

SINTERING WEAR PARTS WITH MICROWAVE HEATING

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ABSTRACT

Zirconia and alumina samples were sintered to over 99.5% of theoretical density in less than 1 hour using a 1.0 kW modified kitchen microwave. The density and Vickers hardness were comparable to dense conventionally sintered specimens with firing cycles greater than 12 hours. The impact of susceptor position and the relationship between the applied power, the heating rate, temperature and properties of the specimens were investigated.

INTRODUCTION

Grain growth during sintering is an inherent problem in the sintering and densification of nanoceramics [1]. More stringent requirements for high tolerance and decreased surface roughness have made the need for fast firing methods crucial. For structural applications, fine grain sizes on the order of microns, and ideally nanometers, are desirable to achieve higher hardness and strength [2].

Microwave sintering of ceramics can offer certain advantages over conventional firing methods, including fast firing times and improved properties [3]. Slow heating rates and long dwell times during a sintering cycle promote grain growth. Heat generated by the interaction of microwaves with the ceramic material results in volumetric heating of the ceramic, thus very high heating rates are possible. It is therefore possible to sinter in a fraction of the time it takes for conventional firing. With short microwave sintering runs, the time for grain

growth is diminished. The resulting smaller grains make the microstructure more uniform and increase the strength.

Common concerns with microwave sintering include questions about whether full density can be reached with microwave heating, and how the properties of microwave-sintered ceramics compare to those of conventionally sintered ceramics. The uniformity and reproducibility of microwave heating are addressed in a companion paper in these proceedings [4].

In this work, microwave sintering was applied to zirconia and alumina to be used for wear-resistant components. These materials have low dielectric loss, reasonably high firing temperature and a wide range of applications in industry. This makes them ideal materials for the evaluation of a new, low cost commercially available microwave furnace that has been developed by modifying a kitchen microwave oven by the addition of a temperature controller and a water cooling system (ThermWAVE, Microwave Research Systems, Alfred, NY).

BACKGROUND

In addition to a microwave furnace, the critical components for microwave sintering include an insulation box and susceptors. The insulation box consists of a small chamber fabricated from low-density rigid insulation board. Low density and very low dielectric loss are required for the box to make it microwave transparent. Microwaves pass through the material with little interaction, allowing the contents to heat. The box, in essence, acts as an oven within the microwave chamber or applicator, as it allows microwaves to pass through but contains the heat generated by the contents.

Many ceramic materials do not absorb microwaves (2.45 GHz) well at room temperature [3]. Susceptors are useful for initial heating of these ceramics. Susceptors are made of a material that absorbs microwaves at room temperature and act as heating elements, which “boost” the temperature until the dielectric loss in the ceramic is high enough that the ceramic couples directly with the field. For example, using silicon carbide susceptors, zirconia will heat primarily by radiation from the SiC, until it reaches approximately 600°C, whereby the zirconia couples preferentially and heats volumetrically.

PROCEDURE

Specimens were prepared from submicron zirconia powder containing 3 mol% yttria (TZ-3Y, Tosoh, Boundbrook, NJ) or alumina (A16-SG, Alcoa Industrial Chemicals, Pittsburgh, PA) by first compacting 5 g pellets in a 2.54 cm diameter hardened steel die to 50 MPa and subsequently cold isostatically pressing to between 115 and 180 MPa.

The microwave furnace used for this study was a 1.0 kW modified kitchen microwave (ThermWAVE Model II, Microwave Research Systems, Alfred, NY),

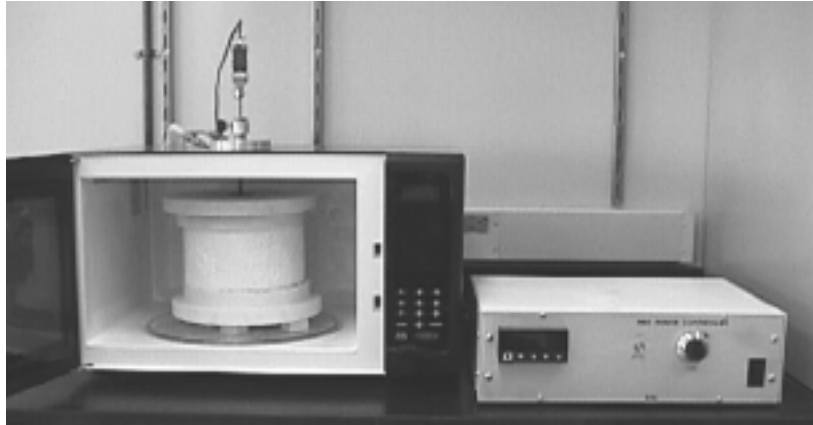


Figure 1. Microwave furnace (MRS ThermWAVE Model II) with thermal pod, thermocouple, and controller.

and is shown in Figure 1. The modifications include, a water-cooled coil system, a thermocouple (type S) and a power controller (with both manual and automatic options).

To fire the specimens, two specimens were stacked one on top of the other and centered on the floor of an insulation box fabricated from low-density rigid alumina insulation board (Eco25B, Zircar, Florida, NY). Two SiC susceptors (Microwave Research Systems) were placed at the far ends of the box so that one was on either side of the specimens (see Figure 2). The top was placed on the box and the box was positioned inside the microwave furnace on setters made of the same insulation board in order to reduce the amount of heat absorbed by the turntable.



Figure 2. SiC susceptor placement inside insulation box (top view).

Once the box was positioned in the microwave, a power level was selected from the controller and the timer on the microwave set. During the run, the power was manually stepped and the temperature measured every minute. When the run was complete, the microwave door was opened and it was possible to remove the box with insulated gloves. After approximately 40 minutes of cooling, the specimens were removed.

Conventionally sintered specimens were fired in an electric furnace (Rapid Temperature Furnace, CM Inc., Bloomfield, NJ) ramping at 5°C/minute to 1500°C for zirconia and 1600°C for alumina, holding for 1 hour and then cooling at 20°C/min.

Dimensional density was calculated from the diameter, thickness and mass. Specimens having a dimensional density of greater than 90% of theoretical (using 6.08 g/cm³ for zirconia and 3.98 g/cm³ for alumina) were additionally measured using the Archimedes method. Specimens having high density were polished and Vickers hardness and toughness values were measured. Then the polished specimens were thermally etched at temperatures of 75°C below the sintering temperature for 18 minutes to highlight the grain boundaries and examined using a scanning electron microscope.

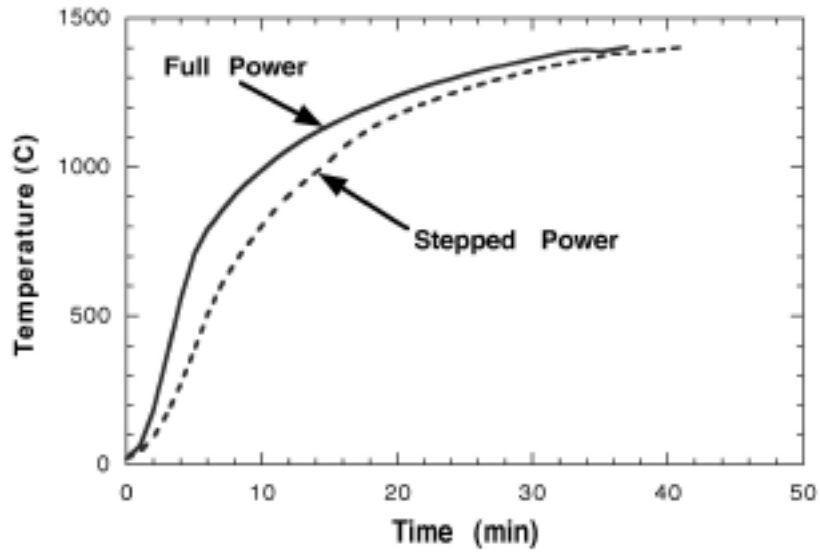
RESULTS AND DISCUSSION

Temperature and Heating Rate

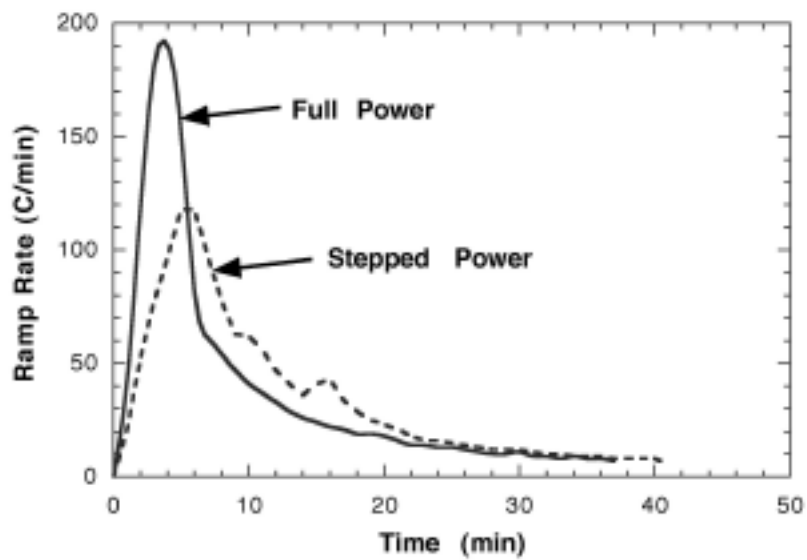
Experiments were carried out in order to compare the effect of firing at full power and a stepped power profile for zirconia and alumina samples. The actual power in the microwave chamber was approximately 0.65 kW at full nominal power of 1.0 kW. Figure 3 shows the power and heating rate as a function of time for the zirconia samples. The curves are taken from an average of at least three runs. It was found that the starting temperature of the box and susceptors had an effect on the time at which the maximum heating rate occurred, so all experiments were started at room temperature.

It can be seen in Figure 3 that the stepped power initially produced a lower temperature due to a lower heating rate than the full power. However, after 40 minutes, both the temperature and heating rates were identical. Samples from the full power vs. stepped runs had comparable density. The total energy consumption would be lower for the stepped power run. This suggests that the initial high heating rate does not offer an advantage in time or energy savings unless the system has sufficient power to maintain the high rate at high temperatures.

The ramp rates from Figure 3 are shown as a function of temperature in Figure 4. The full power runs achieved a heating rate of 190°C/min with no signs of thermal shock, indicating that the samples were heated volumetrically, rather than by radiation from the susceptors. It was found that the initial radiant heating



(a)



(b)

Figure 3. Temperature (a) and ramp rate (b) as a function microwave sintering time in the ThermWAVE (II) with zirconia samples comparing full power with stepped power.

should be fairly uniform to prevent thermal shock at high heating rates. In this test set-up, the susceptors were placed on either side of the samples. When susceptors were placed on only one side of the samples, the samples cracked at high heating rates. This is probably caused by the low thermal conductivity of zirconia and the initial “boost” of heating from the susceptors, producing uneven coupling within the sample. Stepped runs were less likely to cause thermal shock with the uneven susceptors position.

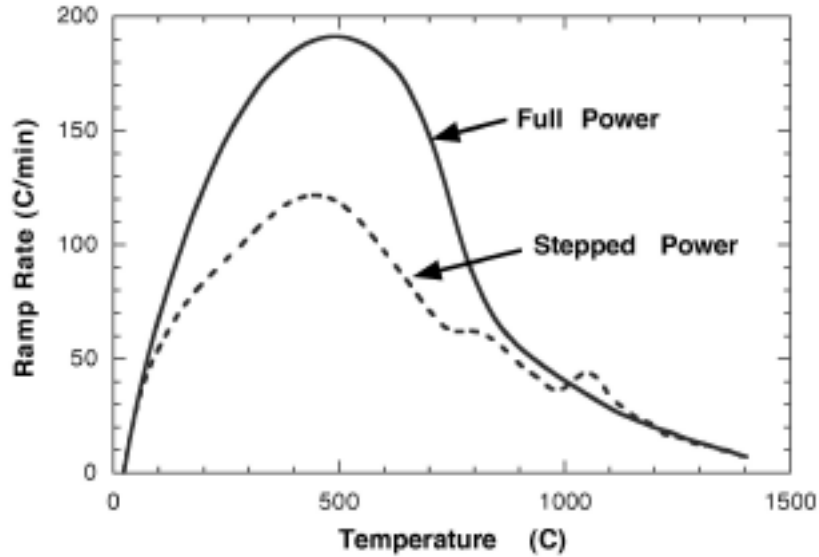
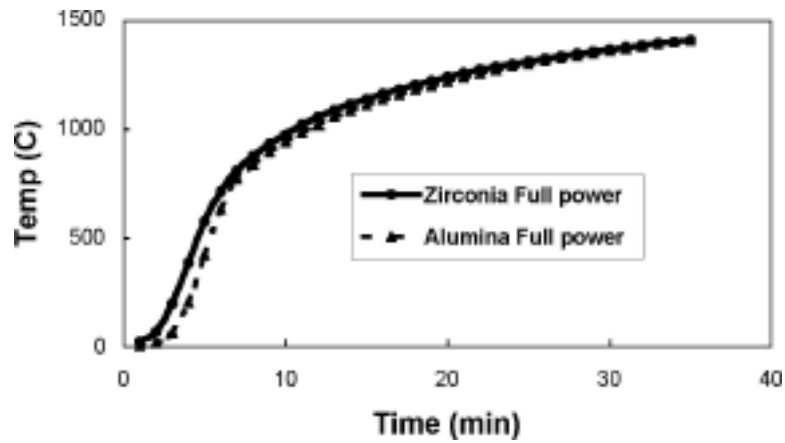


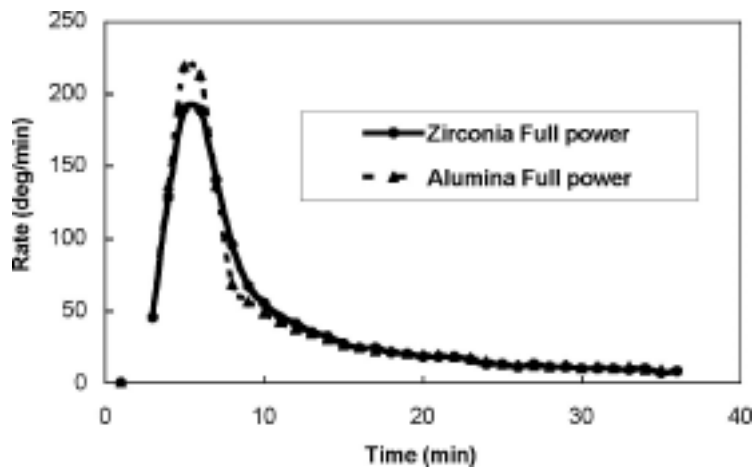
Figure 4. Comparison of full power and stepped power heating rates as a function of temperature (from Figure 3).

In Figure 5, microwave runs at full power are compared for zirconia and alumina. It can be seen that the heating rate and final temperatures achieved were similar, even though the materials have different dielectric and thermal properties. At elevated temperatures the differences may become less significant. When considering the microwave sintering profiles and power requirements, a data base may be developed that focuses on direct evidence of microwave heating effects as well as the dielectric and thermal material properties.

Samples sintered to full density were polished and thermally etched to reveal the microstructures in Figure 6 for zirconia, and Figure 7 for alumina. The microstructures of microwave sintered materials are compared with those conventionally sintered.



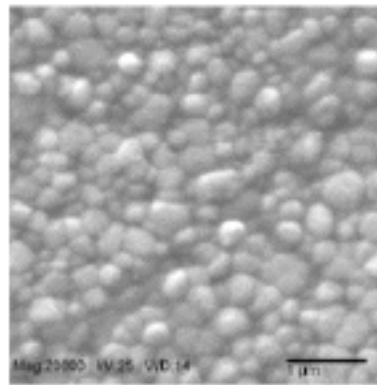
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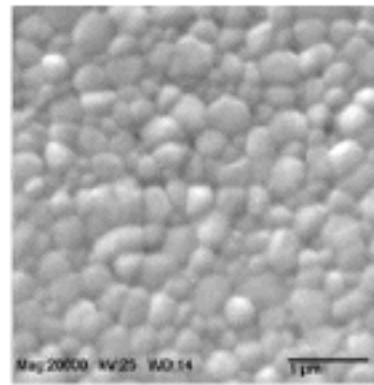
(b)

Figure 5. Comparison of microwave heating at full power for zirconia and alumina; (a) temperature and (b) heating rate versus time.

In Figure 6, the microwave sintered zirconia appears to be slightly finer than the conventionally sintered. However, this is probably within the range of variation, and not a significant difference [4]. The alumina from microwave sintering also appears to have finer grains. Other authors have reported finer grains as a result of microwave sintering [3]. However, it is difficult to make direct comparisons, due to the uncertainties of the temperature. Further study is required to understand the complex mechanisms of sintering in a microwave field.

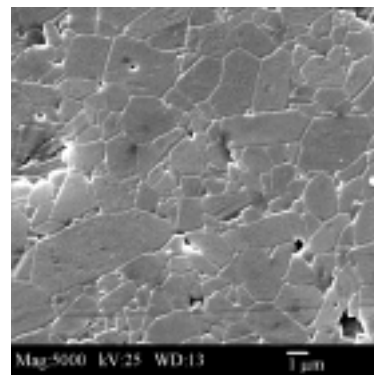


(a)

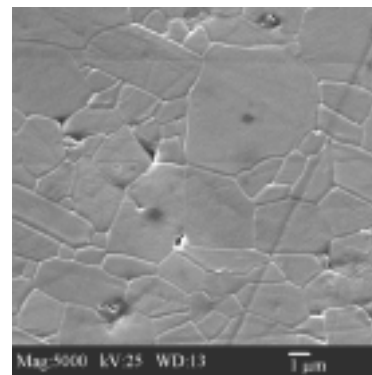


(b)

Figure 6. Microstructure of (a) microwave sintered zirconia and (b) conventionally sintered zirconia.



(a)



(b)

Figure 7. Microstructure of (a) microwave sintered alumina and (b) conventionally sintered alumina.

It is, none the less, an important result that fine-grained uniform microstructures can be produced by microwave sintering.

Table I contains a summary of results comparing microwave sintered with conventionally sintered alumina and zirconia. Materials for wear parts must have a high density, typically greater than 99.5% of theoretical. One of the important objectives for microwave sintering of wear parts, is the attainment of high density with fine grain size. Much of the literature reports less than 99% of theoretical density by microwave sintering. The hardness data in Table I supports the previous observations that microwave sintered zirconia and alumina ceramics achieved high density and maintained fine grain size.

Table I. Summary of Properties of Microwave and Conventionally Sintered Alumina and Zirconia.

| | Density (% theoretical) | Hardness (VHN 20kg) |
|---------------------|----------------------------|------------------------|
| Alumina | | |
| Microwave 1460°C | 99.6 | 15.9 (0.3) |
| Microwave 1500°C | 99.8 | 15.6 (0.4) |
| Microwave 1600°C | 99.9 | 16.0 (0.3) |
| Conventional 1600°C | 99.9 | 15.6 (0.6) |
| Zirconia | | |
| Microwave 1460°C | 99.7 | 13.5 (0.1) |
| Conventional 1500°C | 99.9 | 13.5 (0.3) |

CONCLUSIONS

It was a concern at the beginning of this study that there was a fundamental problem with achieving full density, and maintaining fine grain microstructures with microwave sintering. This has proved not to be the case. The high density and fine grain size of the microwave sintered alumina and zirconia samples makes them at least as hard as conventionally sintered materials. Microwave sintering is an energy and time efficient method for firing advanced ceramics for wear parts.

ACKNOWLEDGEMENTS

This work was funded in part by the New York State Office of Science, Technology and Academic Research through the New York State Center for Advanced Ceramic Technology at Alfred University project 2021-7738, and by the New York State Energy Research and Development Authority, contract number 6495.

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