Characterization of hardness, residual stresses, and porosity of boron carbide/silicon carbide laminates

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Introduction

Ceramics are inorganic and nonmetallic materials that are characteristically brittle. Natural ceramics, such as clay, are hard but brittle and do not conduct electricity, limiting their potential usefulness in many fields. Artificial ceramics, however, are able to be modified in various ways that permit variations from these traditional properties or can enhance the effectiveness of those traditional properties (Mason, 1998). Recently, a method of production for artificial ceramics has been devised that is similar to the process of forming natural ceramics. Using the example of clay again, specific minerals are mixed together to form a viscous solid mass with water. Ceramic bodies can be artificially produced from ceramic particles mixed together with organic polymers under heat to form a similarly viscous mass. The powders that will be used in this project are Silicon Carbide & Boron Carbide (SiC & B_4C). The polymer will be Styrene Ethylene Butylene Styrene (SEBS). Since the SEBS polymer is not useful on its own, it is dissolved in mineral oil to form a gel. This gel is then processed to leave only the ceramic as a solid mass which is then compressed to form a dense ceramic body. To make laminates, various ceramic layers are stacked before compression (Figure 1). This method for producing laminate bodies is promising as it offers a way to get around the rigidity characteristic of ceramics both natural and artificial, but has not been adequately tested. The goal of this project is to test the viability of this method by measuring the properties of the produced laminates. The chosen properties are hardness, residual stresses, porosity, and the length of cracks from hardness testing. It is expected that hardness will go up with more B_4C and both porosity and residual stresses decrease as the material is more homogeneous.

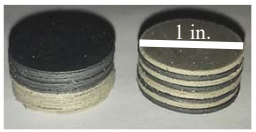


Figure 1 (left): An example of two layered ceramic laminates. The lighter layers are SiC and the darker layers are B_4C.

Materials and Methods

A SEBS based polymer gel was formed from 20 weight percent SEBS powder and 80 weight percent mineral oil which was mixed and was placed into an oven at 120°C in order homogenize the mixture and form a gel. Every 30 minutes, the SEBS/mineral oil mixture was removed from the oven and stirred vigorously to improve mixedness and facilitate the homogeneity process. The mix was stirred three times before being removed from the oven and allowed to cool to room temperature to be stored as a solid.

Materials and Methods (cont.)

When adding the ceramic powder, the gel was then heated to 120°C in order to melt it. Ceramic powder was then added to the gel up to 47 volume percent ceramic. Adding the ceramic powder increases the viscosity of the melt making mixing more difficult. Each time powder was added, it was mixed with the gel for about one minute, and then placed back into the oven to reheat the mixture to 120°C. For the different materials, B_4C and SiC, 34.46 g of gel were mixed with 88.83 g and 113.15 g respectively, with half of each powder mass with 34.46 g of gel for the 50/50 mix. After heating for 2 hours to finish the mix, the coarse mix was allowed to cool before being sent to a ThermoFisher twin-screw extruder to be more finely mixed. With the screws rotating at 50 rpm, the material was twice sent through the heated sections of the extruder to produce a homogeneous fine mix.

The fine mixes were then sent to a melt press where it was pressed into 1-inch thick circular tapes at 20 atm over 2 minutes before a 2-minute cooling period. Thirty 1-inch discs were cut out from the tape. The discs were then placed in isopropanol to remove the mineral oil. After at least 2 hours, the alcohol was changed out. After three exchanges, the discs were placed in methanol for 1 hour before being removed and dried in a vacuum at 40°C.

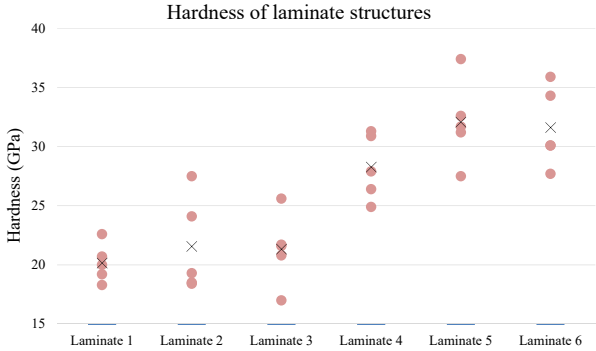
Once the discs were removed from the vacuum oven, they were then sent to have their SEBS polymer removed in a furnace at 600°C under flowing Argon for 20 hours. The furnace reached 600°C at 2°C/min and cooled to room temperature at 5°C/min. The discs were then layered in the desired laminate body and placed into a graphite die before going into a hot press. The hot press was first heated to 1000°C at 50°C/min under 200 lbf of load and vacuum before switching to flowing argon over 10 minutes. After the gas switch and the 10-minute hold the discs were pressed at 2400 lbf as the temperature was raised to 1950°C at the same rate. These conditions continued for 2 hours, after which the load was removed, and the cooling commenced at 50°C/min. After reaching room temperature, the discs were removed and grit blasted to remove the residue from the graphoils used to protect the die. The mass and thickness of the blasted specimens were measured, as was their density on an Archimedes balance.

Results

Chart 1 (below): The average measurements for each of the six testing laminate structures.

Laminate	Average Hardness (GPa)	Average Crack Length (μm)	Average Residual Stresses (MPa)	Porosity (%)
B_4C - B_4C - B_4C	32	35.02	0.00	10.1
SiC - SiC - SiC	19	34.34	0.00	11.6
B_4C - SiC - B_4C	28	27.67	45.5	10.4
SiC - B_4C - SiC	21	25.45	46.5	11.2
B_4C - B_4C/SiC - B_4C	31	27.98	43.0	10.3
SiC - B_4C/SiC - SiC	22	26.61	43.5	11.5

Results (cont).



Graph 1 (above): The hardness values of the laminates listed in chart 1 in order of increasing B_4C content. Monolithic SiC starts the chart on the far left and monolithic B_4C on the far right. The intermediate laminates are ordered by increasing B_4C content. A black X denotes the mean of the data set.

Conclusions

Using a single-factor ANOVA, the means of each of the measurements for each laminate were compared to the other laminates. The data for hardness were found to be significantly different ($F(5,30) = 15.92$, $p = 0.000$), as was expected. This indicates that the materials likely do not interact in a way where they reduce the overall hardness of the body. The data for residual stresses were also found to be significantly different ($F(5,30) = 14.21$, $p = 0.000$) supporting the known difference in the coefficients of thermal expansion between the two materials – 3.2 * 10^{-6}/K for B_4C and 3.8 * 10^{-6}/K for SiC, neither of which was measured in this experiment. The data for porosity was also found to be significantly different ($F(5, 30) = 5.28$, $p = 0.001$), indicating differences in crystalline structuring between the two materials likely has some impact on the final ordering of the materials on a molecular level.

References

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Mason, T. O., (1998). *Advanced Ceramics*. In *Encyclopædia Britannica online*. Retrieved from https://www.britannica.com/technology/advanced-ceramics