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MICROWAVE VERSUS CONVENTIONAL SINTERING OF SILICON CARBIDE TILES

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ABSTRACT

Silicon carbide eutectic green body tiles were sintered at 2100°C in argon for one hour using conventional (resistance heating) and microwave techniques. The sample sintered using microwave energy achieved a final density 91.5 percent of theoretical, whereas, the conventionally-treated sample sintered to 84.4 percent of theoretical. In addition, the Poisson's ratio and acoustic moduli (Young's, shear, and bulk) were considerably higher for the microwave-sintered specimen.

1. INTRODUCTION

Silicon carbide is being evaluated as an armor material because of its lightweight, high-hardness, and excellent armor efficiency. However, one of the problems associated with silicon carbide is the high cost associated with achieving fully dense tiles. Full density requires either hot pressing and sintering or reaction bonding. Past efforts have shown that hot pressed tiles have a higher armor efficiency than those produced by reaction bonded sintering. An earlier study showed that the acoustic properties of fully-dense silicon carbide tiles were enhanced through the use of post-sintered microwave heat treatments. One of the least expensive forming techniques is to isostatically press-and-sinter. In this study, we have used microwave energy to densify silicon carbide green bodies. Microwave sintering has been demonstrated to be a very quick way to sinter ceramics such as alumina to exceptionally high densities. Previous work has shown that microwave post treatment of fully-dense reaction bonded silicon carbide tiles significantly improves the acoustic properties of the tiles. These properties include Poisson's ratio, Young's modulus, shear modulus, and bulk modulus.

2. EXPERIMENTAL PROCEDURE

Silicon carbide green bodies were provided by Cercom, Inc., for processing at Oak Ridge National Laboratory (ORNL). Yttria and aluminum were added as sintering aids. The green bodies were three inches by three inches by one-inch thick. Prior to sintering, the tile samples were baked at 400°C in a conventional furnace to remove organic binders. One sample was heated in a conventional resistance-heated, carbon-lined furnace to 2150°C in argon gas for one hour. This sample was loaded inside a carbon crucible and sintered in a vertical position to reduce thermal stresses and, therefore, cracking. The second sample was loaded horizontally inside a boron nitride crucible packed with a mixture of boron nitride and glassy carbon. This was then placed inside a 12-inch alumina fiberboard box for additional insulation. The boron nitride and glassy carbon are necessary to reduce thermal stresses in the silicon nitride part caused by uneven microwave heating. This sample was heated in a 12 kW microwave furnace at 2100°C under argon cover gas for one hour. The heating rate for both specimens was 10°C/minute. Both samples were furnace cooled to room temperature.

Physical properties were measured on the tiles using ultrasonic velocity measurements. A total of five readings were taken from the samples at the four corners and at the center as shown in Fig. 1. From the velocity measurements, Poisson's ratio, Young's modulus, shear modulus, and bulk modulus were calculated. Each tile was also scanned ultrasonically to map density uniformity as well as irregularities and flaws, such as cracks, which may be present.

Fig. 1. Location of velocity measurements

3. RESULTS AND DISCUSSION

Visual comparison of the tiles after sintering showed that the conventionally-sintered tile was black while the microwave-sintered tile was gray in coloration. The surface of the conventionally-sintered tile was coarser than the microwave-sintered specimen. This coarseness is attributed to the formation of large crystallites on the specimen surface, which is an indication that some vaporization had occurred. The microwave-sintered specimen had a smoother surface but contained a 1.5 cm crack which ran perpendicular to one side, which can be seen in the ultrasonic scan (Fig. 2). This crack was likely caused by uneven heating of the sample by the microwave field.

The sintered results are shown in Table 1. For each specimen the post sintered mass was less than the starting (post bake-out) mass, which indicates that volatilization of the silicon carbide had taken place. The conventionally and microwave-sintered specimens lost 28 and 22 grams of mass, respectively. This indicates that excessive volatilization occurred during both sintering processes. The higher mass-loss for the conventionally-sintered tile was likely due to the higher sintering temperature (2150°C). As shown in the table, the sample undergoing microwave sintering densified to a significantly higher density than the conventionally-sintered sample even though the sintering temperatures and heating rates were essentially the same. These data suggest that microwave sintering techniques may be better than conventional sintering techniques for densifying silicon carbide. However, the uneven heating caused by the microwave field may create internal stresses in silicon carbide bodies that may lead to cracking. This is a very promising result for a beginning sintering attempt. Similar cracks in other materials have

Silicon Carbide 2.5X2.5 plates, IT 5 MHz, 2" Focus, 12-17-98

Microwave Sintered

Oven Sintered

Crack ->

Chip

Figure 2

been eliminated through process refinements. Efforts to improve the uniformity of the microwave field would likely decrease the probability of cracking while maintaining high densification.

Table 1. Results of silicon carbide sintered using conventional and microwave techniques

Sample Sintering Process	Green mass (g)	Bake-out mass (g)	Sintered Mass (g)	Sintered Volume (cm ³)	Density (% of theoretical)
Microwave	258.36	246.94	224.81	76.40	91.5
Conventional	255.79	244.48	216.56	80.19	84.4

As shown in Table 2, the calculated acoustic properties are much higher for the sample that was sintered using microwave energy than for the sample that was conventionally sintered. Poisson's ratios for both specimens were found to be much higher than normal for silicon carbide. The typical value of Poisson's ratio for sintered silicon carbide is 0.14. The higher values obtained from the samples are likely due to the addition of Ytria and aluminum as sintering aids. These sintering aids were necessary when using pressureless sintering techniques such as those used in this study. The increase in Poisson's ratio was 16 percent higher for the microwave-sintered specimen, and the acoustic moduli were also much higher. The values obtained for the Young's, shear, and bulk moduli for the microwave-treated sample compare well with literature values of silicon carbide formed by both sintering and reaction bonding methods, but the values obtained for the conventionally-sintered sample were considerably lower. The Young's and shear moduli were 50 percent higher for the microwave-sintered sample, and the bulk modulus was over 100 percent higher. In addition, the standard deviations for the microwave-processed sample were much lower than the conventionally-processed specimen indicating better uniformity. The higher acoustic properties for the microwave-sintered sample likely resulted from the much higher density of the post sintered tile. For a full treatment of the acoustic properties see the Appendix.

Table 2. Acoustic property measurements

Ultrasonic Property Measured	Conventionally Sintered Sample	Microwave-Sintered Sample
Poisson's Ratio	0.289 ± 0.016	0.336 ± 0.008
Young's Modulus, GPa	234.075 ± 7.048	369.020 ± 2.717
Shear Modulus, GPa	90.823 ± 2.460	138.156 ± 1.275
Bulk Modulus, GPa	185.682 ± 18.555	374.623 ± 17.349

4. CONCLUSIONS

Microwave sintering of silicon carbide containing sintering aids produces a denser and more uniform solid than silicon carbide, which is densified using the conventional press-and-sintering method. Volatilization of the part was also reduced by microwave heating. Physical properties such as acoustic moduli and Poisson's ratios were substantially higher for silicon carbide sintered using microwave radiation.

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APPENDIX

Ultrasonic Material Analysis Report (UMAT Version 4.0.1) 12/17/96

Part Calibrated with Standard Report

Standard File Name: d:\cviapps\umat4\43s1217.std

Standard Description:

Test Config: V3330 Steel Blocks - Through transmission with reference pulse

Weighting: Instrumental

Transducers:

	Mfg	Model	Serial #	Dia	Center Freq(MHz)
Long	Panametrics	V110/M110	46512/148411	0.25	5.0
Shear	Panametrics	V156/V156	46976/48971	0.25	5.0

Part File Names: d:\cviapps\umat4\sida.raw

d:\cviapps\umat4\sldb.raw

Part Description: Oven Sintered

Microwave Sintered

Test Config: Trough transmission with reference pulse

Avg and Std Deviation of 5 readings on sample

	Thickness
Oven Sintered	0.82068
Std Deviation	0.00292

Velocities:

Longitudinal:	0.4234 in/us +/-	0.0135	10.755 mm/us +/-	0.344	3.20%
Shear 0 Deg:	0.2310 in/us +/-	0.0040	5.867 mm/us +/-	0.102	1.75%
Shear 90 Deg:	0.2300 in/us +/-	0.0024	5.841 mm/us +/-	0.060	1.03%
Shear Avg:	0.2305 in/us +/-	0.0031	5.854 mm/us +/-	0.079	1.35%
Avg Shear Anisotropy:	0.43%				
Extensional:	0.3700 in/us +/-	0.0056	9.398 mm/us +/-	0.142	1.51%
Rayleigh:	0.2134 in/us +/-	0.0030	5.421 mm/us +/-	0.075	1.39%

Calculations:

Poisson's Ratio:	0.289 +/-	0.016			
Young's Modulus:	33.950 Mpsi +/-	1.022	234.075 Gpa +/-	7.048	3.01%
Shear Modulus:	13.173 Mpsi +/-	0.357	90.823 Gpa +/-	2.460	2.71%
Bulk Modulus:	26.931 Mpsi +/-	2.691	185.682 Gpa +/-	18.555	9.99%

Avg and Std Deviation of 5 readings on sample

	Thickness
Microwave Sintered	0.78722
Std Deviation	0.00139

Velocities:

Longitudinal:	0.5373 in/us +/-	0.0080	13.647 mm/us +/-	0.203	1.49%
Shear 0 Deg:	0.2678 in/us +/-	0.0007	6.801 mm/us +/-	0.017	0.25%
Shear 90 Deg:	0.2666 in/us +/-	0.0029	6.772 mm/us +/-	0.072	1.07%
Shear Avg:	0.2672 in/us +/-	0.0012	6.786 mm/us +/-	0.031	0.46%
Avg Shear Anisotropy:	0.43%				
Extensional:	0.4366 in/us +/-	0.0016	11.091 mm/us +/-	0.041	0.37%
Rayleigh:	0.2492 in/us +/-	0.0010	6.330 mm/us +/-	0.026	0.40%

Calculations:

Poisson's Ratio:	0.336 +/-	0.008			
Young's Modulus:	53.522 Mpsi +/-	0.394	369.020 Gpa +/-	2.717	0.74%
Shear Modulus:	20.038 Mpsi +/-	0.185	138.156 Gpa +/-	1.275	0.92%
Bulk Modulus:	54.335 Mpsi +/-	2.516	374.623 Gpa +/-	17.349	4.63%