent properties were incorporated into the model. The model suggests that the alumina samples absorb a significant fraction of the microwave energy.

INTRODUCTION

Successful sintering of multiple small alumina cylinders in a large multi-mode microwave oven at 2.45 GHz has previously been reported [1]. In that work the samples were enclosed in an insulating "casket" of low-density alumina or zirconia insulating board. Temperatures were measured by optical-fiber sensors at a single location within the enclosure. In spite of the success of this work, several questions remained. In particular, nothing was known about the temperature distribution within the samples or insulation during the heating cycle. Thus it was not clear whether the samples were being heated primarily by absorption of the microwave energy, or indirectly by radiation and convection from the insulating material. Even though the samples were sintered without cracking, the degree of uniformity of the temperature distribution during the heating cycle remained unknown. Answers to these questions were sought through more detailed temperature measurements on single cylindrical alumina samples, and by development of a detailed mathematical model of these experiments. Comparison of model calculations and measured temperature distributions lead to some interesting conclusions, but some questions remain.

Figure 1: Alumina sample in zirconia insulation casket with top removed.

MATHEMATICAL MODEL

Since the system being modeled consists of a small cubic container (256cm^3) enclosed in a large ($5.66 \times 10^4\text{cm}^3$) cavity, it was assumed that the microwave field is uniform at the surface of the system being heated. The field strength within the insulation and sample was calculated as described below. Since the sample is cylindrical (see Fig. 1), the insulating container was modeled as a cylinder with the same volume as the actual cubic container. Thus the problem was formulated as a 2-dimensional ($r-z$) cylindrical system. The mathematical problem may be stated as

$$\frac{1}{r} \frac{\partial}{\partial r} (kr \frac{\partial T}{\partial r}) + \frac{\partial}{\partial z} (k \frac{\partial T}{\partial z}) + \dot{q}(r, T) = \rho c_p \frac{\partial T}{\partial t}, \quad (1)$$

where T represents the absolute temperature at location r at time t , ρ is the density of the material, c_p is its heat capacity at constant pressure, k is the thermal conductivity, and $\dot{q}(r, T)$ is the volumetric heat generation rate created by dissipation of microwave energy. $\dot{q}$ is computed from

$$\dot{q}(r, T) = 2\pi f \epsilon''(T) |E_f|^2, \quad (2)$$

where ϵ'' is the dielectric loss coefficient of the material at microwave frequency f , and E_f is the local field strength. Eq. (1) describes the temperature in both the sample and the insulating container, when appropriate properties are used for each. The sample surface and the interior wall of the insulating container exchange heat by radiation except where the sample rests on the container floor. Here, heat exchange occurs through a contact conductance h_c . At the outer surface, the container exchanges heat by radiation and convection

with the cavity walls and the air within the cavity, respectively. All of these heat transfer mechanisms are accounted for in the model.

As stated above, the microwave field strength E_f is assumed uniform at the outer surface of the container. Within the container, E_f is diminished by reflection at each interface according to standard methods [2], and is attenuated exponentially with distance from the surface using the "skin depth" approach [2]. Admittedly, this is no substitute for a complete solution of the coupled electromagnetic field and heat transfer equations, but it was considered a reasonable approach to obtain useful results from a problem of manageable size and complexity. The success of this approach will be discussed below.

Eq. (1) is solved using a fully implicit finite-difference scheme [3]. A typical solution utilizes a 40×40 $r-z$ mesh, for a total of 1600 mesh points. The resulting algebraic equations are solved using the sparse-matrix routines Y12m [4]. The time step size is started at 1/3s and decreased as necessary to ensure convergence of inner iterations.

MATERIAL PROPERTIES

In order to mimic physical reality as closely as possible, material properties were treated as temperature-dependent in all cases where reasonably reliable data is available. In the experiments reported here, the samples were high-purity alumina, and the insulation was highly porous yttria-stabilized zirconia. Curve fits for thermal conductivity and specific heat for these materials were generated from literature data. The alumina samples were initially at approximately 55% theoretical density, and reached 98% T.D. during sintering. Thus the density of these samples was treated as a linear function of temperature between these extremes.

The most important temperature-dependent property in this work is, of course, the dielectric loss coefficient ϵ'' . For alumina, we used the data of Hutcheon, et al. [5], and for zirconia, the data of Arai, et al. [6]. The zirconia data [6] only extends to 1200°C, but appears to increase linearly with temperature at all temperatures greater than about 800°C. Thus we extrapolated the available data linearly for temperatures above 1200°C. The zirconia data of Arai et al. [6] is given at two different values of density. Comparing these two curves suggests that ϵ'' of zirconia increases approximately as the third power of density, so this rule was used to correct ϵ'' for porosity. Unfortunately, Ref. [6] does not mention the composition of the zirconia samples used in their measurements.

RESULTS

Data from a typical sintering experiment is displayed in Fig. 2, along with model results. The curve labelled "sample" is the temperature measured by an optical fiber probe positioned at approximately the center of the alumina sample, while the curve marked "insulation" is the temperature measured by another probe positioned approximately halfway through the wall of the zirconia insulation "casket". The temperature monitoring system does not function for temperatures below 600°C. For this experiment, the magnetron power

Figure 2: Measured and computed temperatures during sintering run. The temperatures were measured with optical fiber sensors in holes drilled into materials.

was set at 20% of maximum and maintained at that value throughout the experiment.

The field strength in the cavity at the sample surface was not known in any of these experiments. Consequently, an attempt was made to find the field strength by trial and error in model simulations by matching the final equilibrium temperatures between simulation and experiment. This was not successful, as the system either did not heat at all if the field strength was chosen too small, or the temperatures increased without bound ("thermal runaway") if E_f was chosen too large. Since the actual temperatures always reach equilibrium, we concluded that the microwave field at the surface of the "casket" decreases as the material begins to absorb microwave energy. Thus the computer model was modified to gradually decrease the field strength as the sample temperature increases. This approach leads to the results shown in Fig. 2, which are in reasonable agreement with experimental data. These results do emphasize the importance of using a coupled solution of the electromagnetic field and temperature equations, an approach which is currently being pursued.

The calculated radial temperature profile through the sample and insulation wall at several times during the heating process is shown in Fig. 3. It is clear that in the initial stages of the experiment, the sample is being heated by radiant energy from the casket. These curves also reveal the uniformity of the temperature distribution in the sample, and are consistent with the observed result that these samples usually sinter without cracking using this arrangement.

Microwave power absorption by the sample and insulation is shown in Fig. 4, which shows the average power density in the sample and insulation as a function of time during the heating cycle. Clearly the absorbed power density in the sample greatly exceeds that in

Figure 3: Calculated radial temperature profiles through the midplane of the sample and insulation at various times during the simulation run shown in Fig. 2

the insulation from about 10 minutes onward. The *total* power absorbed is greater in the insulation because of its much greater mass.

CONCLUSIONS

It is clear from the above results that good qualitative information about temperature distributions and power absorption in microwave sintering of ceramic samples can be obtained through a simplified model of the microwave field distribution coupled to a detailed thermal model. These results indicate that alumina samples sintered in low-density zirconia insulation do heat by direct absorption of microwave power, and that temperatures remain uniform during the heating cycle. However, definitive quantitative results must await a coupled solution of the electromagnetic field and heat transfer equations, now in progress.

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