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STRENGTH AND FLEXIBILITY PROPERTIES OF ADVANCED CERAMIC FABRICS

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ABSTRACT

The mechanical properties of four advanced ceramic fabrics were measured at a temperature range of 23C to 1200C. The fabrics evaluated were silica, high- and low-boria content aluminoborosilicate, and silicon carbide. Properties studied included fabric break strengths from room temperature to 1200C, and bending durability after temperature conditioning at 1200C and 1400C. The interaction of the fabric and ceramic insulation was also studied for shrinkage, appearance, bend resistance, and fabric-to-insulation bonding. Based on these tests, the low-boria content aluminoborosilicate fabric retained more strength and fabric durability than the other fabrics studied at high temperature.

INTRODUCTION

Silica fabrics are used as part of a sewn, quilted blanket construction that has functioned as a thermal protection system (TPS) for the Space Shuttle in thermal environments up to 650C.^{1,2} For this application, the silica fabric is further toughened with a ceramic coating to provide enhanced

durability when interacting with the aeroacoustic environment.³ Future space transportation systems under study will require improved versions of this flexible ceramic blanket for thermal protection because they will be functioning in higher aeromaneuvering temperatures.

The behavior of these advanced ceramic fabrics must be determined at temperatures above 1000C for TPS fabric selection, development of fabrication techniques, and TPS storage methods.⁴ It is the objective of this paper to characterize the strength and flexibility differences of these fabrics when used at temperatures up to 1400C.

EXPERIMENTAL

Materials

<u>Ceramic Fabrics</u>. Four commercially available ceramic fabrics representing the high-temperature yarns available were obtained for this study. The fabrics were woven from these yarn types: silica, high-boria $(14\%B_2O_3)$ aluminoboro-silicate, low-boria $(2\%B_2O_3)$ aluminoborosilicate, and silicon carbide. All the fabrics used have a 5 Harness Satin weave pattern. Some of the construction properties of these fabrics are listed in Table 1.

<u>Ceramic Insulation</u>. Four classes of insulation were selected for the fabric/felt interaction studies. These were a silica felt, two aluminoborosilicate nonwoven web types, and an alumina blanket type. Some key properties are shown in Table 2.

Test Methods

<u>At-Temperature Break Strength Measurements</u>. The break strengths of the ceramic fabrics were obtained using an Instron Test Machine, Model 1122

in combination with a custom-built high temperature furnace designed by Smith et al.⁵ The furnace, shown in Figure 1, allows the test to proceed at conditioning temperatures from 23C to 1200C. The furnace consists of a heating chamber, furnace body, spacer, and a controller.

The heating element has a spring shape made of 17 gage Nichrome wire, Type A1,* that is inserted into the furnace body. A heating chamber with a 2.5 cm^2 cut-out area in the center was placed in the furnace body. The whole block is held together by two quartz rods supported by a spacer which is mounted on the attaching plate. A controller was used to adjust the temperature and is connected to the furnace by chromel-alumel, Type K thermocouples. An integrating microvoltmeter was attached to the controller for the voltage output which corresponds to the set temperature. The whole furnace was mounted on a sliding bar located at the base of the Instron Test Machine.

The fabrics (Table 1) were cut into 2.5 cm wide by 23 cm long strips in two different weave directions, warp and fill. The number of yarns per 1.25 cm of width was obtained from yarn count shown in Table 1 and was verified by actual measurement. Owing to the small heat exposure area (2.5 cm^2) of the furnace, the test samples had one-half the yarn count reported in Table 1. Approximate exposure area of sample was 1.25 cm by 2.5 cm.

A common two-part polyester resin was used to harden both ends of the fabric strips to prevent slippage of the fabric from the Instron test grips during tensile pull. The resin was smeared on both ends of the fabric strips covering about 2.5 cm² and was allowed to dry overnight. The extra vertical yarns were removed from both sides until the required yarns per 1.25 cm width were obtained.

^{*}Of type manufactured by Kanthal Corp.

The fabric strips were clamped on the Instron with a small preload (0.11 kg) to remove yarn crimp and then placed in the furnace heating zone which was preset at the test temperature. After 10 minutes of heating, the fabric sample was pulled in tension at the test temperature until the maximum break strength was obtained. The test conditions used for all the fabrics were 0.51 cm/min crosshead speed, 23 cm gage length, and a chart speed of 5.1 cm/min. A minimum of six measurements at each temperature was obtained for all fabrics tested.

Mandrel Bend Test. A mandrel bent set was used to determine the flexibility and brittleness of the ceramic fabrics. The set consists of a series of solid rods with different diameters. For this experiment, only the 2.54 cm, 0.63 cm, and 0.32 cm rods were used. The ceramic fabrics were cut into two sets of 3.8 cm by 5.2 cm pieces and conditioned in a static air environment furnace at 500C, 1200C, 1300C and 1400C for 10 minutes and 60 minutes, respectively. Another set of fabrics was heat cleaned using a procedure developed to clean the silica blankets prior to installation on the Space Shuttle.¹ All fabrics were conditioned by chemical group to prevent any interaction or contamination. After the fabrics cooled to room temperature, observation of stiffness and color was made. The fabrics were placed on the mandrel rod supported between two metal support stands. The fabrics were tightly folded around the rod in the fabric warp direction. An observation of yarn damage or fabric breakage was made using an illuminated magnifying lamp. Two measurements were obtained for each fabric at each temperature and time. Fabric/Insulation Interaction Test. The interaction of ceramic fabrics and insulation (felt) was investigated at 1200C, followed by flexibility limits obtained from the Mandrel Bend Test. The 5 cm^2 pieces of fabric and felt were placed in contact with each other (fabric as face sheet) in an air environment

furnace at 1200C for 1 hour. A second test was also conducted at 1400C for 1 hour. This thermal exposure was performed separately for each fabric/felt combination to avoid interaction of one set to another.

The test sample was removed from the furnace and allowed to cool to room temperature. The fabric was lifted off the felt surface and the physical observations made for felt shrinkage, fabric appearance, bend resistance, and fabric/felt bonding. The Mandrel Bend Test was then used to determine the flexibility of the fabric after the interaction examination.

RESULTS AND DISCUSSION

Fabric Strength

It is important that ceramic fabrics maintain (or retain) strength during TPS fabrication, installation, storage, and flight use. Break strength measurements up to 1200C were obtained to determine differences among the ceramic fabrics selected. These results are shown in Figures 2 and 3.

Figure 2 presents the effects of temperature on the strength performance of the fabrics tested in the fabric warp direction. The silicon carbide fabric had the highest room temperature strength of the four fabrics tested. It was the only fabric to lose strength between room temperature and 400C, and also had the largest loss of strength between 400C and 600C owing to finish removal. The 14% boria content aluminoborosilicate had slightly lower room temperature strength, but does lose about 60% of its original strength due to finish removal between 400C and 600C. The silica fabric had the same characteristic strength/temperature curve previously reported for silica sewing thread.⁶ The 2% boria content aluminoborosilicate fabric had the lowest room

temperature strength, but was remarkably stable through the entire temperature range tested up to 1200C.

Figure 3 shows the results of a similar study of fabric strength vs temperature in the fill direction. Strength properties of all the fabrics tested paralleled the strength curves obtained for the warp direction, except for slightly lower strength values at each temperature. This was due to lower yarn count in the fill direction. A comparison of yarn count is shown in Table 1.

Figure 4 focuses on the strength differences of the ceramic fabrics at temperatures between 600C and 1200C. Both the silica and the low-boria content aluminoborosilicate fabrics maintain fabric strength above 600C. One exception to high temperature strength stability was the high-boria aluminoborosilicate, which has a major strength transition point at about 950C. From 950C to 1200C, a further 20% strength loss occurs from that shown in Figure 2. While this fabric had the highest break strength from 500C to 1100C of all the fabrics tested, it also had the least strength retention. Decreasing strength was observed for the silicon carbide fabric particularly between 1000C and 1200C. Previous studies based on tensile strength measurements of individual silicon carbide filaments showed degradation of tensile strength at similar temperatures.⁷

Table 3 summarizes a comparison of the fabric strength retention normalized to the fabric yarn denier at three temperatures. These are: room temperature, which represents the fabric in the "as received" state; at 600C, where all fabric finish has been removed; and at 1200C, which represents a high temperature reentry condition for future flexible ceramic TPS. At room temperature, the initial break strengths ranking of the four fabrics are:

silicon carbide high-boria aluminoborosilicate silica low-boria aluminoborosilicate At both 600C and 1200C, the strength retention ranking was the same low-boria aluminoborosilicate high-boria aluminoborosilicate silica silica

Fabric Flexibility After Thermal Exposure. Table 4 summarizes the ability of the fabrics to survive bending over three different mandrel diameters after thermal conditioning at five different temperatures, and two exposure times per temperature. (Two of the thermal conditions caused no change to the fabrics. Therefore, these are not reported in the table. These were 500C and the heat-cleaning treatment reported in the test description.)

After 1200C for 10 min, the silica fabric suffered yarn damage after bending over a 2.54 cm mandrel rod, and severe fabric stiffening was observed when removed from the heat. After 60 min at 1200C, the silica fabric was brittle and did not survive a 2.54 cm diameter mandrel bend. Silicon carbide showed no change when bent over a 2.54 cm rod after 10 min at 1200C, but showed yarn damage at both 0.63 cm and 0.32 cm mandrel diameters. After 60 min exposure to 1200C, the silicon carbide fabric had yarn damage from the 2.54 cm mandrel bend. The fabric failed the 0.63 cm bend. In addition, the fabric color changed from a black to a blue-black after the 10 min exposure, and to a blue-green surface color after 60 min along with increased fabric stiffness. This observation of color change for the silicon carbide fabric

(although not pertinent to this study), might indicate some change in optical properties after exposure to temperatures up to 1200C. The flexibility of the high-boria content and the low-boria content aluminoborosilicate fabrics were unaffected at 1200C.

After separate 10 min and 60 min temperature exposures at 1300C, the lowboria aluminoborosilicate fabric was still pliable and maintained flexibility at three mandrel diameters. The high-boria aluminoborosilicate fabric became stiff after 10 min at 1300C; the fabric survived bending only over a 2.54 cm mandrel.

After 1400C exposure for 10 min, and 60 min each, the high-boria content aluminoborosilicate fabric was very stiff and failed instantly when bent over a 2.54 cm mandrel. Only the low-boria content aluminoborosilicate fabric survived all temperature/time conditions at all the mandrel diameters selected. A slight fabric stiffness was noted at both time periods at 1400C. <u>Interaction of Fabric/Felt Layers at 1200C and 1400C</u>. The interaction of the different fabric to felt combinations at 1200C and 1400C was determined from felt shrinkage, fabric appearance, mandrel bend resistance, and fabric/felt bonding. A summary of these evaluations is listed in Table 5 for the 1200C study, and Table 6 for the 1400C study. The observations made as a result of these studies are described below.

<u>Felt Shrinkage</u>. At 1200C (Figure 5), negligible shrinkage was measured for all four insulation materials. At 1400C (Figure 6), the silica felt had a volume shrinkage of 60% resulting in an irregular shape with all four fabrics. (This extensive shrinkage for silica TPS has been reported in the literature for rigid silica tiles.⁸) The remaining three insulations showed negligible shrinkage owing to moisture loss or sizing removal. This was confirmed by weight loss measurements of less than 5%.

<u>Fabric Appearance</u>. The silica fabric was stiff and brittle after thermal exposure to both 1200C and 1400C with each of the four insulation materials. The silica fabric also shrank to the irregular form of the silica felt as a result of the 1400C conditioning. After the 1200C conditioning, the high-boria content aluminoborosilicate fabric became stiff after removal from each of the insulation surfaces, but became stiffer after the 1400C conditioning. The low-boria content aluminoborosilicate fabric showed no change after exposure to 1200C and was only slightly stiffened from the 1400C conditioning. The silicon carbide fabric behaved similarly to the very stiff fabrics and also showed the color change (observed from Table 4) at both the 1200C and 1400C conditions.

Mandrel Bend Resistance. After removal from each of the insulation pieces the silica fabrics were brittle and did not survive the 2.54 cm mandrel bend for either the 1200C or 1400C exposure. The silica carbide fabrics behaved similarly to the silica fabric after the 1400C exposure, but did pass the 2.54 cm and 0.63 cm bend before failing a 0.32 cm bend at 1200C. As reported in Table 6, the high-boria content aluminoborosilicate fabric was mainly unable to pass the 0.32 cm mandrel bend after removal from the insulation materials, except after removal from the low-boria content aluminoborosilicate insulation. In this case, after thermal contact with the low-boria aluminoborosilicate insulation, the high-boria content fabric suffered only yarn damage after bending over a 0.32 cm mandrel. After the 1200C exposure (Table 5), the high-boria aluminoborosilicate fabric survived the 0.32 cm mandrel bend in all cases. From the comparison of the bend resistance shown in Tables 5 and 6, the low-boria content aluminoborosilicate fabric passed the 0.32 cm mandrel bend after removal from each of the insulation materials. It was the only fabric to do this for both temperatures.

Fabric Felt Bonding. No fabric-to-felt interaction occurred for the fabric/felt combinations reported in Table 5. From Table 6, only the silica fabric showed a weak adhesive bond when peeled away from each of the four insulation surfaces. No evidence of any cohesive bonding was observed and none of the other fabric/felt layers exhibited any kind of adhesion. In all cases, the fabric lay loosely on the felt surfaces.

SUMMARY

Fabric strength for all fabrics except the low-boria aluminoborosilicate was significantly higher at room temperature than at 1200C. The fabrics had the following strength ranking at room temperature:

> silicon carbıde hıgh-borıa alumınoborosılicate

sılıca

low-boria aluminoborosilicate

At 1200C, the fabric strength ranking was:

low-boria aluminoborosilicate high-boria aluminoborosilicate

silica

silicon carbide

The low-boria content fabric retains 100% of its strength through the temperature ranges tested. The other three fabrics show the highest rate of strength loss at 400C to 450C. In addition, a strength loss transition point at 950C was observed for the high-boria content aluminoborosilicate fabric. Silicon carbide fabrics were determined to have a 30% strength loss between 1000C and 1200C.

Fabric flexibility rank after exposure to 1200C was:

low-boria aluminoborosilicate high-boria aluminoborosilicate silicon carbide silica

Only the low-boria content fabric survived any mandrel bending after thermal treatment at 1400C.

At 1400C, the silica fabric-to-silica felt combination suffered severe shrinkage and brittleness. Negligible shrinkage occurred for the other three fabric/felt combinations.

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Yarn	Fabric Style	Denier	Yarn Count, YPM	Thickness (cm)	Wt (kg/m ²)	
Silica	570 ^a	300 4/2	1535 × 984	0.074	0.68	
Aluminoboro- silicate ^C (14% B ₂ O ₃)	хс804 ^ь	300 2/4	1457 × 906	0.053	0.68	
Aluminoboro- silicate ^C (2% B ₂ 0 ₃)	440°	900 1/2	1220 × 827	0.061	0.41	
Silicon carbide ^d	xc806 ^b	600 1/4	1260 × 669	0.066	0.57	
^a Manufactured by J. P. Stevens Co., or Alpha Associates						
but we have a lower of the second						

Table	1.	Fabric	Construction
TUDIC		100110	0011501 4001011

^bWoven by Hexcel Corp. ^CManufactured by 3M Co. ^dManufactured by Nippon Carbon Co.

Properties	Silica	Aluminoboro- silicate (14% B ₂ O ₃)	Aluminoboro- silicate (2% B ₂ 0 ₃)	Alumina
Density, kg/m ³	96.1	10.4	12.0	96.1
Thickness, cm	1.27	1.91	1.91	2.54
Form	Felt	Nonwoven web	Nonwoven web	Blanket
Color	White	White	White	White

Table 2. Felt Insulation Properties

Table 3. Comparison of Fabric Strength Retention

Normalized to Denier

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kg/Denier	× 10 ⁻³	(warp dı	rection)	
Fabric	R.T	600C	1200C	
Silicon Carbide	4.82	0.49	0.33	
Aluminoborosilicate (14% B ₂ O ₃)	2.93	1.13	0.40	
Silica	1.24	0.15	0.11	
Aluminoborosilicate (2% B ₂ O ₃)	0.72	0.72	0.72	



Felt					
Fabric	Property Measured	Silica	Aluminoboro- silicate (14% B ₂ 0 ₃)	Aluminoboro- silicate (2% B ₂ 0 ₃)	Alumina
Silica	Felt, % shrinkage Fabric appearance Mandrel bend Fabric/felt bonding	Negligible Very stiff Failed 2.54 cm None	Negligible Very stiff Failed• 2.54 cm None	Negligible Very stiff Failed: 2.54 cm None	Negligible Very stiff Failed. 2.54 cm None
Alumino- borosilicate (14% 3 ₂ 0 ₃)	Felt, % shrinkage Fabric appearance Mandrel bend Fabric/felt bonding	Negligible Stiff Passed 0.32 cm None	Negligible Stiff Passed 0.32 cm None	Negligible Stiff Passed. 0.32 cm None	Negligible Stiff Passed 0.32 cm None
Alumino- borosilicate (2% B ₂ 0 ₃)	Felt, % shrinkage Fabric appearance Mandrel bend Fabric/felt bonding	Negligible No change Passed. 0.32 cm None	Negligible No change Passed• 0.32 cm None	Negligible No change Passed 0.32 cm None	Negligible No change Passed: 0.32 cm None
Silicon carbide	Felt, % shrinkage Fabric appearance Mandrel bend Fabric/felt bonding	Negligible Very stiff Failed. 0.32 cm None	Negligible Very stiff Failed 0.32 cm None	Negligible Very stiff Failed: 0.32 cm None	Negligible Very stiff Failed 0.32 cm None

Table 5 Interaction of Fabric/Felt Layers After Exposure to 1200C, 1 Hour

Felt						
Fabric	Property Measured	Silica	Aluminoboro- silicate (14% B ₂ 0 ₃)	Aluminoboro- silicate (2 % B ₂ 0 ₃)	Alumina	
Silica	Felt, % shrinkage Fabric appearance Mandrel bend Fabric/felt bonding	60 Very stiff Failed• 2.54 cm Weak adhesive	Negligible Very stiff Failed. 2.54 cm Weak adhesive	Negligible Very stiff Failed: 2.54 cm Weak adhesive	Negligible Very stiff Failed. 2.54 cm Weak adhesive	
Alumino- borosilicate (14% B ₂ 0 ₃)	Felt, \$ shrinkage Fabric appearance Mandrel bend Fabric/felt bonding	60 Very stiff Failed 0.32 cm None	Negligible Very stiff Failed 0.32 cm None	Negligible Very stiff Passed. 0.32 cm (Yarn damage) None	Negligible Very stiff Failed 0.32 cm None	
Alumino- porosilicate (2% B ₂ O ₃)	Felt, % shrinkage Fabric appearance Mandrel bend Fabric/felt bonding	60 Slightly stiff Passed. 0.32 cm None	Negligible Slightly stiff Passed. 0.32 cm None	Negligible Slightly stiff Passed: 0.32 cm None	Negligible Slightly stiff Passed 0.32 cm None	
Silicon carbide	Felt, \$ shrinkage Fabric appearance Mandrel bend Fabric/felt bonding	60 Very stiff Failed. 2.54 cm None	Negligible Very stiff Failed: 2.54 cm None	Negligible Very stiff Failed: 2.54 cm None	Negligible Very stiff Failed• 2.54 cm None	

Figure Titles

- Figure 1. Furnace for High Temperature Strength Measurements.
- Figure 2. Effect of Temperature on Warp Break Strength of Ceramic Fabrics.
- Figure 3. Effect of Temperature on Fill Break Strength of Ceramic Fabrics.
- Figure 4. Fabric Warp Break Strength Between 600C and 1200C.







Figure 2



Figure 3



Figure 4

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16 Abstract				······	
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