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Behavior of Composites for the NASP Project

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BEHAVIOR OF COMPOSITES FOR THE NASP PROJECT*

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ABSTRACT

It is well known that ceramics are strong, highly heat resistant, refractory materials. Unfortunately, they are brittle and fail catastrophically. However, the strength as well as fracture toughness of ceramics can be greatly improved with the addition of continuous fibers, thus obtaining ceramic matrix composites (CMC). Normally the addition of aligned continuous fibers to a matrix degrades the transverse (the direction 90° to the fiber axes) properties while greatly improving the longitudinal properties. It has been shown that the addition of whiskers to a continuous fiber CMC will improve moduli and strength properties in both the transverse and longitudinal directions. Any improvement in the transverse direction is a tremendous advantage as loads may not always act along the longitudinal fiber direction, thus avoiding the necessity for lamination and angle plies.

Strengthening ceramics by the addition of fibers and whiskers can hopefully produce a reliable refractory material that will offer service on the National Aerospace Plane (NASP) and other related space/hypersonic vehicles. If CMC can be shown to have high strength and toughness, they could fill a spot on the nosecap, leading edge surfaces as well as primary structures of the NASP.

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INTRODUCTION

Ceramic matrix composites (CMC) are well known to withstand high temperatures (1000°C and higher).[1,2] This characteristic immediately made CMC a contender when the materials search was begun for the National Aerospace Plane (NASP).[3] CMC were considered for nose cap and leading edge structures of the NASP, areas that must withstand extremely high temperatures. But before this potential use is achieved, CMC must overcome their poor transverse properties.[4]

Reinforcing the brittle CMC with continuous fibers increases its strength and fracture toughness in the direction of the fibers while degrading the transverse direction strength.[5] The addition of randomly oriented fibers is theorized to improve the transverse properties of unidirectional fiber reinforced composites. This paper details the research undertaken to test this theory as part of an undergraduate research opportunity.

MATERIALS

Any material that can be considered for use on a hypersonic or NASP application must meet more demanding requirements than ordinary materials. The inherent properties of ceramic matrices meet these requirements: a high oxidation resistance, a high melting point, low coefficient of thermal expansion and chemical compatibility of fiber and matrix. The matrix used was a 8-10 micron cordierite powder from Ferro. Its properties are given in Table I. Continuous silicon carbide fibers were obtained from UBE Industries. Silicon carbide whiskers from the American Matrix Company were used to form the hybrid matrix. Fiber and whisker properties are given in Table II.

Table I. Properties of the Cordierite Matrix

Melting Point (°C)	Density (g/cm^3)	E (GPa)
1410	2.65	110

Table II. Properties of the Silicon Carbide Fibers and Whiskers

E (GPa)	Density (g/cm^3)	Diameter (μm)
200	3.2	16

The fabrication process of CMC is at best time consuming. Four specimens were prepared: matrix (M), matrix-whisker (MW), matrix reinforced with unidirectional fiber (MF) and matrix-whisker reinforced with unidirectional fiber (MWF). Reviewing the fabrication techniques of Carroll et al[2], essentially the same method was followed. The matrix was cordierite powder and the reinforcing fibers and whiskers were silicon carbide.

Pre-Preg Fabrication

The cordierite specimen was easily fabricated. Powder was measured to produce a 76mm diameter disk approximately 3mm thick. The powder was then hot pressed. To adequately mix the cordierite and silicon carbide whiskers for the matrix and whiskers specimen, the components were ball milled in an isopropyl alcohol mixture for 6 hours. The alcohol was then dried off, the powder finely ground and hot pressed.

One of the major concerns of producing fiber reinforced composites is obtaining a even mixture of matrix and fibers.[6] Continuous fibers are contained in a tow of approximately 10,000 close packed fibers and complete wetting of the fibers by the matrix is difficult. Considerable care was taken to insure that maximum fiber wetting was achieved during the fabrication of the continuous fiber reinforced composites. A slurry of cordierite powder, water and organic binders was ball milled overnight. The mass of cordierite used was 165g, enough to produce one disk. The following day the unidirectional prepregs were wound. As shown in Figure 1., the silicon carbide fibers were passed across a flame to release the fiber bundle and then passed through the continuously agitated slurry via a set of rollers. From there, the slurry coated fiber was passed in front of a compressed air nozzle to remove excess slurry and then wound onto the octagonal drum. Three layers of fiber were wound onto the drum, completely exhausting the slurry supply. It was decided to use three layers so that a specimen could be made from one winding. The prepreg was allowed to dry overnight and then it was cut into 76mm diameter disks. Care was taken to assure that the laminae were unidirectionally aligned. The organic binders were burned off of the prepregs in a small oven and then hot pressed. The organic burn-off schedule is presented in Table III.

Figure 1. Pre-Preg Winding Process for CMC

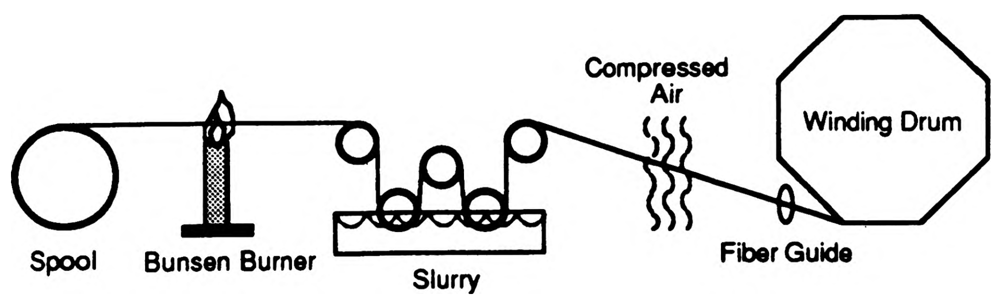


Table III. ORGANIC BURN-OFF SCHEDULE

Temperature (°C)	Burn Duration (hr)
120	1
250	1.5
450	4

The same procedure was followed for the hybrid matrix, whisker and fiber specimen. The slurry contained a 30% V of whiskers. It was prepared as the matrix only slurry with the total mass of cordierite and silicon carbide whiskers totalling 165g. The winding and hot pressing procedures were the same as those of the matrix and fiber specimen.

Hot Pressing

All specimens were hot pressed in a glassy state in a 76.2mm (three inch) graphite mold at 13.8 MPa (2000 psi) and 900°C. The specimens were gradually heated to 900°C and pressure was applied at 500°C. When the specimens reached 900°C, they were held at that temperature and pressure for one hour. All hot pressing was done at McDonnell Douglas Research Laboratory in St. Louis, MO.

Following the hot pressing, a final heat treatment was required to recrystallize the matrix. Table IV. presents the firing schedule that was used.

Table IV. Recrystallization Heat Treatment

Ramp Time (min)	Temp. (°C)	Soak Time (min)
30	275	60
360	815	180
480	950	No Soak
120	1150	120

POROSITY

A good test of the fabrication process is the final density of the composites. Experimental density was calculated by:

$$\rho_a = \frac{m}{\pi (d^2/4)\, t} \tag{1}$$

where m is the mass of the disk, d is the average diameter and t is the average thickness of the disk. The actual densities were compared with the theoretical densities which were calculated by the following rule of mixture:

$$\rho_t = \rho_f V_f + \rho_m V_m \tag{2}$$

where ρ_f is the known density of the silicon carbide whiskers and fibers, V_f is the volume fraction of SiC fibers and whiskers, ρ_m is the known density of the cordierite powder and V_m is the volume fraction of matrix. The comparison and subsequent porosity levels are presented in Table V.

Table V. Porosities of Specimens

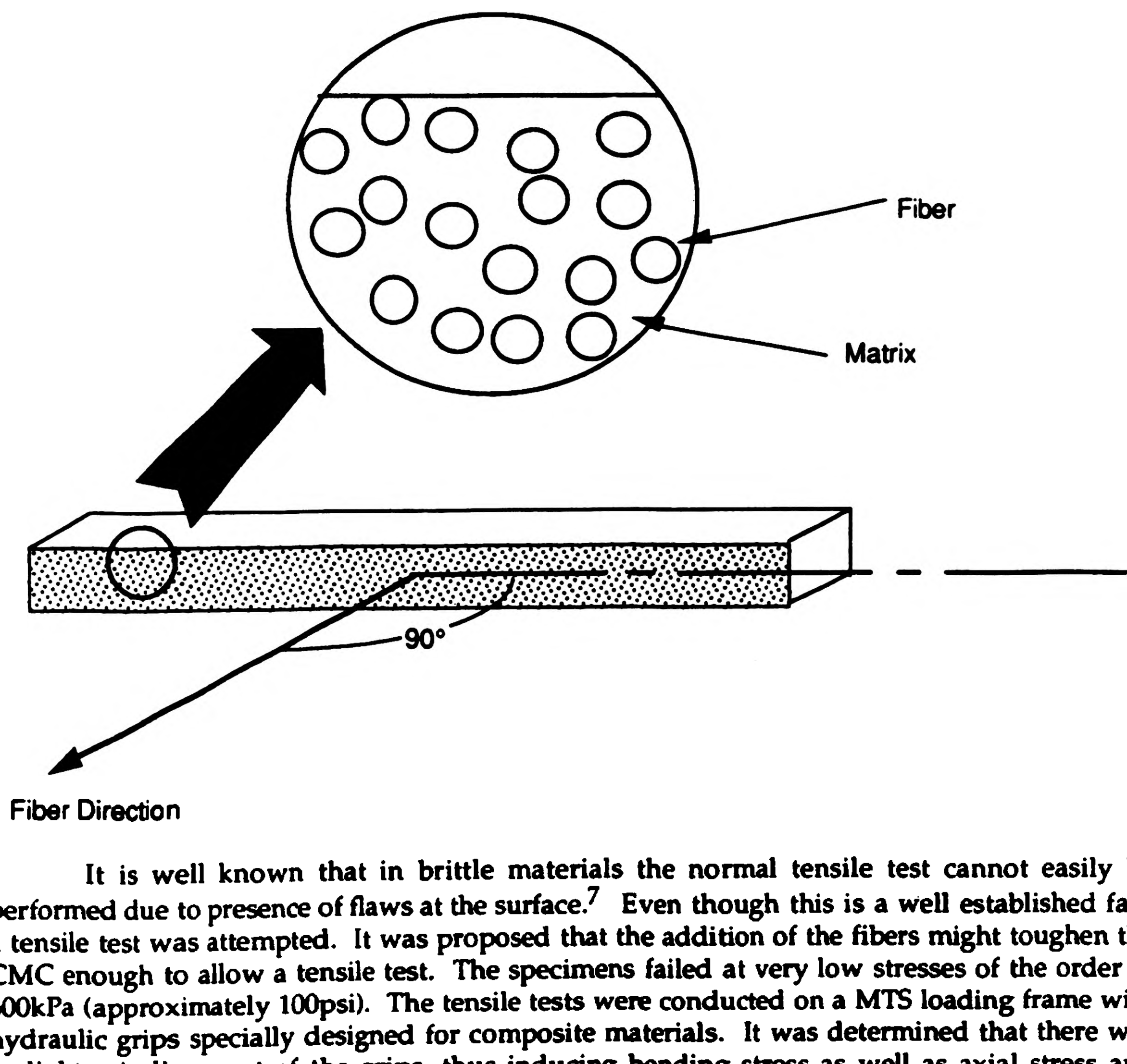
Composite System	Density (g/cm^3) Theoretical	Density (g/cm^3) Actual	% Void
Matrix	2.65	2.59	2.3
Matrix & whiskers	2.82	2.16	33.3
Matrix & fiber	2.87-2.98	1.88	35.0
Matrix, whisker & fiber	2.87-2.98	2.15	26.0

The matrix densified the most, yielding a calculated density of 97.7% of the theoretical density of the cordierite matrix. The continuous fiber reinforced disks were much worse with porosity values of 30-35% of the total disk volume. Possible explanations for these porosity levels have been explored. The most likely problem in the fabrication process is the hot pressing procedure. It is hypothesized that residual organic gases may become entrapped within the pressing die. The three specimens that exhibit the lowest densities all have organic mixers involved in their pre-pressing fabrication. Other work on CMC has produced densities of 99.7% of the theoretical value[6].

TESTING

To test the transverse properties, the four disks were cut into rectangular blocks at least 57.15cm long. Cross section dimensions of the specimens varied: matrix - 5.9mm by 4.3 mm; matrix and whisker - 6.7mm by 4.6mm; matrix and fiber - 5.9mm by 4.2mm; and matrix, whisker and fiber - 5.9mm by 3.2mm. The specimens were cut at a 90° angle to the continuous fiber direction so that the fibers were perpendicular to the length of the specimen as shown in Figure 2.

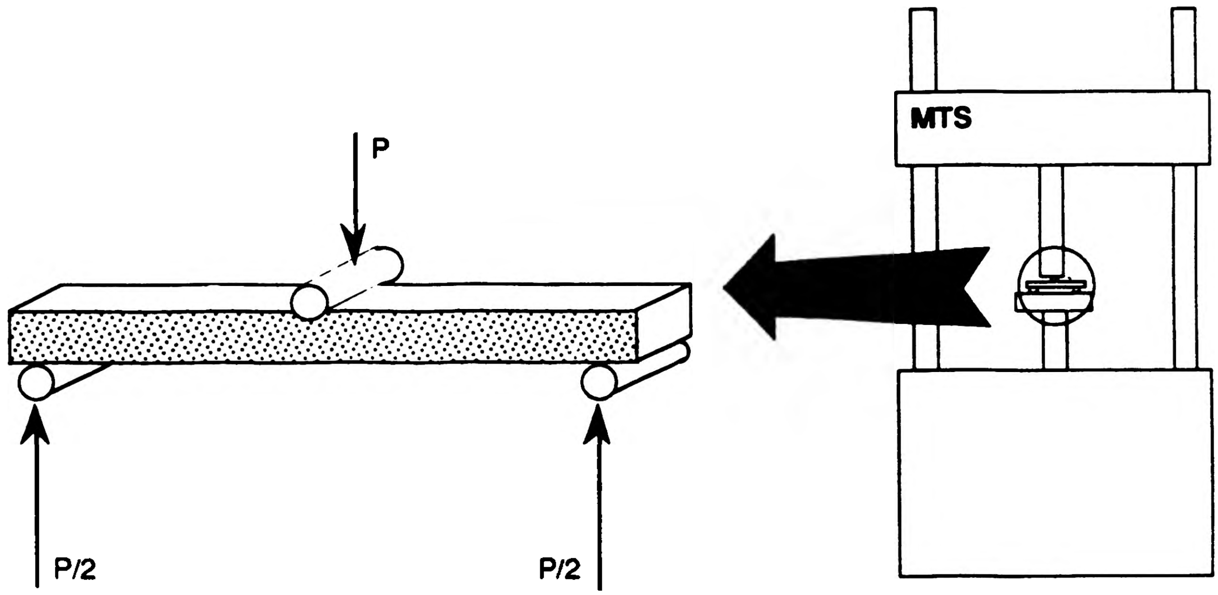
Figure 2. Fiber Orientation of Transverse CMC Specimen



It is well known that in brittle materials the normal tensile test cannot easily be performed due to presence of flaws at the surface.[7] Even though this is a well established fact, a tensile test was attempted. It was proposed that the addition of the fibers might toughen the CMC enough to allow a tensile test. The specimens failed at very low stresses of the order of 600kPa (approximately 100psi). The tensile tests were conducted on a MTS loading frame with hydraulic grips specially designed for composite materials. It was determined that there was a slight misalignment of the grips, thus inducing bending stress as well as axial stress and possibly explaining the failure at low stresses. This problem will be corrected before future tensile tests are performed.

The standard test for brittle materials is the three-point bend test where the modulus of rupture is measured. A schematic of the three-point bend test is given in Figure 3. The load P is applied at midspan of the specimen which is supported by the two rollers beneath it. At the location of the applied load, the top of the specimen is in compression while the bottom experiences a tensile stress. Failure always initiates from surface that is in tension and thus at failure, the measured stress is the ultimate tensile strength.

Figure 3. Three-Point Bend Test Setup



The three-point bend tests were conducted on a MTS loading frame at room temperature. All four types of CMC were tested at a span of 38.1mm (1.5 in) and had a span-to-depth ratio >8.0. The matrix and matrix and whiskers were loaded at a cross-head speed of 0.127mm/sec and the matrix and whisker and matrix, whisker and fiber were loaded at a speed of 0.254 mm/sec. Previous attempts at using an extensometer to measure deflection had not produced significant improvements in deflection reading so the cross head displacement was used for the deflection reading. Testing data (load and displacement) was stored on a PC via a data acquisition program and connection to the MTS Microconsole.

RESULTS

Results of the three-point bend test are given in Table 6. The modulus of rupture or the ultimate tensile strength is given by:

$$\sigma_u = \frac{3\ PL}{2\ bh^2} \tag{3}$$

where P is the applied load, L is the span and b and h are the width and height of the specimen, respectively.

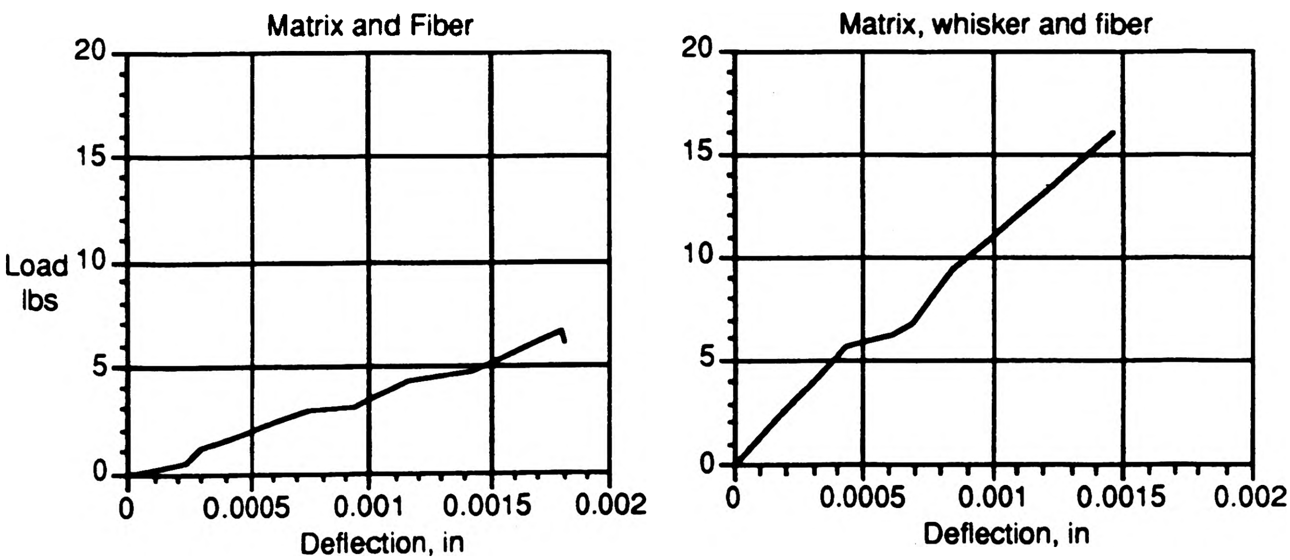
Table VI. Results of the Three-point Bend Test on 90° Fiber CMC

CMC Type	σ_{t_u} (MPa)	L/h
Matrix	112	8.88
Matrix,whisker	145	8.24
Matrix, fiber	25	9.04
Matrix, whisker,fiber	47	11.78

Three specimens of each group of CMC were tested. The values of the ultimate tensile strength given in Table VI represent the average of the three specimens' measured ultimate tensile strength. The test results show that the transverse strength of the CMC can be improved by the addition of whiskers to the matrix. An improvement in ultimate strength of 88% is indicated above. While the failure is still brittle, failure of the matrix, whisker and fiber CMC was less catastrophic as an initial crack was observed momentarily before failure. Failure of the matrix and fiber CMC was quick and catastrophic.

Figure 4. shows the toughening effect of the whiskers on the transverse properties of the matrix, whisker and fiber. The initial change in slope represents the point of matrix cracking and the whiskers provide additional load carrying capability past that point.

Figure 4. Toughening Effect of Whiskers on Transverse Direction



These test results are encouraging. It has been shown that the addition of whiskers to the matrix increase the longitudinal properties of CMC, and it appears that whiskers offer considerable improvement of transverse properties as well.[2,6] Any improvement in the transverse properties is welcome as there are few cases of purely axial loads. Unexpected loads acting in directions other than the fiber direction can now be more confidently carried.

Conducted at room temperature, the conclusion stated above must be qualified. If CMC are to be considered for the NASP and other future hypersonic vehicles, high temperature testing must be performed to complete these findings for the transverse properties. Porosity does affect the strength of the CMC, but the results presented here do serve as a valid comparison of strengths of four classes of CMC.

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