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Initial Results of a High-Power Microwave Sintering Experiment at ORNL*

H. D. Kimrey, T. L. White, T. S. Bigelow

Fusion Energy Division

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and P. F. Becher
Metals and Ceramics Division

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OAK RIDGE NATIONAL LABORATORY
Oak Ridge, Tennessee 37831

Experiments have recently begun at Oak Ridge National Laboratory to develop microwave sintering techniques suitable for large ceramic parts. Microwave sintering offers the advantages of faster heating rates, more uniform heating, and greater energy efficiency than conventional sintering techniques. We are using 28-GHz, 200-kW cw gyrotrons as the heating source. An untuned cavity is used as the applicator to eliminate geometry sensitivity in coupling efficiency.

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Microwave sintering of ceramics (R1,2) offers several distinct advantages over sintering using conventional furnaces. First, ceramic heating rates are substantially faster than in conventional sintering. This is because the ceramic is heated directly by microwave power according to its loss tangent (R3) and the amount of microwave power incident on the ceramic. As a result of rapid heating, the extent of nonisothermal processes such as segregation of impurities to the grain boundaries (R4) is minimized. Since the sintering time can be lowered as well, the possibility for secondary recrystallization (exaggerated grain growth) may be reduced. By minimizing both impurity segregation and exaggerated grain growth, the resulting mechanical properties of the ceramic are strengthened.

Second, because most ceramic materials have a low loss tangent in the microwave frequency range, fairly uniform microwave heating can be achieved even for large and irregularly-shaped samples because the skin depth is much greater than the sample dimensions. Conventional sintering techniques heat the surface of the ceramic from external heat sources and thus produce nonuniform heating until the entire furnace and sample come to an equilibrium temperature. Uniform internal microwave heating should result in a more homogeneous sintered product.

Finally, in conventional thermal sintering, a substantial quantity of heat must be used to bring the specimen and its enclosure (e.g., furnace walls or linings) to the desired processing temperature. In addition, heat will generally be lost through the enclosure to the surroundings. However, in microwave sintering, these problems are largely avoided since the microwave cavity or "oven" is made of high electrical conductivity metals so that the majority of the power is absorbed by the ceramic sample. This results in a more efficient use of power to heat the sample.

Until now, microwave sintering experiments have been performed with small microwave applicators at 2.45 GHz with a few kilowatts of available cw power. We now propose to use the newly developed high-power gyrotron oscillators that produce

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hundreds of kilowatts at 28 GHz (R5). The loss tangents of most ceramic materials increase with frequency so that even more enhanced power absorption should occur. Since the total power available is also much greater than prior experiments, we expect that both these factors will combine to greatly reduce the time required to sinter the ceramic. This will improve the mechanical strength of the ceramic over earlier microwave experiments.

In earlier experiments, small ceramic samples had to be specially shaped to fit into small single-mode cavities or applicators. Our technique using a large cavity whose dimensions are much, much greater than a free space wavelength will allow us to heat large, irregular samples uniformly. The power density in the cavity is made uniform by allowing the microwave energy to reflect off the cavity walls many times, thus exciting a very large number of cavity modes. Cavities of this type are referred to as untuned (R6).

To demonstrate proof of principle, we are sharing the Radio-Frequency Test Facility (R7) 28-GHz, 200-kW, cw gyrotron. Most of the necessary equipment obtained from prior experiments has been used to assemble a microwave furnace for heating the ceramic samples. We are using an available 76-cm-diam by 100-cm-long cavity with O-ring seals as the microwave cavity for sintering the ceramic. This allows flexibility to sinter the ceramic samples in either a high vacuum or with various gas fills. The heating process consists of a low-temperature bakeout phase followed by rapid heating to a 1600° C sintering phase. Special high-temperature thermocouples monitor the ceramic temperature in several locations. Also, a residual gas analyzer monitors gas evolution during vacuum operation of the cavity. The power delivered to the sample is estimated by using system calorimetry and using low power cavity Q measurements.

Because ceramic loss tangents increase with temperature, the samples tend to "run away" thermally at constant power. To correct for this effect, we typically roll back the duty cycle on our modulated output power and thereby stabilize the temperature. The sample holder must withstand the elevated temperatures of the sample and not produce impurities that might contaminate the sample. Tantalum is a prime candidate because of its high operating temperature in vacuum and because it does not react strongly with high-purity alumina samples at elevated temperatures (R8). Also, it has low microwave losses compared to graphite or carbon crucibles. Arcing in the microwave furnace could be a problem if sharp corners or points on metal surfaces are present. This problem has been eliminated by carefully designing the sample holder and by providing a high-quality vacuum in the furnace.

Experiments to date have been limited in size to Al_2O_3 samples pressed to 50% of theoretical density and several hundred cubic centimeters in volume. Preliminary analysis suggests that >98% of theoretical density has been achieved over much of the sample volume. However, all samples to date show cracking indicative of excessive thermal stresses. Typically, crack sites show contamination from small bits of graphite embedded in the sample.

Our future plans are to continue to vary as many operating parameters as possible to achieve uniform density, crack-free samples, once a successful technique is found to scale up the sample volume to at least 1L.

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