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NOTE

New Method for Evaluation of Fiber/Matrix Adhesion†

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KEY WORDS Fiber/matrix adhesion; sandwich composite; short beam shear; phenolics; steel wool; interlaminar shear.

INTRODUCTION

There are several well-known methods for evaluating the level of fiber-to-matrix adhesion. For example, the single filament pull out test is the most direct way of measuring the level of the debonding strength. However, the method is not always applicable because of fiber brittleness or the geometry and/or size of the fibers. Incorporation of fibers in a fiber/matrix composite and arranging the composite failure in such a way that fiber/matrix debonding is the dominant failure mode is another well-known method for evaluating the interfacial adhesion level. The short beam shear method, popular because of convenience, is an example of this class of methods.

Recently, we came across a case where neither approach could be applied directly. The specific case involved evaluating the level of the debonding strength between fibers and matrix in a chopped-steel-wool-reinforced phenolic matrix. Because of the extreme irregularity of the steel wool filaments, the single filament pull out

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method is not reliable. Continuous steel wool filaments are, in addition, curled and entangled. As such, the preparation of a well-consolidated composite with a high fiber content represents a formidable task.

Chopped steel wool reinforcement, the particular material of interest to us, is commonly used to produce consolidated composites. This type of composite, with a high fiber volume content, is especially desirable because it increases the role of the fiber-matrix interface in the failure process. However, even with short beam testing arrangements such composites fail because of normal tensile stresses in the cross-section. The proposed solution was a modification of the short beam shear method.

In order to circumvent this problem and insure that the locus of failure is concentrated at the metal-phenolic interface we prepared the following type of test specimens: the composite testing sample was molded as a symmetrical three-ply sandwich. The core ply was heavily reinforced with chopped steel wool filaments and two external plies were unidirectionally reinforced with continuous glass or carbon fibers (Figure 1).

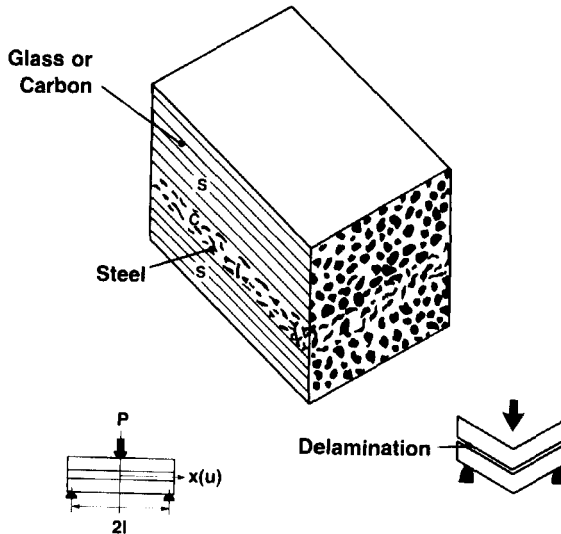


FIGURE 1 Sandwich specimen in 3-point bending.

EXPERIMENTAL

Specimen preparation was designed to characterize adhesion between chopped steel wool and a novolac phenolic resin. Continuous glass or carbon fibers were incorporated in the skins of the sandwich composite. The molding of the sandwich was performed in a single step in order to avoid weakening of the skin/core interface.

The sandwich preparation consisted of the following steps. A solution of phenolic in ethanol (100 g of phenolic powder in 100 ml of ethanol) was prepared. The glass fiber (or carbon fiber) was cut into 3-inch-long strands which were unidirectionally arranged in two 3×3 -inch assemblies, 7 g each. These plies, or skins were thoroughly impregnated by the solution. From 2 to 8 g of chopped steel filaments were uniformly distributed on one of the skins; then the second skin was used to cover the first one. The final package was molded in a 3×3 -inch mold at pressures of 500 psi, and a temperature of 150°C . Total molding time was 20 min.

Control specimens were simple unidirectional composites reinforced with the glass or carbon strands (10 grams per sample) without incorporation of chopped steel. These composites were molded in a similar way and had approximately the same thickness as the sandwich composites.

Specimens about 1 inch long and $3/8$ inches wide were cut and tested in an Instron machine with a span-to-thickness ratio of 4:1. The cross head speed was 0.05 in/min.

RESULTS

The results of representative experiments with carbon and glass external plies and varying steel core thickness are listed in Table I. The failure shear stress was calculated according to the standard method for an isotropic and homogeneous material.

The short beam shear strengths obtained with homogeneous, unidirectionally-reinforced composites of the skin construction (the controls) with glass and carbon reinforcements were 7300 psi and 10,000 psi respectively.

The failed specimens were examined by scanning electron microscopy. Figure 2 shows a representative electron micrograph of a

TABLE I
Short beam shear strength of sandwich composite with chopped steel reinforced core

| Sample | Fiber in the outer plies | Fiber ^a content (g) | Steel content (g) | Matrix content (g) | Short beam ^b shear strength (psi) |
|-----------------------------|--------------------------|--------------------------------|-------------------|--------------------|--|
| 1 | carbon | 7 | 4 | 3.3 | 6600 |
| 2 | carbon | 7 | 4 | 8.0 | 5600 |
| 3 | carbon | 7 | 6 | 10.5 | 6000 |
| 4 | carbon | 7 | 6 | 10.4 | 6700 |
| average for carbon sandwich | | | | | 6225 |
| 5 | glass | 7 | 2 | 3.3 | 4800 |
| 6 | glass | 7 | 4 | 2.2 | 5000 |
| 7 | glass | 7 | 6 | 7.0 | 5600 |
| 8 | glass | 7 | 8 | 7.9 | 4400 |
| average for glass sandwich | | | | | 4950 |

^a Per one layer.

^b Average 8 specimens per sample; stand dev ± 300 psi.

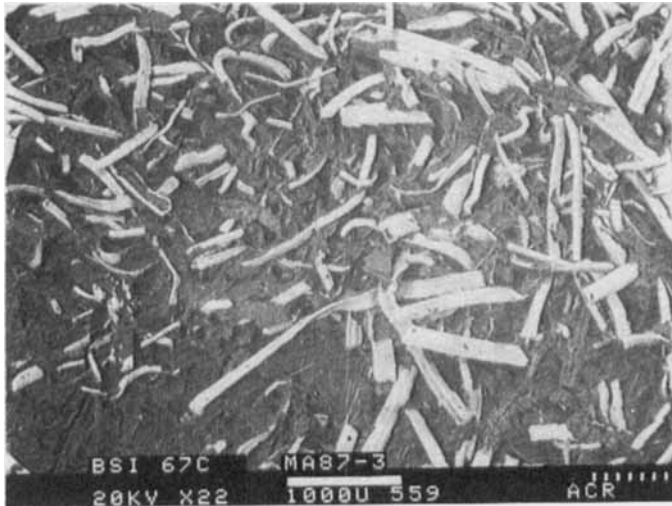


FIGURE 2 SEM of a failure surface.

failed zone at the steel wool filament/phenolic interface. The steel wool filaments appear to have little, if any, residual adhering matrix material. This observation suggests that the dominant failure mode was debonding at the steel wool filament/phenolic interface.

DISCUSSION

Comparison of the data in Table I to the control (homogeneous glass or carbon), and the fact that the shear failure occurs through the central ply, indicate that the present approach can be used for inducing shear failure in the central layer of the sandwich composite. This information can be used in turn to characterize the relative level of adhesive strength between the fiber and the matrix of the central layer. The method can be used to show the effect of fiber surface modification on the strength of the sandwich composite.¹

This technique is also useful for other situations. For example, when the amount of new experimental fiber is not sufficient to make a regular unidirectional composite. In this case a thin layer of the composite reinforced by the experimental fiber can be clad in skins unidirectionally reinforced with available fibers. Another situation is the case of fibers with low compressive strength. If the skins are reinforced with fiber of a significantly higher compressive strength, the probability of compressive failure in the process of bending is significantly reduced. In general, strong skins to a certain degree protect the core composite from the immediate effect of stress concentration at the points of loading. This reduces the probability of its failure from loading modes other than shear.

There are consistent differences (about 25%) in the values for the shear stress at failure between the sandwich composite with carbon and glass skins. These variations are not important if the same skin construction is used for a comparative analysis of a given core reinforcing fiber with and without different surface modification. However, these differences in the value for the shear stress at failure between glass and carbon can be important for the case where fiber or matrices of significantly different rigidities are compared.

On a basic level, the problem can be handled by the theory of laminated plates.² Consider the effect of two symmetrical skins on

shear stress in the skin/core interface in a sandwich (Figure 1) subjected to three-point flex with a span-to-height ratio $L/H = 4-5$. The skins and the core are considered to be perfectly elastic orthotropic materials.

One can write

$$\tau_c = P(E_s S_{sx} + E_c S_{cx}^*) / 2(E_s I_s + E_c I_c) \quad (1)$$

where P is the force; S_{sx} , S_{cx}^* are the static moments of one skin and the respective part of the core; I_s , I_c are the moments of inertia of one skin and the core; E_s , E_c are Young's moduli of the skins and the core (hereafter the subscripts s and c will be used to denote skin and core respectively).

Young's moduli of the skins and core, E , can be estimated³ from

$$E = E_f V_f + E_m (1 - V_f) \quad (2)$$

where V_f is the fiber volume content, and E_f and E_m are Young's moduli for the fiber and matrix. We assume that the thickness of individual skins and the core are equal and that $V_f = 0.6$. The subscripts 1 and 2 are used to denote the cases where both skins are glass or carbon fiber respectively. Typical experimental moduli are: $E_{f1} = 10 \times 10^6$ psi; $E_{f2} = 30 \times 10^6$ psi; the modulus of the matrix, E_m , is 0.5×10^6 psi; the modulus for the steel fibers, E_{fc} is 30×10^6 psi; from (2) above $E_{s2} = 18.2 \times 10^6$ psi; $E_c = 18.2 \times 10^6$ psi. However, we assume that the core modulus, E_c , is 9.1×10^6 psi or one-half the value calculated according to (2), in order to account for the random orientation of the steel wool fiber.

Application of formula (1) for our example predicts a level of shear stress that is only 9% higher in the case of glass-reinforced skins, as compared to the case of carbon-reinforced skins. A larger difference (about 14%) can be obtained if we assume that the core modulus, E_c , is 18.2×10^6 psi, as for the case for unidirectional orientation of fiber in the core. Also note that the effect of the skins is more pronounced for a thicker core. However, we believe that our assumptions on geometry and rigidities of the sandwich components are correct, and therefore formula (1), though reflecting the tendency correctly (a higher level of stresses in the case of the glass-clad sandwich corresponds to a lower level of apparent stresses of failure) underestimates the effect of the skins (which is ~25% in our case) on the failure stresses.

Methods similar to those described in References 3 and 5 for temperature-influenced interfacial strength where the shear rigidities of the composite plies are considered may possibly account for the observed differences.

CONCLUSIONS

The proposed method can be used for evaluation of the level of adhesion between fibers and matrix in composites where: a) a regular arrangement of fibers in the composite poses a problem; b) the amount of fiber is not sufficient to prepare a regular composite specimen; c) for the case of fibers with low compressive strength.

The theory of laminated plates underestimates the effect of rigid skins on shear stresses in a composite sandwich. A more thorough model is needed for analysis of shear stresses in these types of composites.

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