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Improved Consolidation of Silicon Carbide

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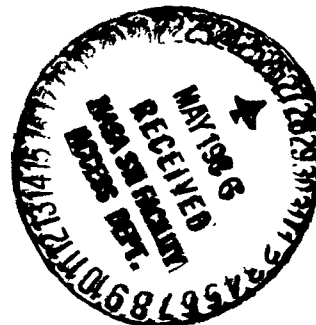
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IMPROVED CONSOLIDATION OF SILICON CARBIDE

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SUMMARY

Alpha silicon carbide powder was consolidated by both dry and wet methods. Dry pressing in a double acting steel die yielded sintered test bars with an average flexural strength of 235.6 MPa with a critical flaw size of approximately 100 μm . An aqueous slurry pressing technique produced sintered test bars with an average flexural strength of 440.8 MPa with a critical flaw size of approximately 25 μm . Image analysis revealed a reduction in both pore area and pore size distribution in the slurry pressed sintered test bars. The improvements in the slurry pressed material properties are discussed in terms of reduced agglomeration and improved particle packing during consolidation.

INTRODUCTION

Silicon carbide and silicon nitride are currently being evaluated as replacements for strategic metals in applications ranging from heat engines to industrial heat exchangers (refs. 1 to 3). Their primary drawback is the inability to control fracture generating flaws which lead to component failure (ref. 4). In most cases, the fracture originating flaws are introduced into the "green" compact prior to sintering (refs. 4 to 6). They are commonly agglomerates and metallic inclusions which arise from the starting powder or the subsequent processing steps.

From previous work, it was apparent that agglomerates, which are difficult to eliminate from dry-powder consolidation processes, were controlling the flexural strength of sintered SiC test specimens (refs. 7 and 8). The mechanical property dependence upon these flaws obscured the effect of such important variables as composition and sintering time and temperature on the sintered properties of SiC. Thus, there was a need to develop new consolidation techniques which would avoid or eliminate agglomeration. One such method is slurry pressing (refs. 9 and 10). Since a slurry is fluid, particle arrangement is greatly enhanced during pressing. This avoids residual stresses due to both contact with the die wall and bridging of particles during dry-powder pressing (ref. 11). Also, colloidal techniques may be used to both improve the dispersion of the powder (i.e., avoid van der Waals attractive forces) and control the flocculation of the powder during consolidation into a uniform, dense body.

It was the intention of this study to first demonstrate a difference between the baseline dry pressing process and the new slurry pressing process. Once a difference was shown, it was believed that further progress could be

made by using colloidal techniques to provide the best particle packing possible with the available starting powder.

EXPERIMENTAL PROCEDURE

The slurry pressing die is schematically shown in figure 1. The die works on a push-pull principle to draw the liquid out of the slurry. The bottom part of the die allows the liquid to be pushed out through exhaust holes in the bottom plunger during pressing. Liquid is pulled out from the slurry through an exhaust hole in the top plunger which is attached to a vacuum pump. The other components of the die include: (1) filter paper to trap the powder, (2) porous stainless steel for uniform expulsion of liquid and forming, and (3) preformed bulk filter paper which swells during pressing to prevent leakage around the plunger.

Both viscometry and sedimentation were used to determine the best dispersion of the powder in water. Twenty grams of SiC were suspended in 200 ml of deionized, distilled water in a 1 liter polyethylene bottle with 400 g of 12 by 12 mm SiC cylinders¹ as a mixing aid. Ten milliliters of the slurry was dispensed into each of enough 2.6 i.d. by 8.4 cm glass bottles to cover the pH range 2 to 14. The pH was adjusted with appropriate additions of acetic acid, ammonium hydroxide, and distilled water to bring the total volume to 15 ml. Each bottle was then stoppered and inverted end over end 10 times and subjected to 100 W of direct ultrasonic energy² for 5 min. Viscosity measurements³ were made immediately after ultrasonic treatment of each slurry. Measurements were made on at least two separate slurries for each pH level. A separate series of slurries was given ultrasonic treatment, stoppered and allowed to stand undisturbed. Sediment height and other characteristics were noted after 2 weeks.

The experimental design for this study is shown in figure 2. The steps involved in preparing, sintering, and evaluating both dry pressed and slurry pressed specimens are shown. The SiC powder was a 50:50 mixture of two commercially available high purity α -silicon carbides.⁴ This mixture was chosen because initial slurry pressing data indicated that the type 1 powder did not sinter alone and the free carbon content of the type 2 powder was excessive. Amorphous boron⁵ and carbon resin⁶ were added as sintering aids to the powder. The chemical composition of the starting powders and the mixtures is shown in table I.

The dry pressed specimens were prepared by combining 200 g of SiC powder and additives with 500 ml of ethanol in a 1 liter polyethylene bottle. Four hundred grams of 12 by 12 mm SiC cylinders were added as a mixing aid and

¹The Carborundum Company, Niagra Falls, NY.

²Braunsonic 1510 ultrasonic probe, B. Braun Instruments, South San Francisco, CA.

³Brookfield Model LVT viscometer, Brookfield Engineering Laboratories, Inc., Stoughton, MA.

⁴Type 1 and Type 2 α -SiC, Hermann C. Starck Co., Berlin.

⁵99.5 percent amorphous boron, Cerac, Inc., Milwaukee, WI.

⁶Resin 22352, Durez Division, North Tonawanda, NY.

the bottle was rotated at 80 rpm for 72 hr. After mixing, the slurry was dried on a hot plate at 150 to 200 °C. This procedure produced large, hard flakes which were then dry milled for several hours to reduce their size. The milled powder was sieved through a 100 mesh screen. Bars (approximately 3.8 by 0.8 by 0.45 cm) were dry pressed at 83 MPa in a double acting WC - lined die. The bars were then sealed in latex tubing and hydrostatically pressed at 413 MPa.

Two groups of slurry pressed specimens were fabricated. The specimens of the first group were prepared by mixing 200 g of SiC powder plus additives with 225 ml ethanol (approximately 22 volume percent solids) and 400 g of SiC media in a polyethylene bottle for 72 hr. After mixing, the slurry was pressed into "green" pucks (approximately 5.0 cm in diameter by 0.8 cm thick) using an applied pressure of 14 MPa. The pucks were kept in a vacuum dessicator over ethanol for 24 hr followed by drying in ambient air for 24 hr. The pucks were vacuum sealed in plastic bags and hydrostatically pressed at 413 MPa. The pucks were removed from the bags and dried at 100 °C in air for 12 hr. These pucks and test specimens derived from them will hereafter be referred to as ethanolic slurry pressed specimens.

The second group of slurry pressed specimens were prepared by mixing 200 g of SiC plus additives with 225 ml solution of water/ammonium hydroxide (pH 11.2) and 400 g of cylindrical SiC media in a polyethylene bottle. The pH of the slurry was adjusted to 11.0 with ammonium hydroxide. After mixing for 72 hr, the pH was readjusted to 11.0. The slurry was pressed into "green" pucks, dried, and hydrostatically pressed in a manner similar to the first group. These pucks and test specimens derived from them will hereafter be referred to as aqueous slurry pressed specimens.

Both the dry pressed and slurry pressed specimens were randomized, loaded in an induction furnace, and sintered at 2150 °C for 1.75 hr under an Argon atmosphere. The heating rate was 50 °C/min. After sintering, the slurry pressed pucks were machined into MOR test bars (approx 3.0 by 0.6 by 0.3 cm). Both the dry pressed and slurry pressed bars were surface ground. The finish surface grinding step required ten passes in the longitudinal direction using 400 grit diamond. All edges of the bar were beveled.

Characterization of the sintered parts included density; determined by weighing and measuring the MOR bars, 1/4 point four point flexural strength (0.95 cm inner span, 1.90 cm outer span), and identification of the fracture origins. In addition, a cross section from two sintered test bars from each of three fabrication processes was polished and etched in order to evaluate the effect of the different fabrication processes on the microstructure. The microstructure of each specimen was evaluated via optical microscopy and image analysis. Grain size was measured by the line-intercept method. The image analyzer⁷ was capable of determining features on the surface of the specimen that were different from the matrix. It was also capable of being programmed to scan about a fixed reference point so that the microstructure of more area could be efficiently evaluated. Five random points were selected on each specimen and at each of these points 25 areas were examined (5 across and 5 down). The total area evaluated on each specimen was >0.015 m².

⁷Quantimet 900, Cambridge Instruments Inc., Monsey, NJ.

RESULTS

The results of the viscometry and sedimentation are combined in figure 3. The viscosity versus pH curve shows two areas of low viscosity at pH 2 and 11. This indicates that two regions of maximum dispersion exist. The sediment height versus pH curve has a minimum at pH 11 to 12. The large sediment volume indicates that large floccs formed in the region of pH 2 to 8.5. When the particles repel one another and disperse, the packing arrangement becomes more ideal as they settle. Such a situation is illustrated in the pH range 9 to 13. The two curves indicate that the better operating pH would be approximately 11.0.

The "green" densities of the dry pressed, ethanolic slurry pressed, and aqueous slurry pressed specimens were 2.05, 2.06, and 2.10 g/cm³, respectively. The sintered densities for the same specimens were 2.93, 2.91 and 3.16 g/cm³, respectively. No significant difference in density was observed between the dry pressed and ethanolic slurry pressed bars. However, bars fabricated by aqueous slurry pressing showed a significant increase in both "green" and sintered density over bars produced by both the dry pressing and ethanolic slurry pressing.

A comparison of the flexural strengths for each forming method is shown in table II. The results indicate that significant improvements of 57 and 87 percent in strength were obtained by ethanolic slurry pressing and aqueous slurry pressing, respectively, when compared to dry pressing. Also, aqueous slurry pressing produced specimens which were 19 percent stronger than those produced by ethanolic slurry pressing.

Photomicrographs of polished and etched cross sections of the surface of dry pressed and ethanolic slurry pressed specimens looked similar, as shown in figures 4(a) and (b). The average grain size was 7.0 and 6.8 μm , respectively. However, figures 5(a) and (b) show that the distribution of pores is different. In the dry pressed case, there are large regions of porosity which surround more dense areas to form large circumferential cracks. The ethanolic slurry pressed specimens have a more uniform distribution of pores without the obvious crack formation; however, relatively large domains still exist. The aqueous slurry pressed specimens exhibited a large average grain size (8.0 μm) as shown in figure 4(c). Also evident is grain elongation with grains as long as 50 μm . The largest measured aspect ratio was 5:1. This would suggest that grain elongation occurred after densification since theoretical density was achieved (ref. 12). This indicated that the sintering time was too long for this case. The microstructure in the aqueous slurry pressed case was more uniform and did not exhibit the domains that were present in the other cases (fig. 5(c)).

A compilation of the image analysis data is shown in table III. In this table the total number of features which were evaluated are shown for each specimen as well as the area examined, average, and the largest pore areas (the area is determined using an elliptical-size pore approximation) and the average and largest pore length. Analysis of the data presented in table III indicates that the average pore area is significantly reduced by aqueous slurry pressing. The average pore area was reduced by aqueous slurry pressing by approximately 80 and 84 percent from those values obtained via dry pressing and ethanolic slurry pressing, respectively. No significant difference in the average pore length was observed between any of three fabrication processes. Figure 6 shows that the distribution of the pore lengths for the aqueous slurry pressed specimens is shifted to lower values than for the other two fabrication processes.

There is no apparent difference in pore length distributions between dry pressed and ethanolic slurry pressed specimens.

Representative fracture surfaces for the three conditions are shown in figure 7. All three exhibit typical transgranular fracture (ref. 13). The dry pressed and ethanolic slurry pressed specimens showed a much greater amount of porosity than the aqueous slurry pressed specimens. The dry pressed specimens typically failed at an approximately 100 μm agglomerate (fig. 7(a)). Other critical flaws such as pores and cracks were noted, but in much lower frequency. The ethanolic slurry pressed specimens exhibited a wide range of fracture origin types; from large (approx 120 μm) agglomerates to small elongated cracks. A typical fracture origin is shown in figure 7(b). Fracture origins for the aqueous slurry pressed specimens were difficult to identify since the majority of the specimens shattered upon fracture. The typical fracture origin in the remaining specimens was a small, 25 to 30 μm , agglomerate as shown in figure 7(c). Again, other flaw types were noted, but in much lower frequency.

DISCUSSION

A 57 percent improvement in room temperature flexural strength was attained by ethanolic slurry pressing instead of dry-powder pressing. This improvement was demonstrated even though the average densities of the two populations were the same and the carbon content of the slurry pressed specimens was lower (table I). Furthermore, the distribution of pore lengths as well as the grain size were nearly identical for the two populations.

The data suggest that the improvement in strength is due to differences in microstructure and fracture origin. As shown in figure 5, the porosity in the dry pressed specimen was inhomogeneous and was frequently associated with large agglomerated structures within the microstructure. Such a microstructure is typically encountered when an agglomerated powder is sintered. The agglomerates sinter at a different rate than the matrix, creating large circumferential cracks in the microstructure (ref. 5). This situation is also encountered on the fracture surface where the critical flaw is a dense agglomerate surrounded by a void area. In contrast, the microstructure of the ethanolic slurry pressed specimens exhibits a more uniform porosity and the domains are much smaller and more evenly dispersed than in the dry pressed case. The fracture origin has a grain morphology comparable to the surrounding matrix and the circumferential crack is not nearly as wide. This indicates that this "agglomerate" did not exist in the slurry and formed as a result of cracking during drying or hydropressing of the puck. It is clear that the differences shown in figure 5 are due only to changes in the fabrication technique, not the chemical composition.

Another possible reason for the strength increase might be the slight reduction in oxide contamination (table I) in the ethanolic slurry pressed specimens relative to those that were dry pressed (ref. 14). It is not known if the decrease in free C in the ethanol pressed specimens contributed to the increase in strength.

The increase in strength for the aqueous slurry pressed specimens is due to the 8 percent reduction in porosity (ref. 15). Also, the homogeneity of the pore distribution is greatly improved and there are no visible domains as noted in the other two cases. The average pore size is reduced by 50 percent,

and although the fracture origins are typical of both of the other cases, they are up to 80 percent smaller.

The combined use of colloidal techniques and slurry pressing reduced the residual agglomerate size and allowed for more uniform particle arrangement (ref. 16). Pressing in water also allowed for a greater retention of free C in the "green" state relative to pressing in alcohol. The source of carbon in the Starck SiC powder is soluble in ethanol and not in water. These aspects led to a stronger, more uniform, and dense structure.

SUMMARY OF RESULTS

A new slurry pressing technique improved the sintered strength of SiC by 57 percent over the dry powder pressing process. The improvement was due to the elimination of agglomerates in the dry powder by going to a wet system and the resulting improvement in microstructural uniformity.

Colloidal techniques improved the dispersion and suspension of the SiC in water and improved the strength by 87 percent over dry pressing. This also represents a 19 percent improvement in strength over ethanolic slurry pressing. This improvement was primarily due to reduced porosity as shown by image analysis and reflected in improved green and sintered densities. Also contributing to the improved strength was a smaller pore size distribution and a reduced critical flaw size.

Thus, with careful colloidal control of the slurry, aqueous slurry pressing provides a means of consolidating ceramic powders to form simple to moderately complex "green" compacts without added binders. This technique should prove useful for producing parts made from particulate and whisker toughened as well as monolithic ceramics. It should also provide an avenue for studying the many other variables which contribute to the reliability of current structural ceramics.

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TABLE 1

CHEMICAL COMPOSITION OF STARTING POWDERS AND EXPERIMENTAL MIXTURES (wt%)

	FREE C	O	B	N	Al	Fe	Ti	V
TYPE I ^a	1.94	0.60	----	0.15	0.015	0.025	0.016	0.018
TYPE II ^b	6.95	1.25	0.65	0.17	0.013	0.027	0.016	0.013
DRY PRESS MIX	4.80	0.93	0.60	0.15	0.015	0.025	0.016	0.015
SLURRY PRESS - ETHANOL ^c	3.48	0.83	0.60					
SLURRY PRESS - H ₂ O/NH ₄ OH ^c	4.75	0.95	0.60					

^a HERMANN C. STARCK, BERLIN - TYPE 1 α - SiC^b HERMANN C. STARCK, BERLIN - TYPE 2 α - SiC^c COMPOSITION AFTER-PRESSING

TABLE 2

COMPARISON OF SPECIMEN PROPERTIES

TREATMENT	NUMBER OF SPECIMENS	GREEN DENSITY $\rho_g(\text{g}/\text{cm}^3)$	SINTERED DENSITY $\rho_s(\text{g}/\text{cm}^3)$	SINTERED MOR (MPa)
DRY PRESSED	18	2.05 ± .02 (64.4% TD)	2.93 ± .02 (92.1% TD)	235.6 ± 18.4 (34.2 ± 2.7 ksi)
SLURRY PRESSED ETHANOL	22	2.06 ± .01 (64.7% TD)	2.91 ± .04 (91.5% TD)	370.5 ± 56.7 (53.8 ± 8.2 ksi)
SLURRY PRESSED H ₂ O/NH ₄ OH	24	2.09 ± .02 (65.7% TD)	3.16 ± .04 (99.3% TD)	440.8 ± 100 (640 ± 14.5 ksi)

TABLE 3

COMPILATION OF IMAGE ANALYSIS DATA

DESCRIPTION	FEATURES	TOTAL AREA EXAMINED (cm ²)	PORE AREA		PORE LENGTH	
			AVG.	LARGEST*	AVG.	LARGEST*
			(μm^2)	(μm^2)	(μm)	(μm)
DRY-PRESS	25072	0.015	9	600, 440, 400	4	80, 60, 60
	29279	0.018	10	840, 550, 400	4	60, 60, 50
SLURRY-PRESS W/ETHANOL	22928	0.018	13	210, 180, 170	5	30, 30, 30
	29618	0.018	12	1200, 800, 400	5	80, 50, 40
SLURRY-PRESS W/H ₂ O/NH ₄ OH	28622	0.018	2	190, 130, 120	2	20, 20, 20
	20894	0.018	4	360, 250, 190	2	80, 60, 60

*THE THREE LARGEST FEATURES DETERMINED FROM IMAGE ANALYSIS ARE CITED

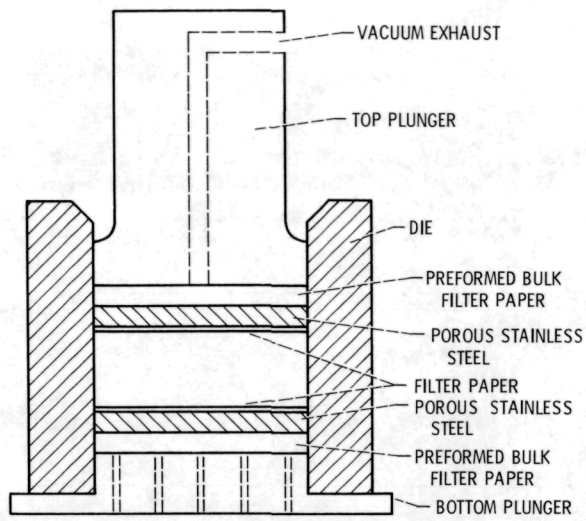


Figure 1. - Schematic drawing of the slurry pressing die.

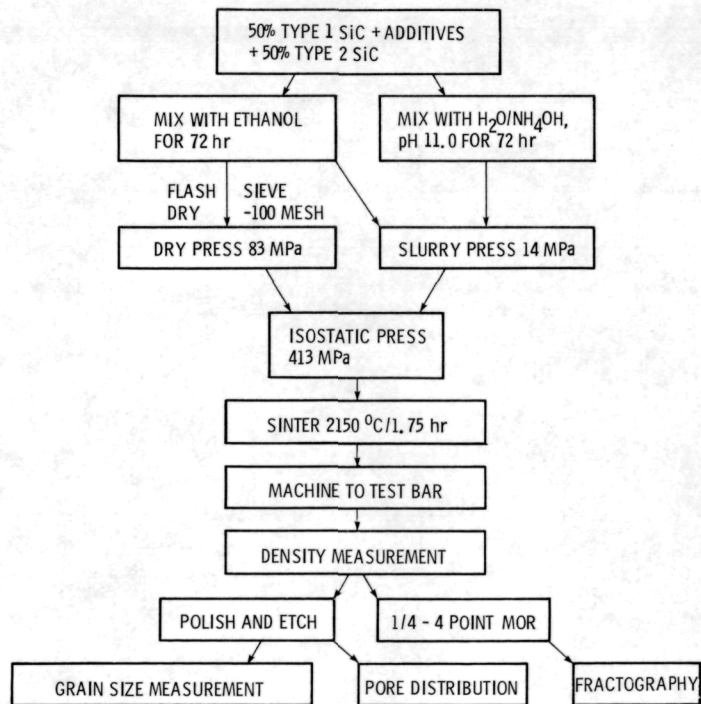


Figure 2. - Flow diagram of experimental procedure.

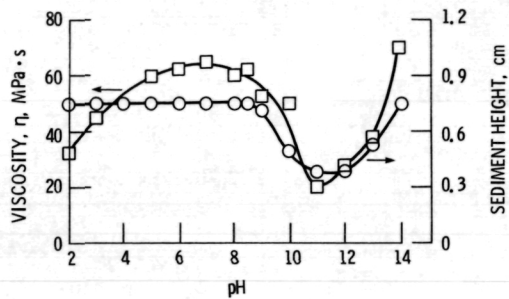


Figure 3. - The effect of pH on the dispersion of alpha-SiC.

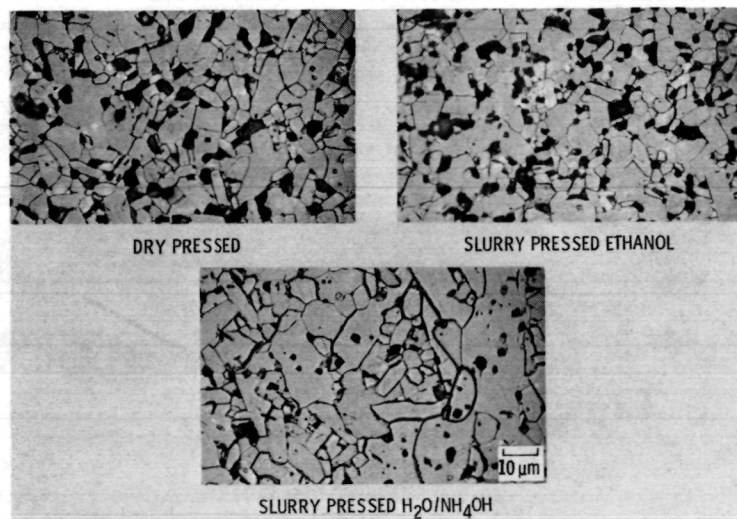


Figure 4. - Photomicrographs of polished and etched cross-sections of alpha-SiC specimens sintered for 1.75 hr at 2150 °C and fabricated by (a) dry pressing, (b) slurry pressing from ethanolic slurry, and (c) slurry pressing from pH adjusted aqueous slurry. (1000 X).

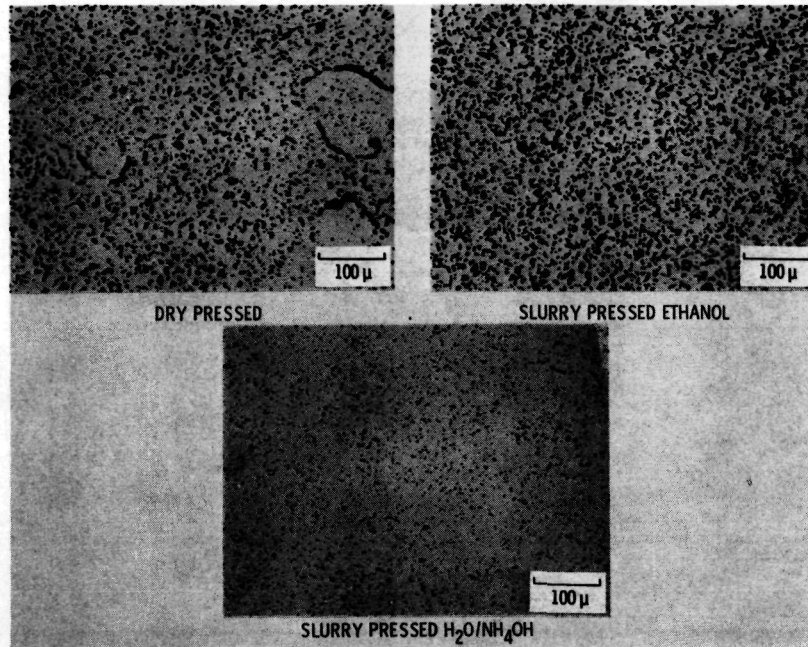


Figure 5. - Photomicrographs of polished and etched cross-sections of alpha-SiC specimens sintered for 1.75 hr at 2150 °C and fabricated by (a) dry pressing, (b) slurry pressing from ethanolic slurry, and (c) slurry pressing from pH adjusted aqueous slurry. (250 X).

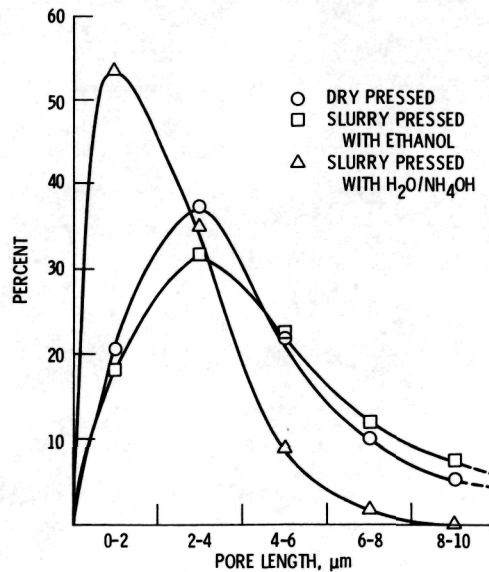


Figure 6. - Pore distribution of sintered alpha-SiC for three different fabrication methods.

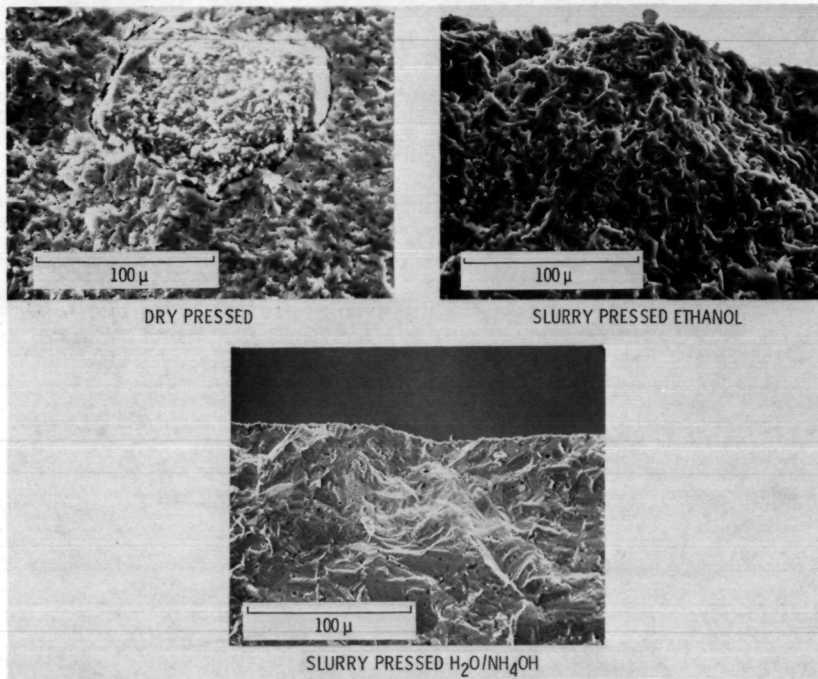


Figure 7. - Typical fracture origins found in sintered alpha-SiC specimens fabricated by (a) dry pressing, (b) slurry pressing from ethanolic slurry, and (c) slurry pressing from pH adjusted aqueous slurry.

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