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Strength and machining of gelcast SIC ceramics

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The aqueous gelcasting technique was employed to form silicon carbide green samples for mechanical strength measurements and machining tests. The monomers used in this work were acrylamide (AM) and methylenebisacrylamide (MBAM). Polymerisation reaction was promoted by the addition of catalyst (*N, N, N′, N′*-tetramethyl ethylenediamine) and initiator (ammonium persulfate). Characterisation of the green, dried gelcast samples included measurements of density and bend strength (flexural strength). It was found that the bend strength of dry, green, gelcast samples depends critically on the slurry solid loading, the amount of monomers and the ratio of monomers contents (AM/MBAM). The highest strength value of 29 MPa was found with samples produced from the slurry with 25 vol% solid loading, the monomers content of $\sim$10 wt% and the ratio AM/MBAM of $\sim$16. An increase in solid loading from 25 to 35 vol% caused a decrease in the green sample strength to 20 MPa. Successful machining was achieved with all samples having bend strengths of over $\sim$6 MPa using standard machining equipment. In one set of samples, machining was done with cemented carbide (W-Co) tools and in the other set with ceramic cutting tools (Al_2O_3-TiC) using the same cutting speeds, feed rate and depths of cut. Ceramic cutting tools exhibited approximately three times longer life than the cemented carbide tools. The effect of solid loading and the concentration of monomers on the sintered density were also investigated.

1. Introduction

Gelcasting of ceramics has emerged as one of the promising forming techniques with potentials of replacing the traditional ceramic forming methods such as dry pressing, slip casting, and injection molding. The two major advantages of the gelcasting method are the low content of the organic binder (3–5 wt%) and relatively high dried green body strength which allows forming of complex shapes and machining in green state [1]. Other advantages include short processing time, more homogeneous distribution of organic binders, and easier binder removal. Compared with injection molding, gelcasting avoids problems associated with slumping during binder removal and minimizes the creation of large molding defects [2]. Also, the process enables development of slurries with high solid loading and casting of thin wall structures.

Although gelcasting was originally developed as a near-net-shape forming process, green machining of gelcast components can be particularly useful for producing prototypes, for custom manufacturing or for adding features to a formed part which would be too difficult or too costly to include in the mold. In many cases, extensive machining is required in order to produce intricate shapes and surface finish normally expected with structural components [3, 4]. Previous machining experiments have shown that the steel tool wares rapidly when used for cutting green gelcast ceramics [5]. In order to reduce the wear, the cemented carbide tools were used for the machining of gelcast Al_2O_3 and Si_3N_4 ceramics [5].

Other studies have also shown that gelcast samples normally have strengths of 5 to 20 times higher than that of dry pressed samples and about two times higher than slip cast material [6]. The higher strength of the gelcast materials was attributed to the homogenous distribution of the polymer and to the high inherent strength of the polymer formed in the polymerisation process. The gelcasting technique has been successfully applied to ceramics such as alumina, silicon nitride, silicon and TiN [7].

The objective of this paper was to develop a gelcasting process for forming SiC ceramics and to examine the role of monomers with regard to the strength of the green bodies. Another objective was to develop a correlation between the level of monomers and the density of the sintered bodies. Finally, a series of machining tests were performed in order to understand the factors affecting the machining of the green gelcast bodies.

2. Experimental procedure

Aqueous gelcasting of SiC was carried out using acrylamide (AM) and methylenebisacrylamide (MBAM) monomers. The submicron size β-SiC powder produced in-house using silica and carbon was used as a raw material. High purity alumina (Alcoa A16-SG) and Yttria were used as sintering aids. The average particle size of SiC, Al_2O_3 and Y_2O_3 was 0.6, 0.5, and

1 μm, respectively. The slurries were prepared by suspending the powder in distilled water with ammonia (concentrated) as dispersant. Prior to casting, the slurry was mechanically stirred, ultrasonicated and degassed under vacuum. The concentration of solid in the slurry was varied from 25 to 40 vol%, the concentration of monomers in the premix solution from 6 to 18 wt% (4 to 12.6 wt% calculated on the amount of powder), and the ratio of monomers (AM/MBAM) from 12 to 30. The polymerisation reaction was performed by the addition of catalyst (N, N, N', N'-tetramethylethylenediamine and initiator (ammonium persulfate). Gelcasting was done by pouring of the slurry into rectangular and cylindrical metal moulds having dimensions 75 × 50 × 25 mm and diameter 58 × 65 mm, respectively.

After gelcasting, samples were washed under a tap water stream, than held at room temperature inside a sealed plastic bag for 48 hours (>95% relative humidity) and another 48 hrs in open atmosphere. Room temperature dried gelcast samples of various sizes were heated up to 500°C with the rate of 1°C/min and then held at that temperature for 60 minutes in order to remove the binder. After binder removal, the samples were heated at a rate of 10°C/min to sintering temperatures of 1820 to 1895°C and held at sintering temperature for 1 hour under static argon.

Linear shrinkage during sintering was determined on the basis of measuring the dry green (105°C) and sintered samples dimensions. The density of the green samples was determined on the cut and fine ground rectangular bars by measuring the dimensions and weight. The density of the sintered samples was measured using the water displacement technique. The strength measurements of the green bodies were carried out using a standard four-point-bending fixture with inner and outer spans of 20 and 40 millimetres, respectively.

The dried green gelcast samples were machined in turning and facing operations using standard metal machining lathes with cemented carbide tools and a specially prepared ceramic cutting tool based on Al_2O_3-TiC. Boring of the green bodies was carried out using standard milling machining and cemented carbide tools.

3. Results and discussion

3.1. Strength of green samples

The variation of bend strength of the green bodies as a function of solid loading is presented in Fig. 1. The results show a gradual decrease of strength with the increase in the amount of solid loading, reducing the strength value to only 2 MPa at 40 vol% solid loading.

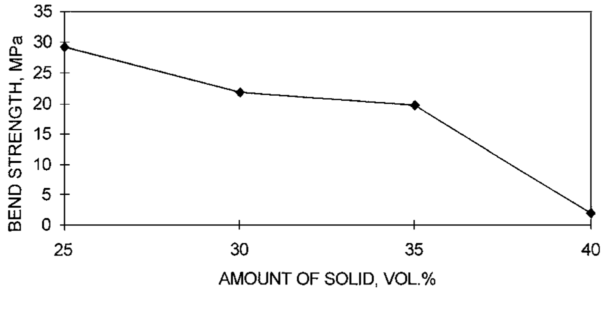

Figure 1 Dependence of the strength of green gelcast SiC on the concentration of solid in the slurry for the constant monomer concentration of 9.9 wt%, and the monomer ratio (AM/MBAM) of 16.

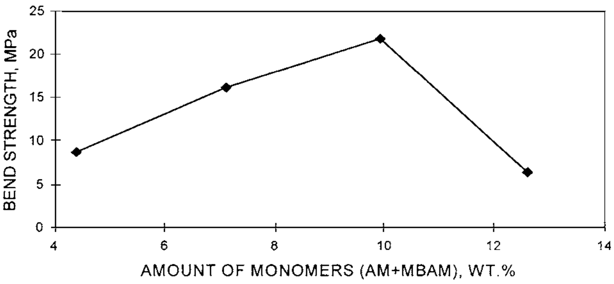

Figure 2 Variation of strength with monomers concentration in gelcasted body. The solid loading was 30 vol% and the ratio AM/MBAM was 16.

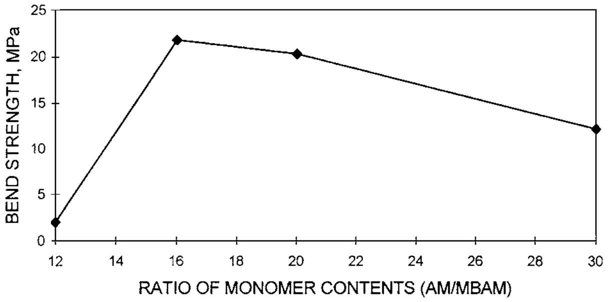

Figure 3 Change of bend strength with a monomer ratio for solid loading of 30 vol% and a monomer concentration of 9.9 wt%.

The highest strength (~30 MPa) was measured with samples containing 25 wt% of solid. The decrease in strength with solid loading appears to be related to the increase in viscosity of the slurry and consequently to a decrease in homogeneity of the slurry as observed with alumina and TiN powders [7, 8]. As the solid loading is increased, the probability of finding bigger strength controlling flaws in the green bodies is also increased. Unlike the solid loading, the addition of monomers (AM + MBAM) to the slurry first increased the flexural strength of the green body, reaching a maximum strength of 22 MPa at approximately 10 wt% monomers, and then decreased sharply to 6–7 MPa at ~12.5 wt% monomers (Fig. 2).

The monomers ratio also plays an important role in determining the strength of the green gelcast bodies (Fig. 3). As with the amount of monomers, the flexural strength of the gelcast samples first increased with the increase of the ratio of monomers reaching the maximum at approximately 16 and then decreased.

The role of monomers ratio (AM/MBAM) in strengthening green gelcast sample is not well understood at this point and further testing is required to clarify it. It is conceivable, however, that the maximum in strength at a particular monomer ratio is associated with the inherent strength of the acrylamide polymer. The strength may be controlled by the MBAM monomers whose presence creates additional collateral bonding.

3.2. Sintered density

Fig. 4 shows the change of sintered density as a function of solid loading. Inspection of Fig. 4 indicates

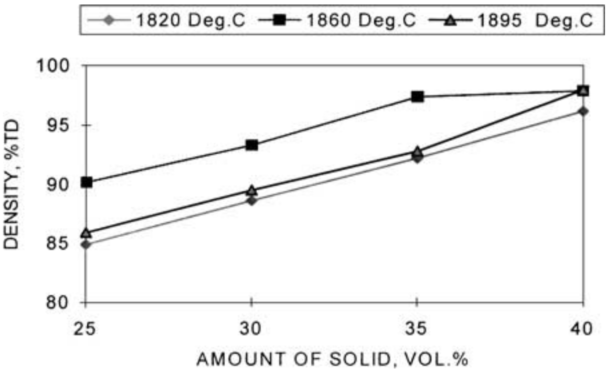

Figure 4 Dependence of sintered density on the slurry solid loading with the constant monomer concentration of 9.9 wt%, and the monomer ratio (AM/MBAM) of 16.

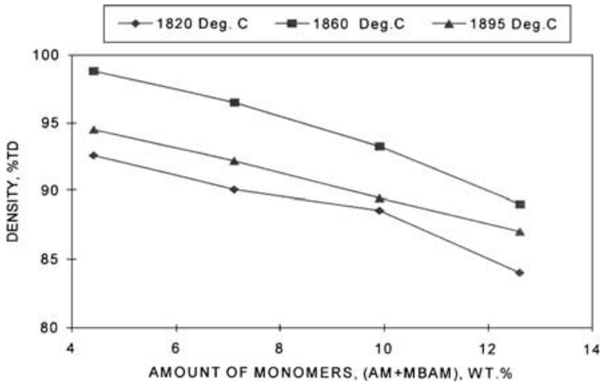

Figure 5 Sintered density vs. amount of monomers in SiC gelcast body, for the solid loading of 30 vol% and the monomers ratio of 16.

that the highest density was achieved at temperature of 1860°C for a solid loading of 40 vol%. Fig. 5 shows the change of sintered density as a function of the amount of monomers. Although an increase in the amount of monomers has a small effect on green density, it leads to a continuous decrease in sintered density. This behaviour could be associated with the larger level of residual porosity left on removal of binder from the green bodies and the inhibition of sintering.

Clearly, the smaller the amount of monomer present in the green bodies the higher is the sintered density. Sintered densities as high as 98%TD were achieved in these experiments (Fig. 5).

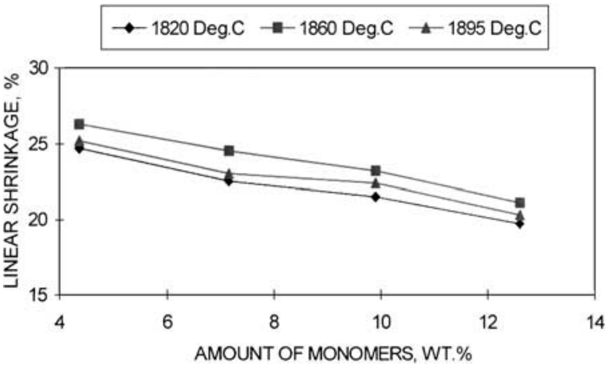

Figure 6 Linear shrinkage vs. amount of monomers. The level of solid loading was 30 vol% and the monomer ratio 16.

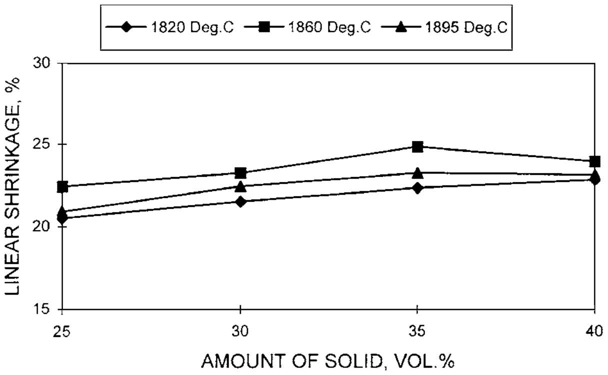

Figure 7 Dependence of linear shrinkage on the slurry solid loading. The level of monomers was 9.9 wt% and the monomers ratio was 16.

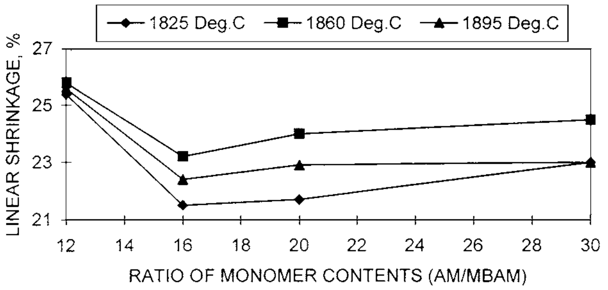

Figure 8 Variation of linear shrinkage with the ratio of monomers (AM/MBAM). The solid loading was 30 vol% and the amount of monomer was 9.9 wt%.

The role of monomers in the sintering of silicon carbide can be found from Fig. 6, which depicts the change of linear shrinkage of sintered body during sintering as a function of the amount of monomer. Comparison of the results of sintered density in Fig. 5 and those of shrinkage in Fig. 6 presented as dependent on the amount of monomers, show the same decreasing trend. Clearly, any addition of monomers to SiC powder above approximately 5 wt% has negative effect both on linear shrinkage and sintered density.

As expected, the amount of solid loading increases the linear shrinkage in almost the same fashion as it increases the sintered density (Figs 4 and 7). From the sintered density viewpoint, the best solid loading is found to be between 35 and 40 vol%.

The effect of monomers ratio on linear shrinkage is shown in Fig. 8. Clearly the highest shrinkage was obtained with monomer ratio of 12 suggesting that this ratio produces the sintered body with the high density. However, high sintered density in this case does not have practical significance, because the samples produced with monomer ratio of 12 have low green strength (Fig. 3) and did not allow successful machining.

3.3. Machining of green samples

Machining experiments were carried out on dried gelcast samples using two different speeds of rotation. In one set of experiments, the speed of 165 rpm was applied to the cylinders of up to 10 mm in diameter. This provided a maximum rotation speed of 11 surface meters per minute (sm/min).

In the second set of machining, the speed was increased to 230 rpm and was applied to turning of cylinders with diameters ranging from 30 to 58 mm.

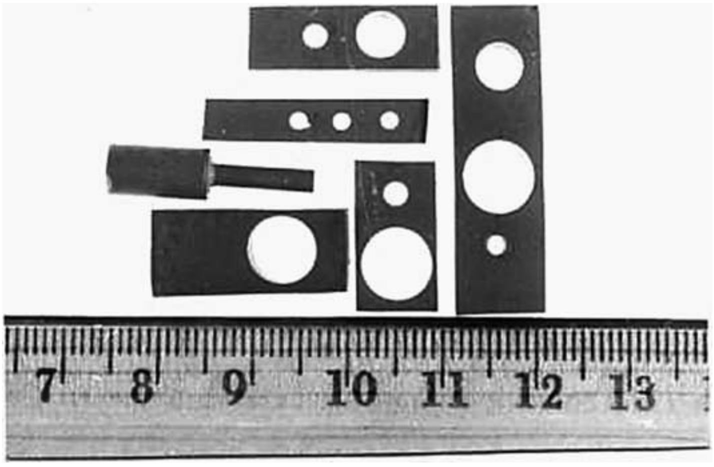

Figure 9 Examples of machined and sintered samples of different size and shape.

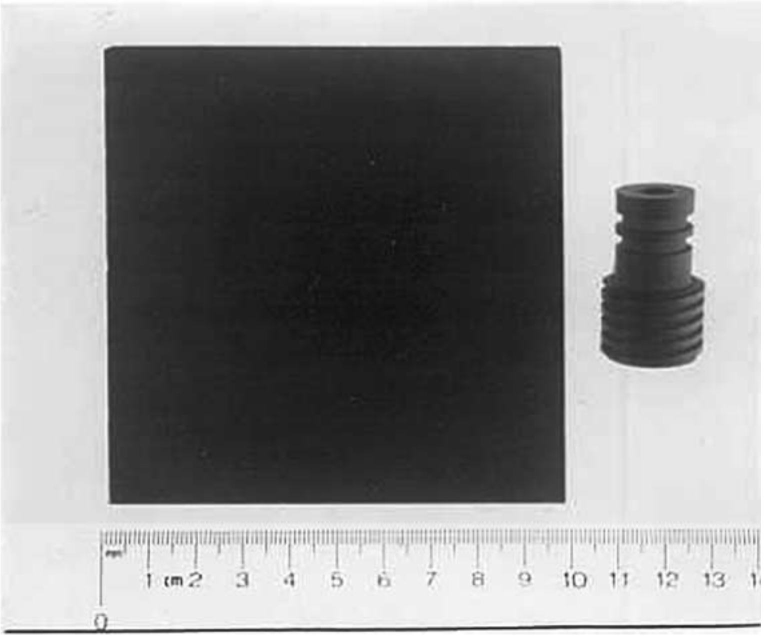

Figure 10 The tile and the nozzle produced by gelcasting, sintering and machining.

The cutting speeds in these tests were between 22 and 40 surface meters per minute, the feed rate 0.175 mm/revolution and the depth of cut 2 mm. Examples of machined green samples are shown in Fig. 9. All samples had an excellent surface finish. As can be seen in Fig. 9, the high strength of the green bodies allowed drilling holes very close to each other and to the edge of the sample. It was possible to drill holes with a wall thickness as low as 0.5 mm. The picture of the sintered tile having dimensions of 100 × 100 × 11 mm is presented at the Fig. 10, showing that a large range of sample sizes could be achieved by gelcasting. The sintered nozzle displayed in the same picture is an example how the complex pieces with grooves, threads, holes, etc. could be produced with acceptable tolerances by this technique.

A series of machining tests were carried out on gelcast samples using both tungsten carbide and ceramic tools in order to determine which one wears more. At least five gelcast samples with diameters of 50 to 58 mm were machined by turning using a standard lathe under the same conditions. These experiments have shown that the life of the tool increased from ∼5 minutes for cemented carbide tools to over 15 minutes for ceramic cutting tools. An excellent surface finish was achieved for the samples machined with ceramic cutting tools.

4. Conclusions

The green strength of SiC bodies prepared by gelcasting was shown to vary from ∼2 MPa to over 28 MPa, depending on the solid loading, amount of monomers, and monomers ratio. The highest strength was obtained with gelcast samples containing 25 vol% solid, approximately 10 vol% monomers, and a monomer ratio of 16.

The sintered density increases with slurry solid loading and decreases with the amount of monomers.

All gelcast samples, which had strengths of over 6 MPa, exhibited satisfactory machining characteristics. Due to high green strength, it was possible to drill holes close to the surface and with a wall thickness of less than 0.5 mm, achieving an excellent surface finish. The process appears to be highly suitable for manufacturing complex shaped components requiring no post sintering machining.

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