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RECENT ADVANCES IN CARBON-CARBON SUBSTRATE TECHNOLOGY AT NASA LANGLEY RESEARCH CENTER

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INTRODUCTION

Figure 1 shows a comparison of specific strengths of candidate high-temperature materials as a function of temperature. From this figure, it is apparent why there is an interest in carbon-carbon composites for applications as strong, light-weight TPS, or as hot structure, for applications above 2500°F. The lower bound of the the carbon-carbon band is representative of the tensile strength of cross-ply Advanced Carbon-Carbon (ACC). The upper bound represents capabilities of various experimental carbon-carbon composites. Thin carbon-carbon composites, such as would be used as TPS panels or hot aero-structure, are usually constructed of layups of 2-D fabrics of carbon-fiber yarns (tows). Although the in-plane strengths of these composites can be very attractive, a major problem area is low interlaminar strength. The low interlaminar strength is the result of a relatively weak carbon matrix and poor interaction between the fibers and matrix. The purpose of the present paper is to discuss strategies being employed to improve the interlaminar strengths of the materials at the upper bound of the carbon-carbon band, and to present some recent encouraging results. The emphasis of these strategies is to improve interlaminar shear and tensile strengths while maintaining, or even improving, the inplane properties.

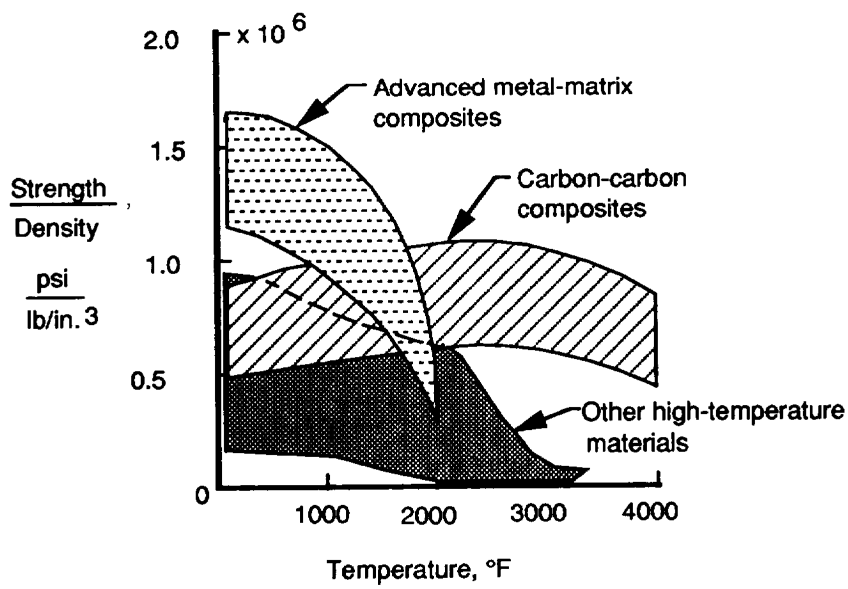


Figure 1. Specific strength versus temperature for candidate high-temperature materials

BASELINE ACC MECHANICAL PROPERTIES AND GOALS FOR IMPROVEMENT

In the 1978 to 1982 timeframe, NASA Langley sponsored the development of Advanced Carbon-Carbon (ACC) by LTV Corporation, with a major goal of obtaining a 25 percent increase in strength over that of Reinforced Carbon-Carbon (RCC), the material used on the wing-leading-edges and nose-cap of the Space Shuttle (ref. 1). Significant improvements in in-plane mechanical properties were realized with ACC, but its interlaminar strengths were actually lower than those of RCC. Even today, however, ACC largely remains the baseline to which new experimental materials are compared, and it is thusly used in this presentation. From 1982 to the present, investigators have endeavored to improve the interlaminar strengths of 2-D carbon-carbon composites over those of ACC, but improvements have not come without giving up some in-plane strength and/or stiffness (refs. 2,3). Conversely, attempts to improve in-plane properties over those of ACC have, at best, resulted in no improvement in interlaminar strengths and have usually resulted in a loss (ref. 3). Clearly, it is desirable to improve both the interlaminar and in-plane properties simultaneously. Figure 2 shows the mechanical properties of the baseline ACC-4 (the numeral 4 suffixed to ACC implies 4 densification steps after initial carbonization). The potential for improvements in 2-D carbon-carbon properties, which are believed by the authors to be achievable, are also indicated. The reference ACC property data are for 7- and 9-ply ACC-4 cross-ply panels fabricated by LTV Corporation as contract deliverables for reference 1 .

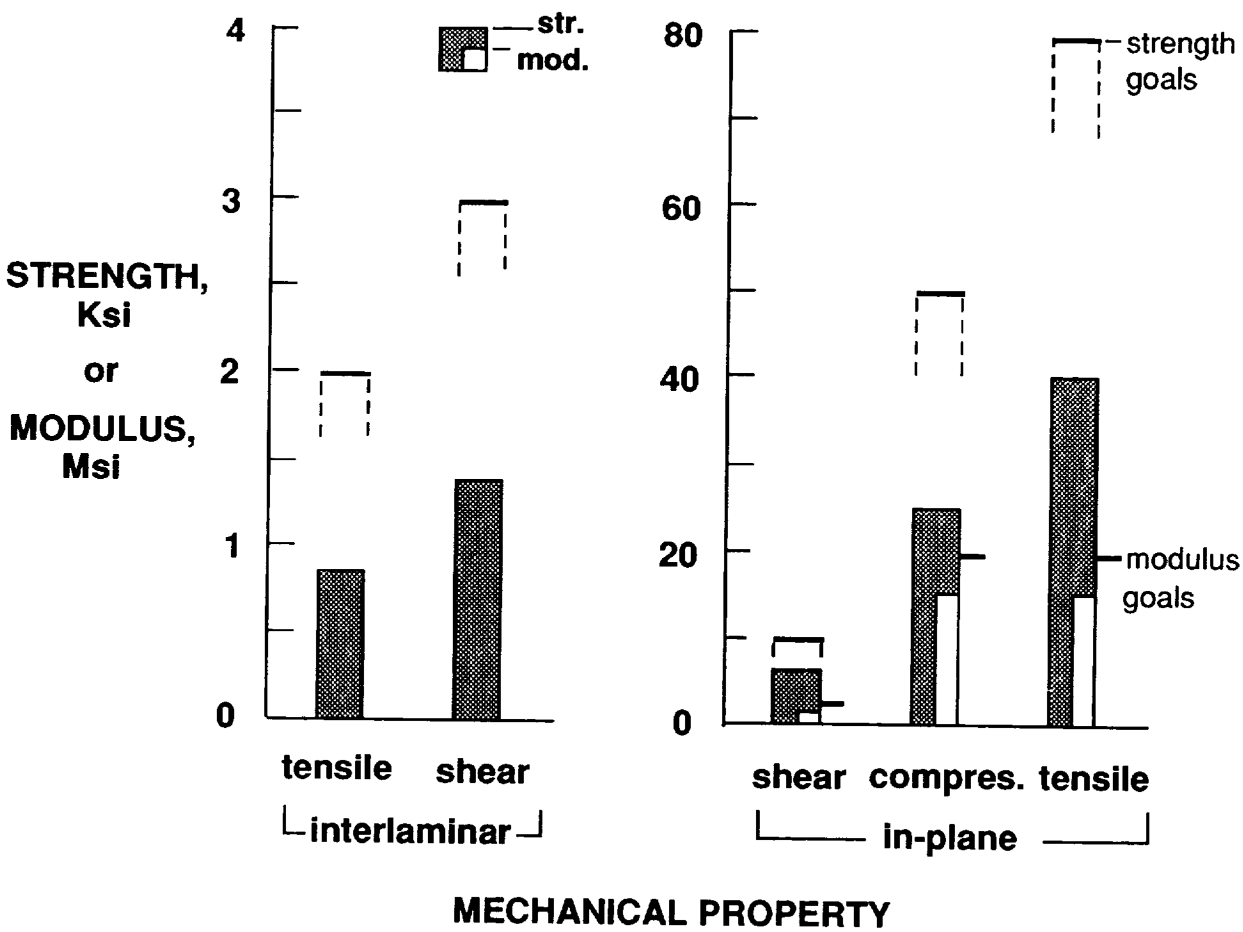


Figure 2. Baseline ACC mechanical properties and potential for improvement in 2-D carbon-carbon.

FACTORS CONTROLLING INTERLAMINAR STRENGTH IN 2-D CARBON-CARBON SUBSTRATES

The interlaminar strength of a 2-D reinforced carbon-carbon depends on many factors as listed in figure 3. The first factor, mechanical interaction of plies, has been the basis for many approaches to improving interlaminar strength in 2-D substrates. These approaches have generally involved the use of reinforcing fabrics having "rough" surfaces for the purpose of increasing fabric surface-area (for increased ply-to-ply contact) or for promoting nesting of tows or entanglement of fibers between adjacent plies. Examples of the use of such approaches can be found in references 2 and 3. The problem with these approaches, however, is that they are counterproductive to improving or even maintaining the in-plane strengths because tow strength is sacrificed. Factor 2, constituent properties, has been an area of investigation and reference 4 is an example of published results related to this area. Factor 3, fiber/matrix interaction, can have a significant influence on in-plane properties as well as interlaminar properties. Although the interlaminar strength can be significantly increased with increased surface chemical activity of the fiber, the in-plane tensile strength can be greatly diminished at appreciably lower levels of surface activity than required for maximum interlaminar strength. This research area is addressed in references 5-7. The inherent propensity for fiber-surface chemical activity may be dependent, to a large degree, on the fiber's surface microstructure (types of fiber microstructure and their effects on carbon-carbon composites are treated in ref. 8). The fiber's actual surface activity is dependent on the proprietary surface treatments given to the fiber and the post-surface-treatment thermal history of the fiber. The shape of the fiber and degree of crenulation of the surface may also affect mechanical interactions between the fibers and matrix. A discussion of the interaction of all these factors and their effects on mechanical properties of 2-D carbon-carbon can be found in reference 9.

- **MECHANICAL INTERACTION OF PLIES**
  - **DEGREE OF PLY-TO-PLY CONTACT**
  - **NESTING OF SURFACE RELIEF IN ADJACENT PLIES**
  - **ENTANGLEMENT OF FIBERS FROM ADJACENT PLIES**

- **CONSTITUENT PROPERTIES**
  - **MATRIX STRENGTH**
  - **FIBER VOLUME FRACTION**

- **FIBER / MATRIX INTERACTION**
  - **CHEMICAL**
  - **MECHANICAL**

Figure 3. Factors controlling interlaminar strength in 2-D carbon-carbon substrates.

STRATEGY FOR DEVELOPMENT OF HIGHER STRENGTH, THIN CARBON-CARBON SUBSTRATES

The NASA Langley strategy addresses the many factors listed in figure 4. The 8 harness-satin weave construction, which is used in the baseline ACC, is retained to minimize the frequency of fiber crimping. High frequencies of crimping are not conducive to good in-plane strength. Only continuous-filament tows are considered for the highest possible translation of fiber strength to the composite. A different approach to increaseing mechanical contact between plies is being used in lieu of some approaches involving high-relief or "fuzzy" fabrics (refs. 2,3). It is reasoned that reductions in fabric thickness and tow size will increase the surface-to-volume ratio of the tows so that fiber-to-fiber contact across plies is increased. Also, the reduced fabric thickness reduces the crimp angle of the tows and should be beneficial to in-plane properties. Furthermore, the reduction of fabric thickness and/or tow size reduces the repeating unit dimensions in the composite, which should result in a redistribution and refinement of shrinkage damage that occurs during initial carbonization of the polymer matrix. This refinement and redistribution is expected to be beneficial to interlaminar strength. Combining these potential benefits with the selection of a fiber having the best mechanical properties, surface chemistry, and cross-section shape, and embedment of the optimum volume fraction of these fibers in the best-performing matrix should go a long way toward maximizing both the interlaminar and in-plane mechanical properties of the the 2-D composite. Through-the-thickness reinforcement is also being explored for applications where interlaminar strength requirements may exceed the potential of 2-D composite.

- **HARNESS-SATIN WEAVES (AS OPPOSED TO PLAIN WEAVES)**
- **CONTINUOUS FILAMENT TOWS (AS OPPOSED TO STAPLE TOWS)**
- **REDUCED TOW SIZE / FABRIC THICKNESS**
- **FIBER TYPE**
- **FIBER SURFACE CHEMISTRY**
- **FIBER CROSS-SECTION SHAPE**
- **MATRIX**
    - **TYPE OF INITIAL MATRIX**
    - **TYPE OF DENSIFICATION MATRIX**
    - **VOLUME FRACTION**
- **THROUGH-THE-THICKNESS REINFORCEMENTS**
    - **WOVEN 3-D**
    - **STITCHED 2-D**

Figure 4. Strategy for development of higher strength, thin carbon-carbon substrates.

BENEFITS OF FINE-TOW, THIN FABRIC AND CVI DENSIFICATION ON MECHANICAL PROPERTIES OF 2-D CARBON-CARBON SUBSTRATE

A 2-D substrate was fabricated using a fine-tow, thin fabric constructed of T-300 fibers, the same fiber as used in ACC. The initial processing of the test material was similar to that used in fabrication of ACC, but densification was accomplished by CVI. The resulting composite was found to have 40 to 60 percent higher interlaminar strengths than for the ACC to which it is compared in figure 5. In-plane shear strength was improved by about 30 percent. The compressive and tensile strengths were improved by about 60 percent. Moduli are slightly lower than for ACC because the volume fraction of fibers in the fine-tow material was somewhat lower than that in ACC. The target volume fraction, although not as high as in ACC, was not achieved in this first attempt at making the fine-tow composite. It is anticipated that a fine-tow composite of the optimum volume fraction for CVI densification would have higher moduli without adversely affecting the strengths. It can also be seen from figure 5 that significant progress has been made toward achieving the program goals for strength improvement.

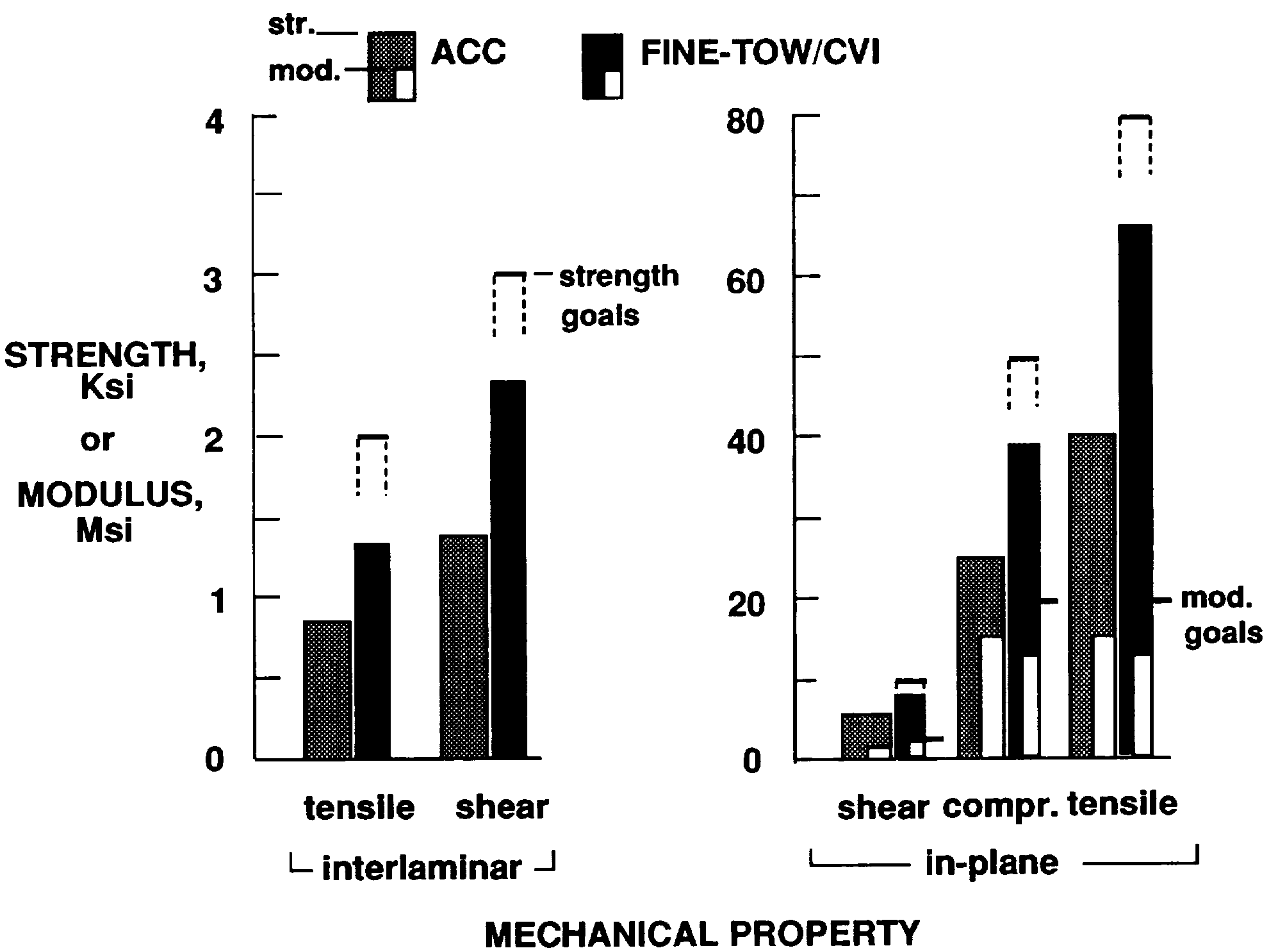


Figure 5. Benefits of fine-tow, thin fabric and CVI densification on mechanical properties of 2-D carbon-carbon substrate.

The improvements in strength shown in figure 5 were obtained using T-300 fiber which had been heat-stabilized to a temperature above the expected use temperature of the carbon-carbon (C-C) composite. This temperature is high enough to remove the manufacturer's surface treatment (which is applied to the fiber to increase chemical bonding of the fiber and matrix in resin matrix composites). The influence of fiber surface treatment on fiber-matrix bonding in carbon-carbon composites has been investigated, and the results presented in the next several figures indicate the potential for this approach and also suggest limitations.

Since fiber surface treatment and fiber sizing can influence the nature of the interaction between the fibers and matrix, they can exert an important influence on the mechanical properties of the material. Investigations have been carried out to determine the importance and potential utility of these factors in determining the mechanical properties of phenolic-based C-C composites.

Fiber surface treatment studies have been conducted for a high strength (Hercules AS-4) and a high modulus (Hercules HM) fiber. The general relationships between the degree of fiber surface treatment and the resultant C-C mechanical properties have been consistent (refs. 5-7). Unidirectional composites have been used for experimental purposes, and the results obtained with Hercules HM fibers are typical. Figure 6 shows the interlaminar shear strengths obtained from C-C composites incorporating unsized HM fibers subjected by the manufacturer to different levels of surface treatment, here quantified by fiber surface oxygen concentration. It can be seen that the interlaminar shear strength can be controlled over a wide range, and the overall trend observed for all fibers investigated to date is for interlaminar tensile strength as well as interlaminar shear strength to increase with the degree of fiber surface treatment.

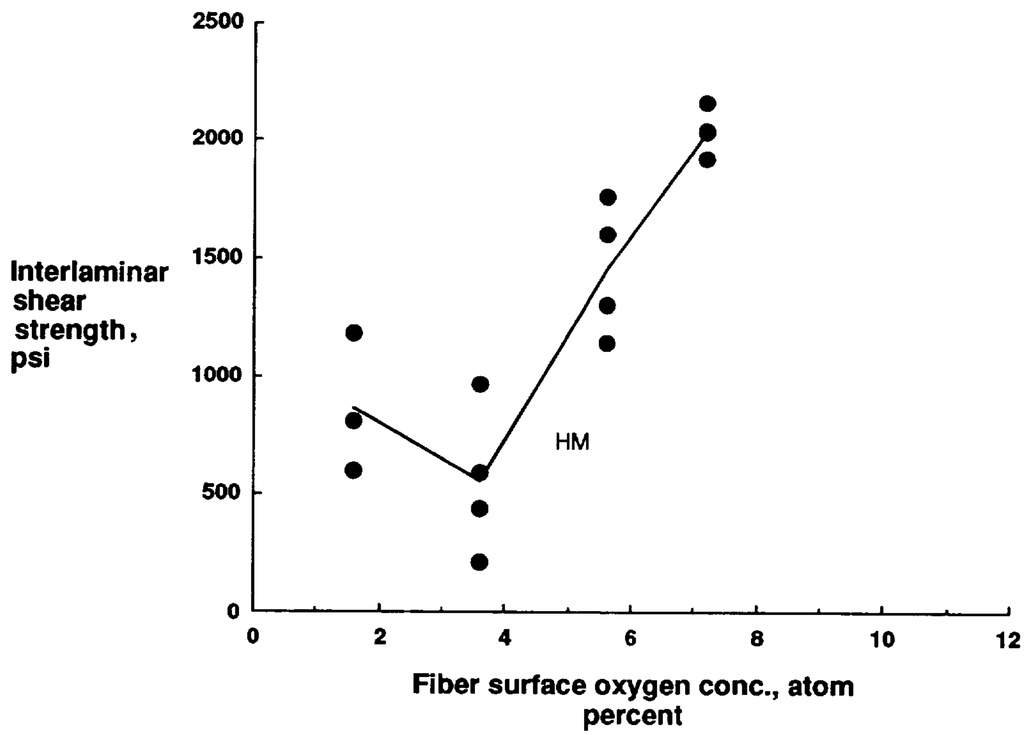


Figure 6. Relationship between interlaminar shear strength and fiber surface oxygen concentration for surface-treated Hercules HM fibers.

INFLUENCE OF FIBER SURFACE TREATMENT AND SIZING ON THE MECHANICAL PROPERTIES OF CARBON-CARBON COMPOSITES (CONT'D)

Different fiber surface treatment levels can produce differences in fiber-matrix interaction which are readily visible under magnification. Shown in figure 7 are scanning electron micrographs of polished sections of C-C composites incorporating HM fibers treated to 0, 50, and 150 percent of the commercial standard level. The corresponding fiber surface oxygen concentrations are 1.6, 3.6, and 7.2 atom percent, respectively. At the 0 percent treatment level, the undensified composite (top row) has numerous fiber-matrix gaps caused by matrix shrinkage during the initial pyrolysis. Note that as the fiber surface treatment level increases, the undensified composites exhibit relatively fewer gaps and also less distinct interfaces between the fibers and matrix. This difference is evident for both undensified and densified composites.

Top row: undensified
Bottom row: densified

Fiber surface treatment level:

0 Percent	50 Percent	150 Percent

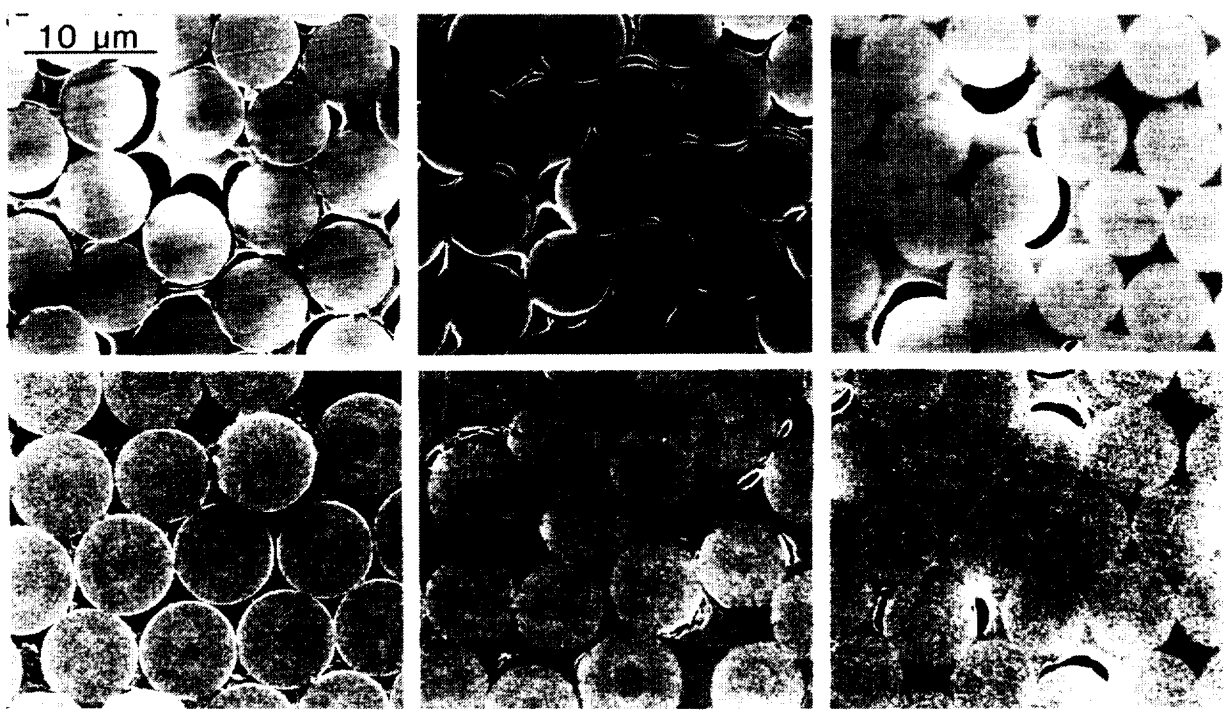


Figure 7. Fiber-matrix interactions in unidirectional composites with HM fibers treated to 0, 50, and 150 percent of standard treatment (fiber surface oxygen concentrations of 1.6, 3.6, and 7.2 atom percent, respectively).

Although increased C-C interlaminar strengths are desireable, the effect of increased fiber-matrix interaction on axial mechanical properties dictates that the degree of fiber surface treatment be chosen with some care. Figure 8 shows the axial tensile and compressive strengths for the unidirectional HM-fiber composites. For low to moderate fiber surface oxygen concentrations, composite tensile strength is little affected, but for high concentrations, a rapid decrease in strength occurs. On the other hand, compressive strength continually increase as the degree of fiber surface treatment is increased. Elastic modulus, in either tension or compression, is relatively unaffected by the degree of fiber surface treatment.

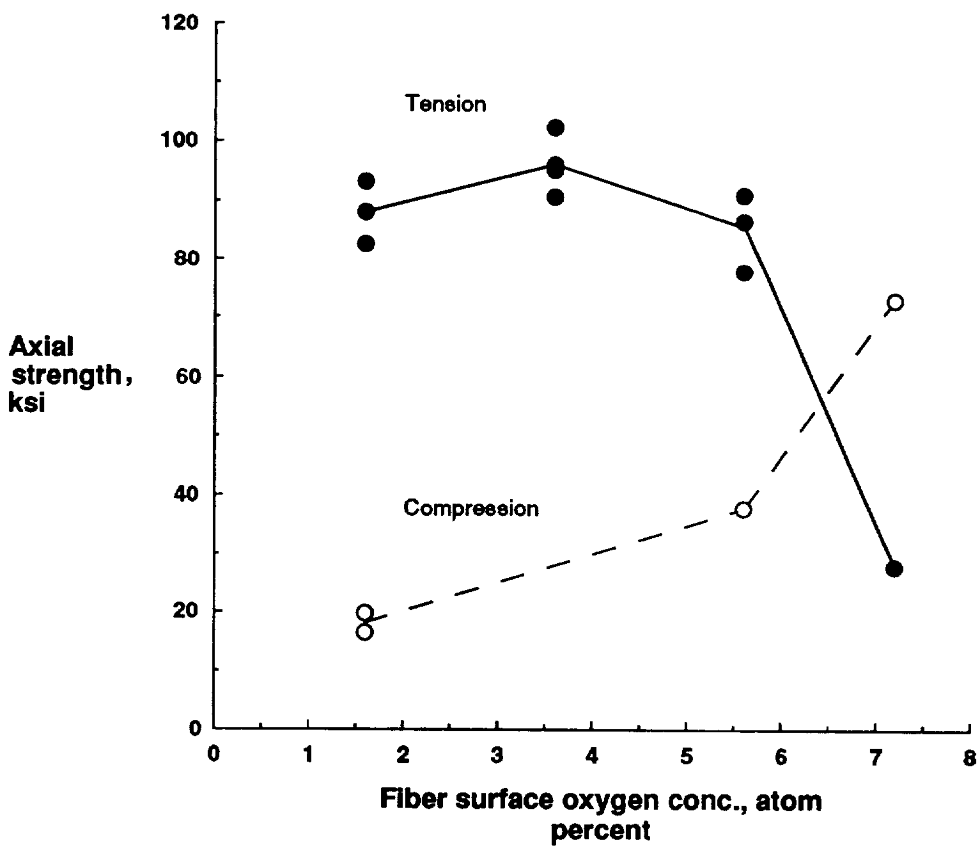


Figure 8. Relationship between axial tensile strength, compressive strength, and fiber surface oxygen concentration for surface treated Hercules HM fibers.

INFLUENCE OF FIBER SURFACE TREATMENT AND SIZING ON THE MECHANICAL PROPERTIES OF CARBON-CARBON COMPOSITES (CONT'D)

The influence of fiber surface treatment on the mechanical behavior of C-C composites is also apparent upon examining the tensile failure modes, as shown in figure 9. Note that as the fiber surface oxygen concentration increases, the decrease in tensile strength is accompanied by a change in failure mode from a fibrous one (with extensive fiber pull-out) to a brittle one (with little or no pull-out). Simultaneously, however, the interlaminar shear strength and compressive strength of the composite is increasing. This leads to the conclusion that, while a wide range of properties is possible by varying fiber surface treatment level, there is no one treatment level at which all mechanical properties are maximized, and some intermediate treatment levels are indicated when a balanced complement of properties is desired.

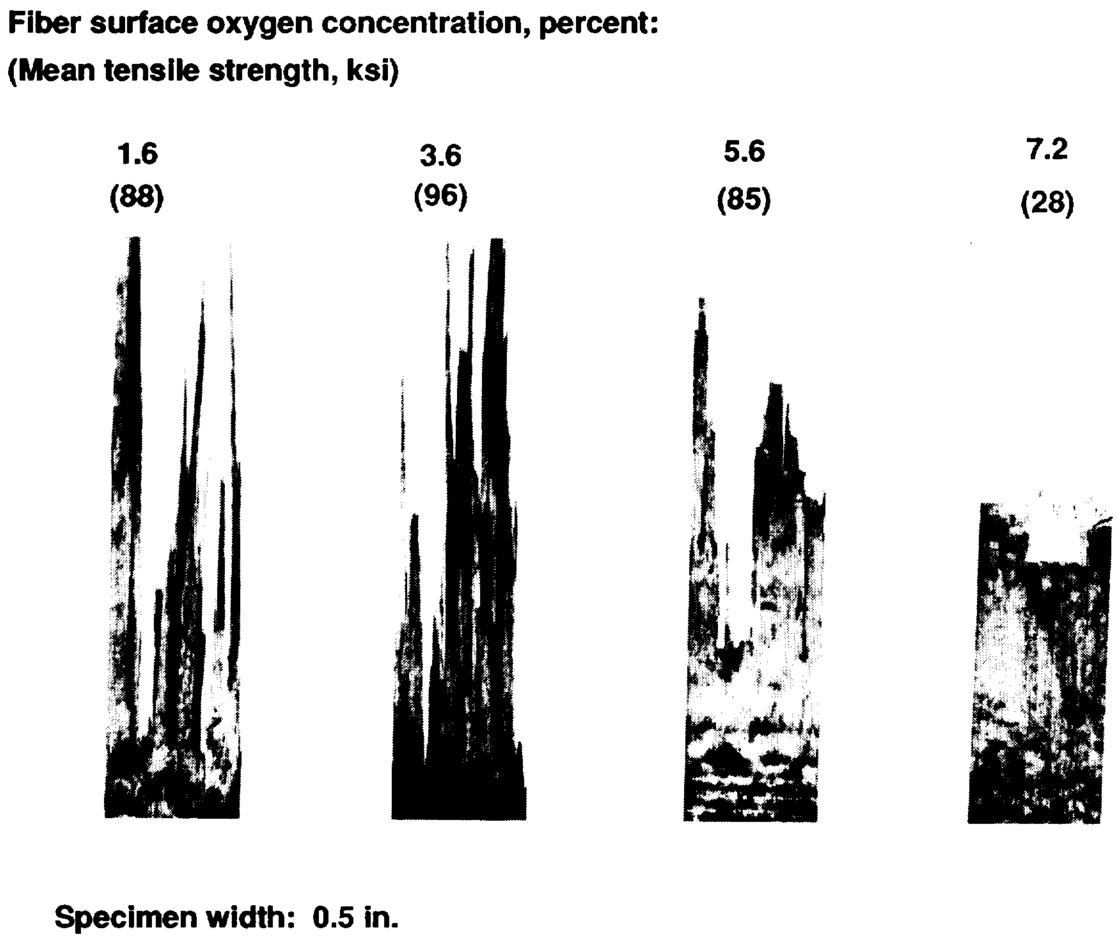


Figure 9. Tensile failure modes of unidirectional C-C composites with surface treated Hercules HM fibers.

The most common form of reinforcement in structural C-C composites is woven fabric. In order to weave carbon fiber tow, some type of fiber sizing is generally required for handleability and durability, and this sizing, like fiber surface treatment, can influence fiber-matrix interaction. However, sizings for carbon fibers have been developed primarily for organic matrices, and these sizings are often intended for epoxy resins. Such resins have low char yields–on the order of ten percent, as opposed to phenolic resins with yields on the order of 60 percent.

Therefore, a study was conducted to determine whether it would be advantageous to employ a high-char sizing for the fabrication of C-C composites. Again, Hercules HM fiber was employed for the fabrication of unidirectional specimens, and four variants of this fiber were compared. The variants comprised combinations of surface-treated (HMS) and untreated (HMU) fibers with either phenolic resin (Ph) sizing or a Hercules epoxy-compatible (G) sizing. Figure 10 illustrates that, on a given fiber, the G sizing resulted in a higher interlaminar shear strength, and therefore greater fiber-matrix interaction, than the phenolic sizing; however, the presence or absence of fiber surface treatment (HMS vs. HMU) was a more influential factor in determining the interlaminar shear strength of the composites. The HMU-Ph composite possessed such a low level of fiber-matrix interaction that the composite delaminated during processing and was unusable for testing.

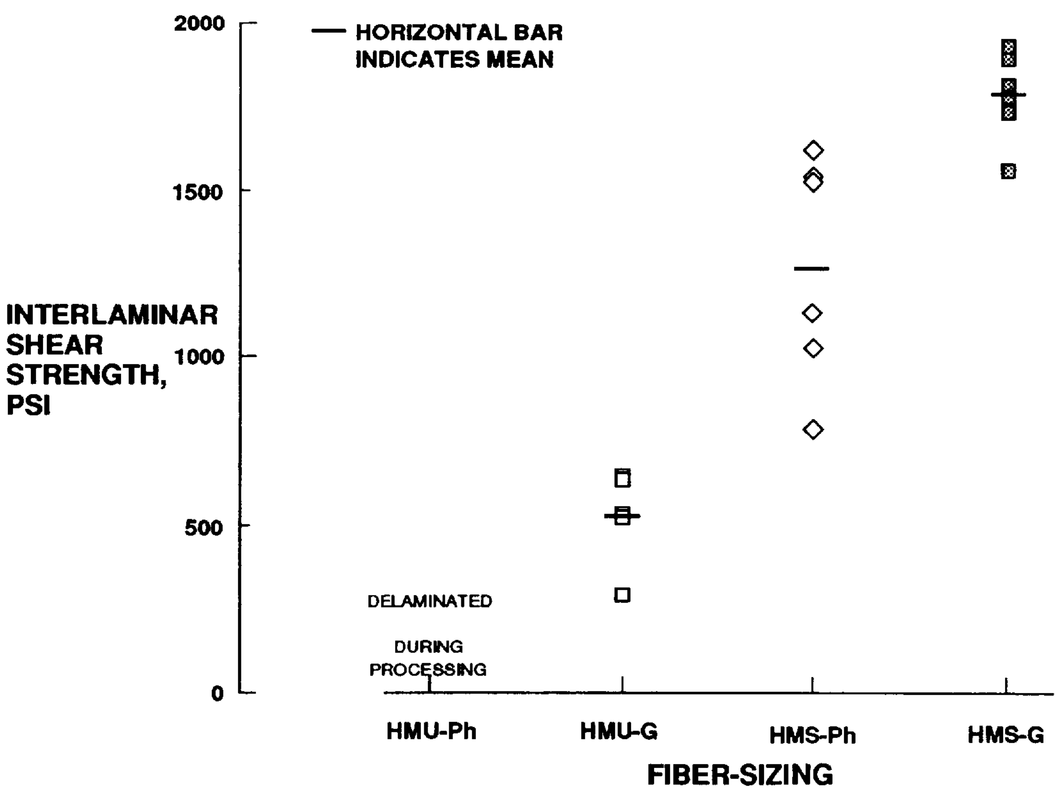


Figure 10. Interlaminar shear strengths for HMU-Ph, HMU-G, HMS-Ph, and HMS-G composites.

Figure 11 shows the compressive strengths for the same composites as in figure 10. It can be seen that the relative ranking of compressive strengths is the same as that for the interlaminar shear strength. This is not unexpected since, as mentioned previously, a higher degree of fiber-matrix interaction results in greater resistance not only to interlaminar fracture, but also to compressive fracture. While fiber surface treatment was found to be a more powerful influence on mechanical properties, another important conclusion of the investigation of sizing effects is that because the epoxy-compatible "G" sizing was not detrimental to performance, it is not considered necessary to employ specialty sizings for fabricating phenolic-based, fabric-reinforced C-C composites. However, this is not to say that the choice of sizing is inconsequential. Since the specific nature of sizing materials is generally held proprietary by each fiber manufacturer, there is no assurance that other sizings will perform similarly to the Hercules "G" sizing.

For fabrics woven from surface-treated fibers, the influences of both fiber surface treatment and sizing on mechanical properties need to be taken into account. Any attempt to "de-size" surface-treated, woven fabric will probably not be totally complete, and may well affect the underlying fiber surface treatment also.

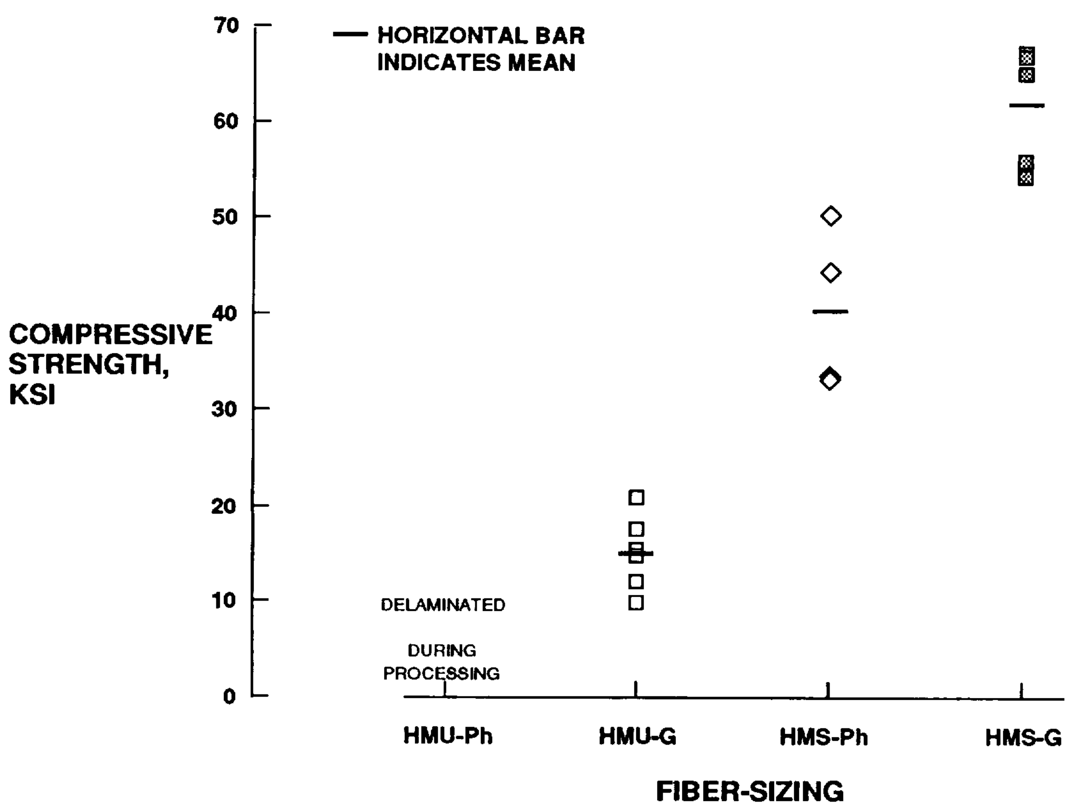
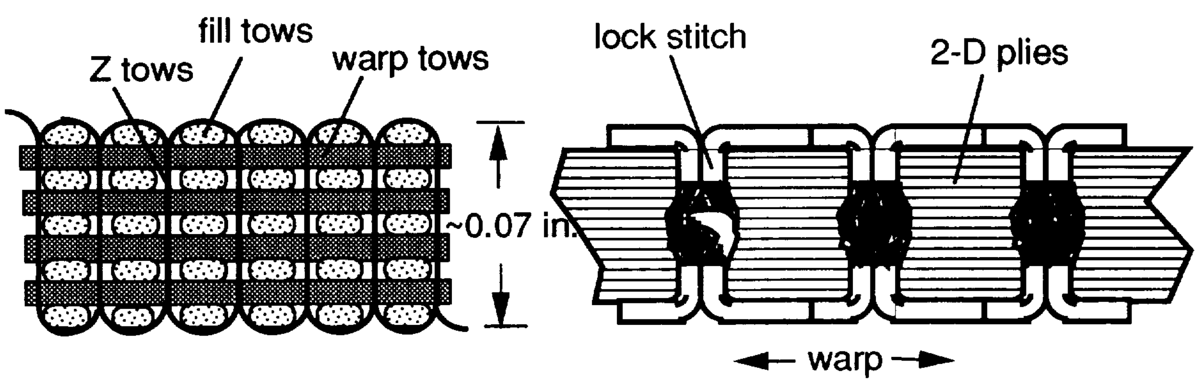


Figure 11. Compressive strengths for HMU-Ph, HMU-G, HMS-Ph, and HMS-G composites.

THROUGH-THE-THICKNESS REINFORCEMENT CONCEPTS FOR THIN CARBON-CARBON SUBSTRATES

The program goals shown in figure 2 for improving mechanical properties are believed to be attainable for 2-D reinforced substrates. For a particular application, the need may well arise for interlaminar strengths not attainable by 2-D substrates. Utilization of through-the-thickness (Z-direction) reinforcement is an approach to be considered in this situation. For such an approach, NASA Langley has evaluated thin woven 3-D orthogonal preforms, as in the example shown in figure 12a, and stitched layups of 2-D fabric, as in the example shown in figure 12b. Using these approaches, interlaminar strengths have been achieved which exceed the program goals. Unfortunately, these reinforcement constructions give rise to inherently lower volume fractions of fibers in the in-plane directions and in-plane stiffness suffers.

Although these types of reinforcements have the disadvantages of lower stiffness as well as poor drapability (preforms are relatively rigid), their use affords potential advantages. For example, the 3-D orthogonal construction could be used to produce preforms for integrally stiffened panels, or the stitching technique could be used to selectively reinforce 2-D panels near stiffeners and flanges where particularly high interlaminar strengths are required.



a. 3-D orthogonal construction

b. stitched 2-D construction

Figure 12. Through-the-thickness reinforcement concepts for thin carbon-carbon substrates.

STRENGTH BENEFITS OF A CVI-DENSIFIED 3-D ORTHOGONAL CARBON-CARBON COMPOSITE

Figure 13 compares the mechanical properties of a CVI-densified, 3-D orthogonal-reinforced carbon-carbon composite to those of the baseline ACC. The interlaminar tensile strength is well over twice that of the ACC and, in addition, exceeds the 2-D strength goal by some indeterminable amount (the 3-D strength exceeds the test capability, as indicated by the arrow at the top of the bar). The interlaminar shear strength exceeds the 2-D strength goal by as much as 50 percent and exceeds ACC strength by over 200 percent. The 3-D substrate has considerably more in-plane tensile and compressive strength than the ACC, but its tensile and compressive moduli are lower. The moduli are lower because of the much lower volume fraction of fiber in the 3-D composite (~43% compared to ~60% for ACC). The strengths remain high because the warp and fill tows are straight in the orthogonal construction; only the z-tows are weavers. Encouragingly, the in-plane shear strength and modulus of the 3-D composite remain comparable to ACC properties, in spite of the low volume fraction of fibers.

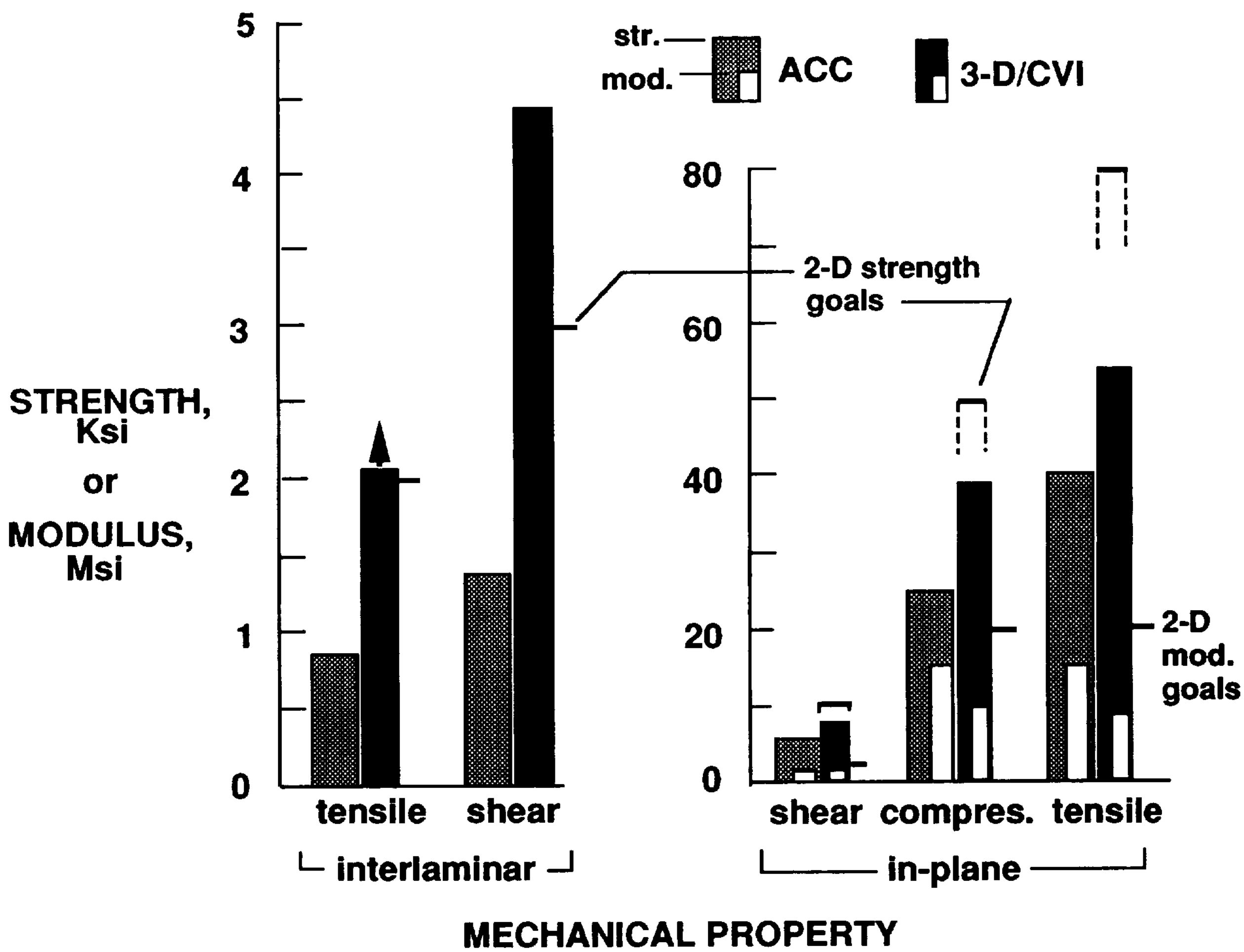


Figure 13. Strength benefits of a CVI-densified 3-D orthogonal carbon-carbon composite.

STITCHING AS INTERLAMINAR REINFORCEMENT IN THIN CARBON-CARBON COMPOSITES

Stitching of fabric lay-ups was investigated as a means of providing interlaminar reinforcement in C-C composites (ref. 10). Nine-ply lay-ups of dry Thornel T-300 fabric were reinforced by stitching with either two-strand or three-strand Torayca T-900 carbon thread in various stitch patterns with six to twelve stitches per inch in the x-direction and six to twelve stitch rows per in. in the y-direction. The resulting preforms, with interlaminar reinforcement densities ranging from 60,000 to 360,000 fibers per square inch, were processed into C-C composites using phenolic resin as the densifying agent. Mechanical testing indicated that stitching could provide markedly improved interlaminar properties in comparison with unstitched reinforcement; however, axial compressive strength was degraded.

In figure 14, unstitched, stitched, and three-dimensionally woven reinforcements are compared on the basis of interlaminar shear strength obtained from double-notch specimen tests. At a reinforcement density of approximately 300,000 fibers per square inch, it can be seen that the two-strand stitching furnishes a shear strength 50 percent higher than unstitched material, and that it is also more effective than the three-strand stitching at equivalent reinforcement densities. In comparison with the three-dimensionally woven Celion material, the two-strand stitching is able to achieve an approximately equivalent strengh at a lower reinforcement density. Meaningful interlaminar tensile strength data could not be obtained for the stitched materials due to the inability of the existing test method to effectively transfer load to the reinforcing stitches; however, the interlaminar tensile strength for the stitched material is likely much higher than that for the unstitched material.

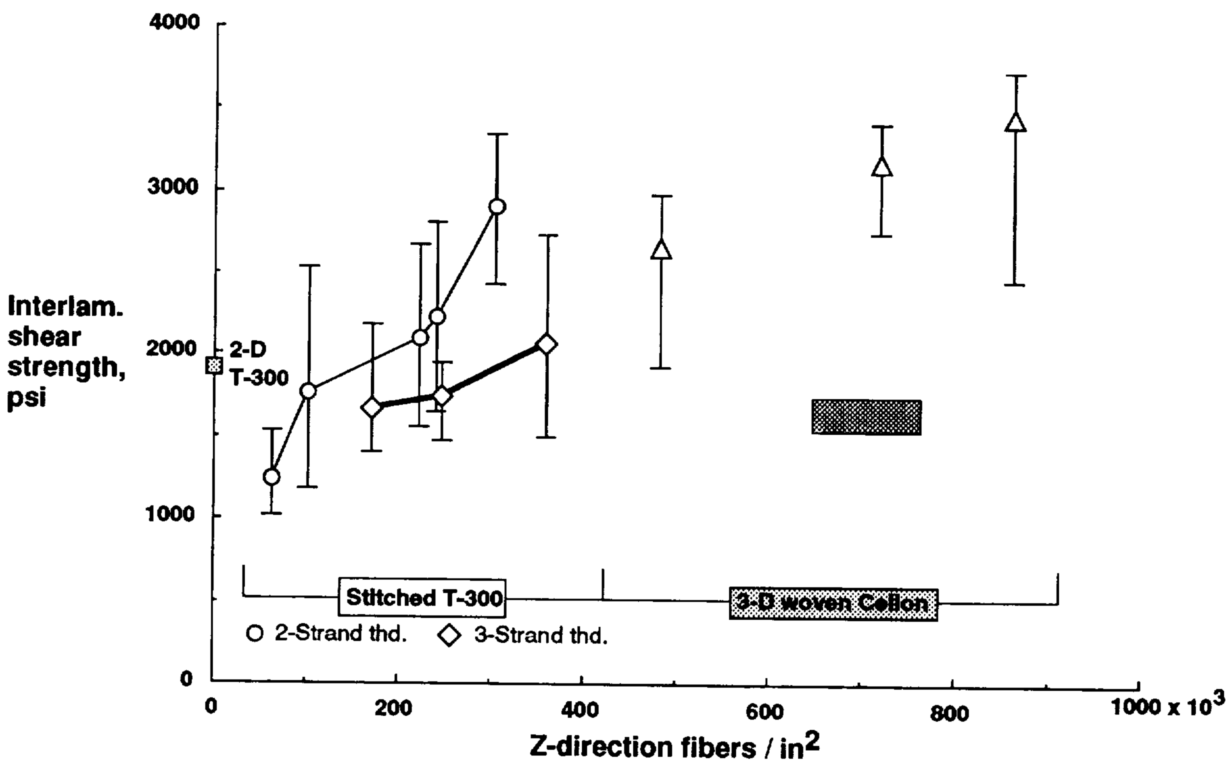


Figure 14. Comparison of interlaminar shear strengths for C-C composites reinforced with unstitched T-300 fabric, stitched T-300 fabric, and 3-D woven Celion fabric.

While stitching did markedly improve the interlaminar properties of fabric-reinforced C-C, it did produce degradation in the axial compressive strength of the material. Axial strengths are compared for unstitched, stitched, and three-dimensionally woven materials in figure 15. It can be seen that the stitched T-300 and 3-D woven Celion materials furnished approximately 60 to 65 percent of the compressive strength of the unstitched T-300 reinforcement. Tensile strength, however, suffered little or no degradation as a result of the stitching, and the stitched materials possessed approximately the same tensile strength as the three-dimensionally woven materials.

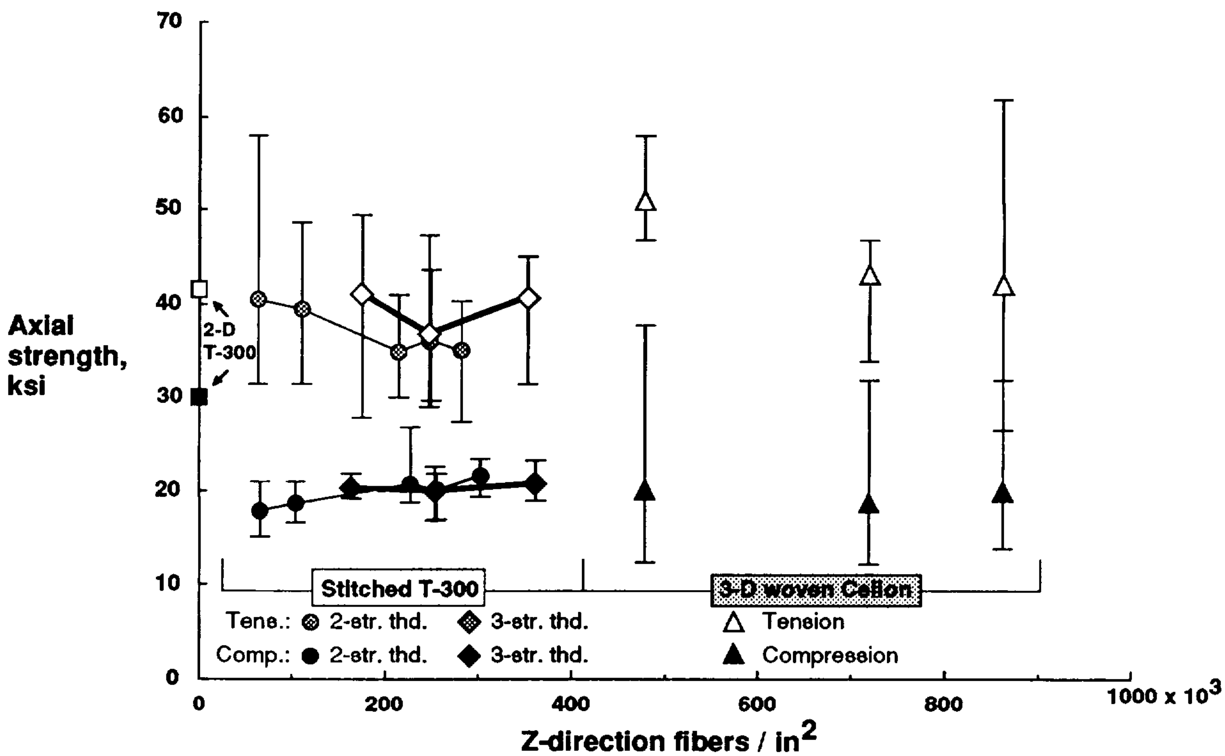


Figure 15. Comparison of tensile and compressive strengths for C-C composites reinforced with unstitched T-300 fabric, stitched T-300 fabric, and 3-D woven Celion fabric.

The axial moduli resulting from the unstitched, stitched, and three-dimensionally woven reinforcements are shown in figure 16. Not surprisingly, the data show that stiffness in either tension or compression tends to decrease as the proportion of z-direction reinforcement increases and the proportion of in-plane reinforcement correspondingly decreases.

In summary, it can be stated that stitching can produce marked improvements in the interlaminar properties of two-dimensional, fabric-reinforced C-C composites, accompanied by some degradation in compressive strength and both tensile and compressive moduli. Stitching is a reinforcement technique which lends itself to those particular applications driven by a need for high interlaminar properties; however, refinements such as optimized stitch patterns and thread weights could yield an improved balance of interlaminar and axial mechanical properties. In addition, stitching is a technique which affords considerable flexibility during fabrication since it can be applied to selected areas of structural components.

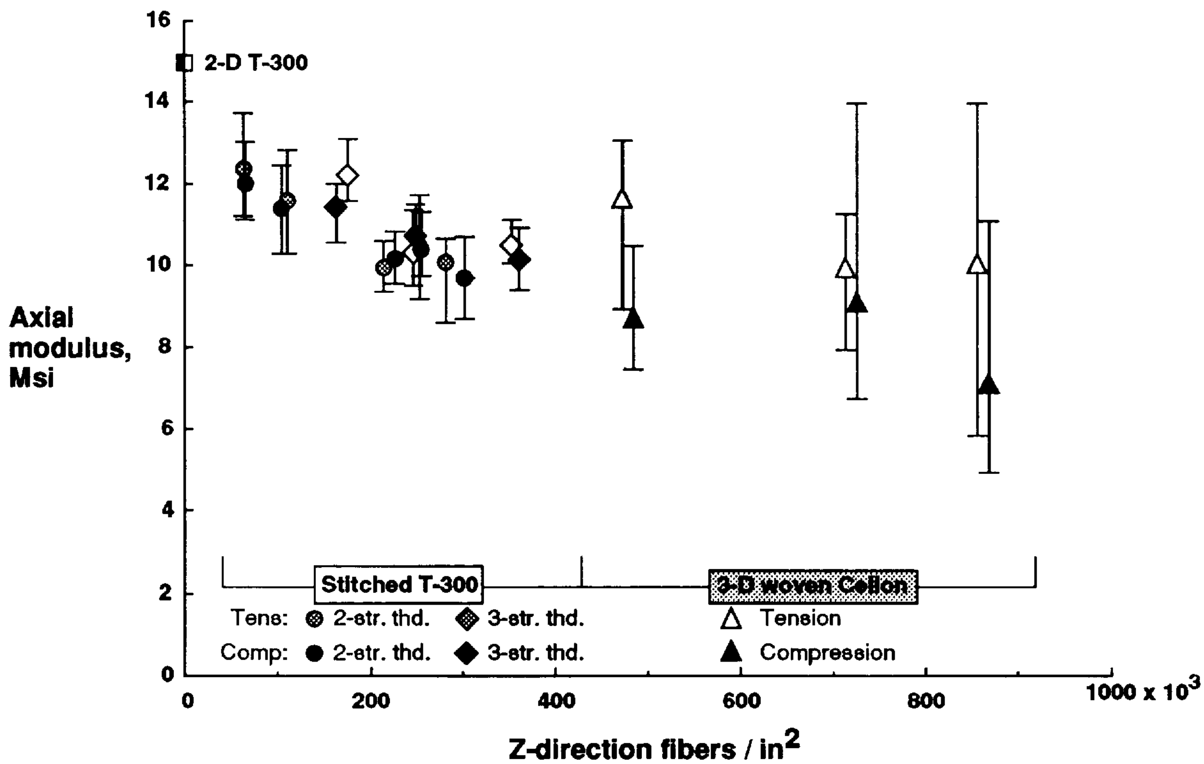


Figure 16. Comparison of tensile and compressive moduli for C-C composites reinforced with 2-D T-300 fabric, stitched T-300 fabric, and 3-D woven Celion fabric.

SUMMARY AND CONCLUSIONS

Thus far it has been demonstrated in this program that interlaminar strength of 2-D carbon-carbon can be significantly increased without having to give up in-plane strength. In fact, the use of the thin, fine-tow fabric improves in-plane strengths as well as interlaminar strengths. The improvements demonstrated thus far, for 2-D composites, have been achieved using reinforcements on which the fiber surface chemistry has mostly been removed as an unavoidable byproduct of fiber stabilization by heat-treatments. The use of newer high-strength, high-modulus fibers, which do not require heat stabilization, presents an opportunity to further improve interlaminar properties through optimization of fiber surface chemistry. Much higher percentages of improvement in interlaminar strengths are achieved by using 3-D orthogonal reinforcement; however, the use of through-the-thickness reinforcement, whether in the form of the 3-D orthogonal construction or the stitched 2-D construction, necessitates a tradeoff in stiffness (and in-plane compressive strength, depending on the type of densification matrix used). When the 2-D reinforcement is used in conjunction with CVI densification, interlaminar strengths are improved by 40 to 60 percent over baseline. At the same time, the in-plane tensile and compressive strengths are improved by 60 percent or better. When the 3-D orthogonal reinforcement is used in conjunction with CVI densification, interlaminar strengths are improved by 100 to 200 percent. The in-plane strengths are better than those of baseline ACC, including compressive strength. It is anticipated that similar results would be obtained if the 2-D stitched reinforcement were used in conjunction with CVI densification.

- **FINE TOW / THIN FABRIC COMBINATION SIMULTANEOUSLY IMPROVES IN-PLANE AND INTERLAMINAR STRENGTHS IN 2-D COMPOSITES**
- **OPTIMIZING FIBER SURFACE TREATMENT / SIZING AFFORDS HIGH POTENTIAL FOR IMPROVING INTERLAMINAR STRENGTHS**
- **THROUGH-THE-THICKNESS REINFORCEMENT NECESSITATES PROPERTY TRADE-OFFS**
- **SIGNIFICANT INCREASES ACHIEVED IN INTERLAMINAR STRENGTHS RELATIVE TO BASELINE:**

	TENSILE	SHEAR
2-D	40%	60%
3-D	100%	200%

Figure 17. Summary and conclusions

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