Matrix/Fiber Interface Effects on Kevlar 49[®] Pressure Vessel Performance

N.A. Mumford,* P.C. Hopkins,† and B.A. Lloyd‡

Morton Thiokol/Wasatch Division,

Brigham City, Utah

Introduction

THE use of Kevlar 49® in lightweight filament wound rocket motor cases is now state-of-the-art for strategic missile and space motor applications. The fiber stress performance of Kevlar 49® in pressure vessels is considerably lower than the nominal strand tensile strength. Utilization of a higher fraction of Kevlar fiber's ultimate strength in a biaxial load field has been demonstrated in two ways: the use of DC-20§ release coating on the fiber¹-³ or the use of a semiflexible resin matrix.⁴ It is proposed that both of these approaches act to limit the transverse loading of the fiber and/or alter the failure mechanism to allow higher tensile performance.

Scope and Method of Approach

Variations in resin and resin/fiber interface properties were achieved in several ways.

- 1) Chemical—Resins from three generic classes were used: a rigid epoxy-anhydride, a semiflexible epoxy-aromatic amine, and a film-adhesive-type rubber-toughened epoxylatent amine. These resins, through differences in functional groups, polarity, viscosity, and mechanical properties, provided composites with a wide range of properties.
- 2) Physical—Broad physical property differences were provided by the major changes in resin formulations referred to in item 1. Finer changes in physical properties were achieved by conducting tests over a temperature range. The distinct advantage here is that all other chemical and processing parameters of the composite are constant.
- 3) Fiber release—The employment of a silicone fiber release finish resulted in a low, but controlled interfacial bond strength.
- 4) Fiber only—A vessel with resinless hoops wound over cured polars was used as a control with no fiber/fiber coupling.

The effects of these factors on fiber stress performance in 5.75-in. pressure vessels and on short beam shear strength were determined. Resin content, fiber volume, and void content are a function of the manufacturing process, which was essentially the same for all pressure vessels. Some variation resulted from the different resin properties and could have had an effect on short beam shear strength. However, processing conditions are similar to those found in the actual winding of rocket motor cases, and good correlation between subscale and full-scale bottle/case tests has been demonstrated.

Experimental

Aerospace grade Kevlar 49® used in all tests was dried for more than 16 h at 250°F immediately prior to resin ap-

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*Associate Scientist, Materials Department.

†Senior Engineer, Filament Winding Materials Section.

‡Supervisor, Filament Winding Materials Section.

§Reference to a product name does not imply approval or recommendation to the exclusion of others.

plication. Resins are identified as follows:

- 1) rigid—thiokol UF-3283 preimpregnated on Kevlar 49®;
- 2) semiflexible—Thiokol UFX80-29 (Hercules HBRF-241) wet wound:
- 3) rubber toughened—American Cyanamid BXP 31075 preimpregnated on Kevlar 49®:
- 4) Silicone mold release—Dow Corning-20 applied to the fiber from a solvent bath to yield a 5-9% nonvolatile finish.

The fabrication and hydrotest of the 5.75-in. pressure vessels were in accordance with ASTM D2585. Fiber stresses reported are average values calculated from burst pressures by netting analysis at a stress ratio of 0.851. Apparent horizontal (NOL) short beam shear strength was determined by the ASTM D2344 method.

Results and Discussion

The fiber stress performance of Kevlar 49® in the pressure vessel mode and the short beam shear strength for the different matrix and interface conditions at 70°F are shown in Table. 1. There is a range of fiber performance from 51 to 77% of the nominal impregnated strand tensile strength. Proposed mechanisms for explaining the variations in fiber tensile strength performance in pressure vessels are: 1) transverse fiber load conditions, 2) shear load transfer for failed fiber filaments, and 3) composite failure mode differences. All of these mechanisms are interrelated and separation for observation is difficult. However, in this study, variations in the matrix material systems were used to emphasize one or more of the proposed mechanisms. It has been extensively observed in our research that the apparent fiber tensile strength decreases when loaded in the biaxial stress field.⁵ This is especially true of Kevlar 49®, which is highly anisotropic. The transverse loading of the fiber in Kevlar

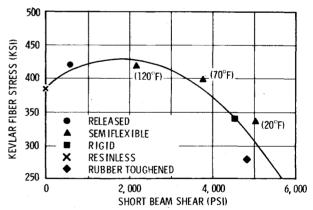


Fig. 1 High shear strength decreases Kevlar 49® fiber performance.

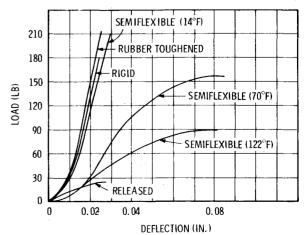


Fig. 2 Short beam shear dependence on matrix/interface properties.

Table 1 Kevlar 49° fiber stress and short beam shear strength at 70°Fa

Matrix	Fiber stress, ^b ksi	Coefficient of variation, %	No. of tests	Percent strand tensile,	Shear strength, psi (percent variation)
Nominal impregnated strand tensile	545	(2.81) ^a	2036	100	
Rubber-toughened epoxy	279	(2.7)	12	51	4840 (3.6)
Rigid epoxy	341	(3.9)	75	63	4550 (4.2)
Semiflexible epoxy	399	(2.8)	. 17	73	3770 (3.0)
Rigid epoxy released fiber	421	(3.9)	37	77	580 (5.7)
None	385	(0.3)	3	71	

^a Data per Du Pont tests. ^b Stress ratio = 0.851.

composites is dependent on both matrix/fiber interfacial bond strengths and resin mechanical properties. The short beam shear strength gives an indication of the strength of the interface bond. Thus a "rough" correlation of the fiber stress performance with the shear strength can be shown as in Fig. 1. The load/deflection curves of the short beam shear tests shown in Fig. 2 explain, in part, why this correlation is not 100%. Although the ultimate composite "shear" strength of the semiflexible system is approximately the same as the rigid system at 70°F, at a given deflection prior to failure the load is considerably lower. Thus the semiflexible resin, with a modulus of 50 ksi relative to the rigid resin with a modulus of 400 ksi, reduces the transverse loading of the fiber, allowing higher longitudinal tensile performance. For further discussion, see Ref. 6.

In addition to transverse loading effects, improved fiber performance in the semiflexible system may result from a change in failure mode. In the rigid system, the energy of a filament failure can be transferred by the brittle elastic matrix and propagates failure through adjacent filaments, whereas the tougher plastic properties and/or poorer fiber/matrix bonding characteristics of the semiflexible system act as an energy absorber or change the failure mode from through the adjacent filaments to along the filament-to-matrix interface. These effects would tend to isolate filament failures, allow shear load transfer, and result in later overall failure at higher loads. ¹

The rubber-toughened epoxy resin was designed to absorb the energy of individual filament failures like the semiflexible system and act as a crack arrester to prevent transverse crack propagation. Apparently the effects of transverse loading due to a high interfacial bond strength were dominant, and this strong adhesive yielded the lowest Kevlar 49® performance.

The silicone released fiber performed the highest because of the combination of low interfacial bond strength to limit transverse loading, energy absorbing failure around individual filaments rather than through, and sufficient shear strength to redistribute the load of failed filaments to the surrounding composite. In the test with no matrix resin, transverse loading and filament failure propagation by the matrix are eliminated. In addition, very little shear transfer of load from failed filaments can occur. Table 1 shows these pressure vessels delivered fiber tensile strengths less than the rigid-released matrix systems, but greater than the rigid and rubber-toughened epoxy matrices. The shear load transfer effect is also observed in strand tests where dry strands test lower than impregnated strands and nontwisted strands test lower than twisted strands.

The effects of specific variations in the mechanical properties of the rigid and semiflexible resins and, hence, variations in the fiber/resin interface were investigated by conducting the vessel hydroburst at either subambient or elevated temperatures. The results of these tests are shown in Fig. 3, where the fiber performance is plotted as a function of temperature.

The slope of the rigid and semiflexible curves reflects the temperature dependence of the resin mechanical properties. This dependence is much greater for the semiflexible resin

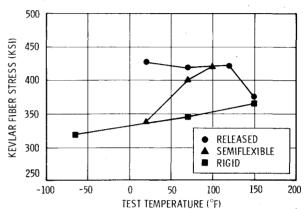


Fig. 3 Kevlar 49® performance reflected in temperature sensitivity of matrix

than for the rigid resin. Figure 1 shows the relationship of composite shear strength to pressure vessel tensile fiber strength performance for the semiflexible resin matrix with Kevlar 49[®]. This gives a clear picture of the effects of matrix and interfacial properties unencumbered by variations introduced by resin formulation or processing changes. It also indicates that improved performance in pressure vessel tensile strength results at the expense of structural properties.

The released fiber system further emphasizes the latter point. Figure 3 indicates this system is fairly independent of temperature except at elevated temperatures, where apparently the silicone releases too well and a situation similar to the matrix-free condition arises and strengths approach that level.

Conclusions

It was determined from these studies that matrix resin systems which form good structural Kevlar 49® composites can decrease tensile strength performance up to 48% of the reported strand tensile strength values. Mechanisms proposed to account for this strength reduction include transverse loading effects, shear load transfer of failed fiber filaments, and differences in composite failure modes. The use of semiflexible resin systems or fiber prefinish release coatings can result in significant pressure vessel tensile strength increases, but at the expense of structural properties. For motor case applications, structural and temperature/moisture conditions must be considered carefully before selecting a high-performance matrix system for Kevlar 49®. Where low composite shear strengths are acceptable, special matrix resins or fiber prefinishes can and are employed in the industry which increase tensile strength performance to within approximately 20% of strand values.

References

¹Hughes, A.A., "Design of a High Performance Kevlar® Epoxy Rocket Motor Case for the MX Upper Stage Program," 1976