

Interlaminar Shear Properties of Some Glass-Fiber-Reinforced Phenolic and Polyester Laminates

A. KAMINSKI,* *British Rail Research and Development Division, Railway Technical Centre, Derby, United Kingdom*

Synopsis

Chopped strand mat and glass-fabric-reinforced thin and thick laminates were made from phenolic and polyester laminating resins using three modifications of a hand lay-up method. Short-beam interlaminar shear strengths were determined in three-point bending at a span-to-depth ratio 3:1. Plane and perpendicular specimens were used. It was found that the perpendicular specimens were more useful. Interlaminar delamination energies were determined statically and dynamically. It was found that the results for the phenolic and polyester laminates cannot be simply compared because of differences in the fracture mechanism.

INTRODUCTION

Despite the use of fire-retardant additives provided to improve the fire properties of polyester laminates used in the construction of British Railways rolling stock they are still combustible. Further, the use of fire retardants frequently increases the amount of smoke evolved in a fire.¹ More stringent regulations in public transport systems create a demand for more fire-resistant plastic materials. A new generation of phenolic resins has been recently developed² for the production of glass-fiber-reinforced laminates using similar processing techniques as for polyester laminates.³ Flammability and smoke generation of these resins are reported to be improved.⁴ As they are used for the production of structural items their mechanical properties should be characterized.

Interlaminar shear properties of advanced composites, e.g., carbon-fiber-reinforced plastics, have been widely investigated.⁵ This has led to a subsequent improvement of the interlaminar shear strength of these materials from 20 up to 100 MPa.⁶ On the other hand, there is a complete lack of information for new phenolic laminates and little published data for standard polyester laminates which cannot be compared because of use of different test methods by different authors.

Chiao et al.⁷ evaluated some interlaminar shear test methods for glass-reinforced polyester (GRP) and found that the results depended critically on the test method used because it is difficult to obtain a state of pure shear. Methods, like torsional shear of a thin-walled tube or the ten-degree off-axis tensile test developed by Chamis and Sinclair⁸ are expensive and present some practical difficulties. The three-point bending short-beam test method described by Ewins⁹ seems to be the most advantageous method for measurement of the interlaminar shear strength.

* Present address: Centralny Osrodek Baden i Rozwoju Techniki Kolejnictwa, Warsaw, Poland.

Static and dynamic method for measurement of the interlaminar shear delamination energy based on easy to produce triple-notched specimens were described by Kimpara and Takehana.¹⁰ The static variant of the method is performed on Instron-type testing equipment and the dynamic variant requires the use of a Charpy impact machine. The results of static tests are always lower than the dynamic ones. After an analysis of both variants it is suggested that the difference could be related to the kinetic energy of the broken specimens leaving the Charpy impact machine.

It was also thought to be worthwhile to compare the results of the short-beam tests with the static and dynamic measurements of the delamination energy. It was especially interesting because the structural laminate items of the railway rolling stock can be subjected in use to the static, dynamic, and impact stresses. Glass reinforcements more or less compatible with the phenolic resins were chosen for investigation because it was thought that the adhesion between glass and resin could be of importance as far as interlaminar shear properties are concerned.

It should be noted that until now no commercially available chopped strand mats have been found fully compatible with the phenolic resins.¹¹ Sizes and binders used must effect the condensation reaction mechanism of the resins and produce laminates having poorer mechanical properties than expected.¹² The development of suitable reinforcements is reported by the phenolic resins producers.

MATERIALS

The phenolic resin used in this investigation was a low-viscosity resin Norsophen 1201 (CdF Chimie, France via KWR Chemicals Ltd., U.K.) and manufactured in 1980. The resin was cured with two different acid catalyst systems as supplied: 12% of a standard-grade aqueous solution of catalyst C 1650 and 17% of a nonaqueous solution of a boron compound fire-retardant additive containing catalyst C 2101 "Aniphen."

The polyester resin used was a fire-retardant resin Crystic 323 (Scott Bader Co., U.K.). The resin was cured with 2% MEKP, supplied by Laporte Industries Ltd., U.K. and 2% cobalt octoate.

The polyester gel coat was Gel 39 (Scott Bader Co.). The following glass reinforcements were used for the preparation of phenolic and polyester laminate samples:

- (a) a powder bound chopped strand mat with a silane finish, type 902 (Owens Corning Fiberglass, Belgium), which has been found to be one of the most compatible with the phenolic resins presently available¹³;
- (b) a heat-cleaned plain weave fabric type Y 449/165 (Fothergill and Harvey Ltd., U.K.), which was thought to be compatible with the phenolic resin because of lack of a finish.

Both types of glass were obviously well compatible with the polyester resin. Exact proportions of each resin and reinforcement system used are given in Table I.

Three modifications of a hand lay-up laminating technique were used for the production of the samples reinforced with the chopped strand mat (CSM); 1, brush laminating; 2, brush and roller laminating; 3, brush laminating followed

TABLE I
Evaluated Phenolic and Polyester Laminate Systems

System	Material	Type	Amount/sample (g)	
			Thin samples	Thick samples
1	Phenolic resin	Norsophen 1201	350	1200
	Catalyst	C 1650	42	140
	Reinforcement	OCF 902 (400 g/m ²)	120	420
2	Polyester resin	Crystic 323	350	1200
	Initiator	MEKP	7	24
	Accelerator	Cobalt	7	24
	Reinforcement	As system 1	As system 1	As system 1
3	Phenolic resin	As system 1	As system 1	As system 1
	Catalyst	C 2101 "Aniphen"	60	200
	Reinforcement	As system 1	As system 1	As system 1
4	Phenolic resin	As system 1	...	As system 1
	Catalyst	As system 1	...	As system 1
	Reinforcement	Fabric Y449/165	...	800
5	Polyester resin	As system 2	...	As system 2
	Initiator	As system 2	...	As system 2
	Accelerator	As system 2	...	As system 2
	Reinforcement	As system 4	...	As system 4

by closing the mold with a top panel and squeezing in a plain screw press (pressure about 0.1 MPa).

The method of manufacture of the laminates is identified in the figures and tables by these numbers. All unlabeled results were taken on samples prepared using laminating technique No. 2.

Simple plywood molds with a melamine formaldehyde surface finish were used for the production of two types of samples (3 and 15 mm thick) called "thin" and "thick" samples, respectively.

The thin phenolic laminate samples were prepared with phenolic primer and polyester gel coat 39. The polyester laminate samples and the thick phenolic laminate samples were prepared without any surface finish. All phenolic laminate samples were cured in an oven for 2 h at 80°C. All polyester samples were cured at ambient temperature and then postcured for 2 h at 80°C. Test specimens were cut from the laminate samples by means of a diamond dust covered wheel saw with water cooling. The notches in the delamination energy specimens were machined by means of a specially designed rig.

Two different groups of short-beam test specimens were cut from the thick laminate samples. The first group, called "plane specimens," was identical in structure to the specimens cut from the thin laminate samples, with the reinforcement in layers in the plane of the specimen (see Fig. 1). The other group was cut so that the reinforcement layers were located perpendicularly to the specimen's plane (see Fig. 1). All the specimens were stored for one month at room temperature before testing.

METHOD OF TESTING

Barcol hardness, oxygen index, and flexural strength measurements were performed to the British Standards BS 2782: Part 10: Method 1001: Part 1:

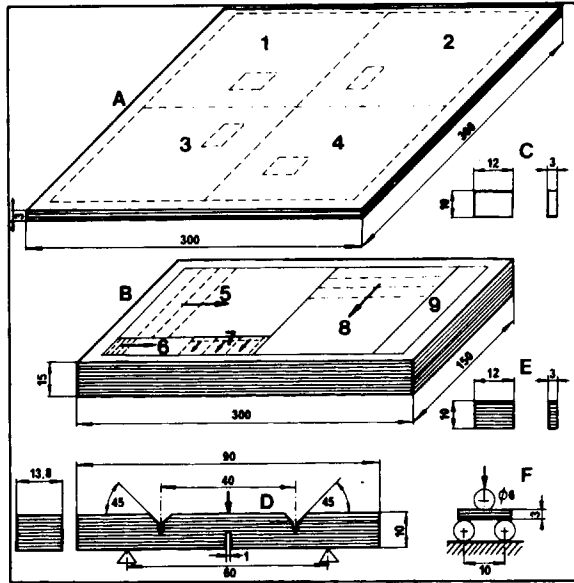


Fig. 1. Laminate samples and specimens for interlaminar shear measurements. A, Thin laminate sample: 1, 2, short-beam specimens tested with surface finish up; 3, 4, specimens tested with surface finish down. B, Thick laminate sample: 5, 8, specimens as shown on D; 9, specimens as shown on C; 6, 7, specimens as shown on E. C, Standard short-beam specimens. D, Delineation energy specimens. E, Perpendicular short-beam specimens. F, Short-beam load tool.

Method 141C : 1978, and Part 1 : Method 1005 : 1977, respectively. Density was estimated by weighing and measuring.

Glass content was evaluated as ash content after 3 h at 650°C. The interlaminar shear strength measurements were carried out by using the short beam three-point method described by Ewins.⁹ The loading edges were 6mm diameter as shown in Figure 1(F) and the span-to-depth ratio was 3:1. The tests were performed using an Instron with a crosshead speed of 20 mm/min.

The interlaminar shear strengths were calculated from the simple relationship:

$$R_s = 3P/2hl \quad (1)$$

where R_s is the interlaminar shear strength, P is the breaking force applied, and h and l are the specimen's width and length, respectively. The method used to determine the static and dynamic interlaminar delamination energies was based on three-point loading with a 60 mm span of triple-notched specimen as described by Kimpara and Takehana.¹⁰ The static tests were performed using an Instron machine with a crosshead speed of 10 mm/min, dynamic tests were carried out using a Charpy impact machine having a capacity of 2 J.

The kinetic energy of a specimen after break in the Charpy impact machine was calculated from the relationship:

$$E = 1/4 ms^2g/h \quad (2)$$

where E is the kinetic energy, m is a specimen mass, s is a distance covered by

the broken specimen, g is the acceleration of gravity, and h is the height fallen.

Each value appearing in any figure and table of this paper is an average of tests of at least 15 specimens cut from at least three separately prepared laminate samples. All outlying values were rejected by Dixon's test before estimation of the means.¹⁴ The confidence limits were calculated according to the formula described by Volk.¹⁵

RESULTS

General Properties

The results of measurements of glass content, Barcol hardness, oxygen index, and flexural strength of all evaluated laminate systems are shown in Tables II and III.

Shear Strength

The comparison between the behavior of the phenolic and polyester specimens cut from the thin laminate samples and plane specimens cut from the thick laminate samples is presented as a stress/strain diagram in Figure 2. Statistical distribution of the interlaminar shear strength results as measured with the specimens cut from the thin laminate samples is shown in Figure 3. All individual results are plotted and stresses calculated from eq. (1).

Statistical distribution of the results of the transverse shear strength of the system 1 phenolic laminates is compared in Figure 4 to the theoretical histogram for the normal distribution.

The calculated average values and 95% confidence limits for the interlaminar shear strength measurements with the plane specimens are shown in the form of histograms in Figure 5 and with the perpendicular specimens in Figure 6.

Delamination Energy

Delamination energy in static tests was measured from the area under the stress/strain curve. The comparison of results and 95% confidence limits for the static and dynamic interlaminar delamination energy for all tested laminate

TABLE II
Properties of Thin Laminates Used for Interlaminar Shear Measurements

Property	System 1	System 2
Glass content (% m/m)	34.5	32.4
Barcol hardness (934-1)	34.1	57.5
Density (Mg/m ³)	1.228	1.455
Oxygen index (%)	47	30
Flexural strength (MPa)	172	189

TABLE III
Properties of Thick Laminates Used for Interlaminar Shear Measurements

Property	System 1			System 2			System 3			System 4			System 5					
	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3	No. 1	No. 2	No. 3
Glass content (% m/m)	34.9	36.2	36.4	29.6	32.9	34.9	29.6	32.9	34.9	39.7 ^a	59.0	47.4	59.0	59.0	39.7 ^a	59.0	59.0	47.4
Barcol hardness 934-1	29.0	28.6	28.3	55.5	58.4	58.7	55.5	58.4	58.7	20.1	28.2	58.8	28.2	28.2	20.1	28.2	28.2	58.8
935	76.9	77.8	77.6	90.4	92.0	91.7	90.4	92.0	91.7	70.2	75.5	92.3	75.5	75.5	70.2	75.5	75.5	92.3
Density (Mg/m ³)	1.206	1.264	1.231	1.470	1.442	1.467	1.470	1.442	1.467	1.188	1.407	1.600	1.407	1.407	1.188	1.407	1.407	1.600
Oxygen index (%)	63	63	63	30	30	30	30	30	30	77	63	32	63	63	77	63	63	32
Flexural strength (MPa)	130	153	148	179	174	198	179	174	198	126	123	240	123	123	126	123	123	240

^a Boron compound fire-retardant additive is included as glass content was measured as ash content.

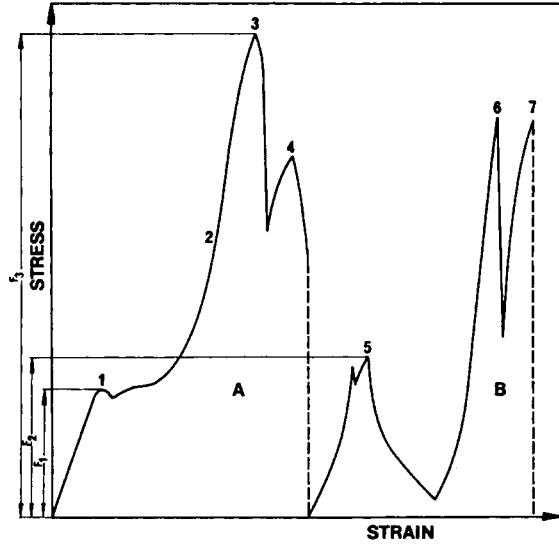


Fig. 2. Stress/strain relationship for laminates at short-beam test. A, Phenol laminate specimens: 1, interlaminar shear failure; 2, development of transverse shear cracks; 3, transverse shear failure at one side of a specimen; 4, final transverse shear failure. B, Polyester laminate specimens: 5, tensile-type failure in the most stressed cross-section; 6, first compressive failure; 7, final compressive failure. F_1F_2 , Stresses for calculation of interlaminar shear strength; F_3 , stress for calculation of transverse shear strength.

systems is shown as a histogram in Figure 7. Differences between the static and dynamic delamination energies and those computed by eq. (2) are presented in Table IV.

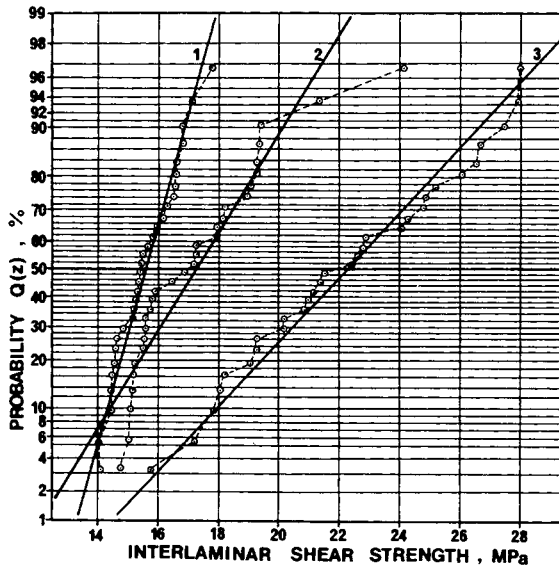


Fig. 3. Distribution of interlaminar shear strength results. 1, Phenolic laminate thin specimens, gel coat down. 2, Phenolic laminate thin specimens, gel coat up. 3, Polyester laminate thin specimens.

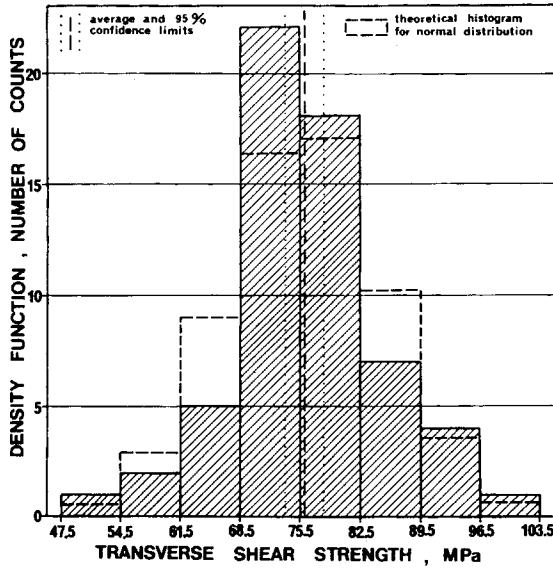


Fig. 4. Distribution of transverse shear strength results for phenolic laminate thin specimens.

DISCUSSION

Chopped Strand Mat (CSM) Reinforced Laminates

The tests show that neither the variation of laminating techniques nor the actual glass content in the laminates influences the results of interlaminar shear measurements significantly (see Figs. 5 and 6). When plane specimens are used to give shear in the same plane as the plane of the layers of glass, the final fracture of the short-beam specimens is different for the two materials. In the case of the phenolic laminates the first cracks due to interlaminar shear stresses develop

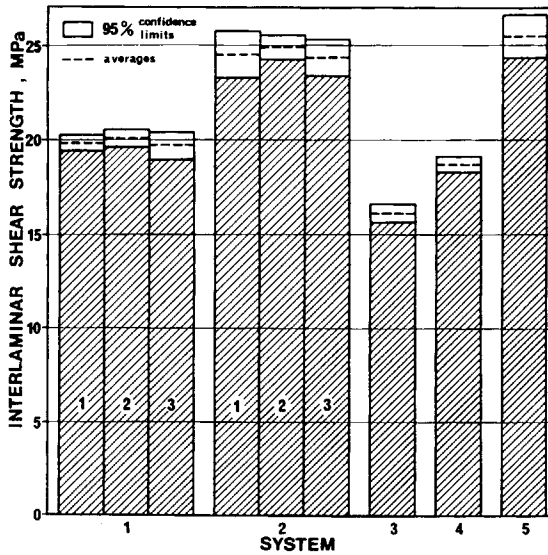


Fig. 5. Interlaminar shear strength of the laminates measured with plane specimens.

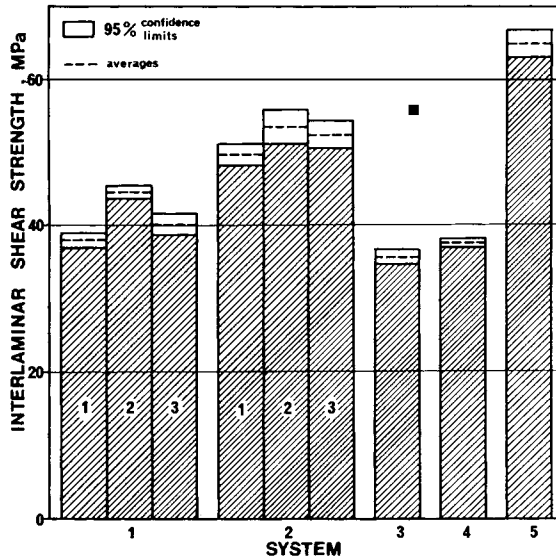


Fig. 6. Interlaminar shear strength of the laminates measured with perpendicular specimens.

when these stresses reach about 15 MPa. The interlaminar failure occurs at stress levels of about 20 MPa as shown in Figures 2 and 5, but the specimens are not broken unless stress reaches levels of about 76 MPa. Tensile fracture in the most stressed cross-section of the specimens is avoided because of the relatively high elongation at break of the resin of up to 3%.¹³ Until this stress level is reached the development of transverse cracks is observed, especially in the cross-sections previously weakened by the interlaminar cracks as shown in Figure 8. When the stresses exceed the transverse shear strength of the phenolic laminates a shear fracture occurs at one side of a specimen followed by the final shear

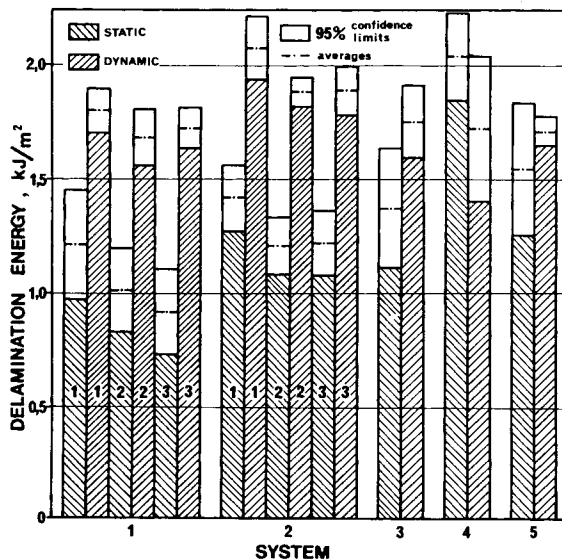


Fig. 7. Static and dynamic delamination energies of the laminates.

TABLE IV
Measured and Computed Differences between Static and Dynamic Delamination Energies for
Evaluated Laminates

System	$E_{dyn} - E_{stat}$ (kJ/m ²)	Difference computed by eq. (2) (kJ/m ²)	$\frac{E_c}{E_{dyn} - E_{stat}}$ (%)
1 No. 1	0.5871	0.5985	- 1.9
No. 2	0.6602	0.6390	+ 3.2
No. 3	0.8101	0.6227	+ 23.1
2 No. 1	0.6524	0.7359	- 12.8
No. 2	0.6798	0.7359	- 8.3
No. 3	0.6667	0.7437	- 11.5
3	0.3815	0.1533	+ 59.8
4	Specimens broken due to shear and bending forces		
5	0.1665	0.2066	- 24.1

fracture of the remaining side. A specimen after this kind of break is shown in Figure 9. The distribution of results of the transverse shear strength is similar to the normal distribution as shown in Figure 4 but shows higher peakedness, i.e., kurtosis.

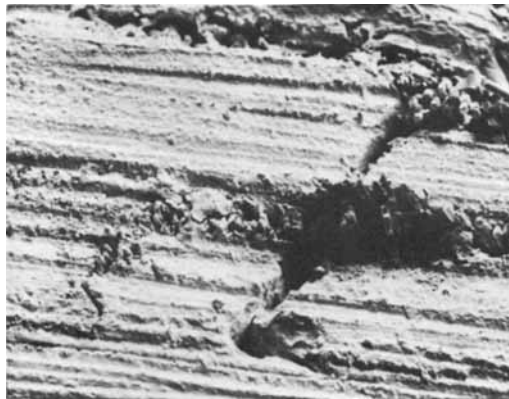


Fig. 8. Development of the transverse shear cracks in the short-beam plane specimens cut from the phenolic laminate samples.

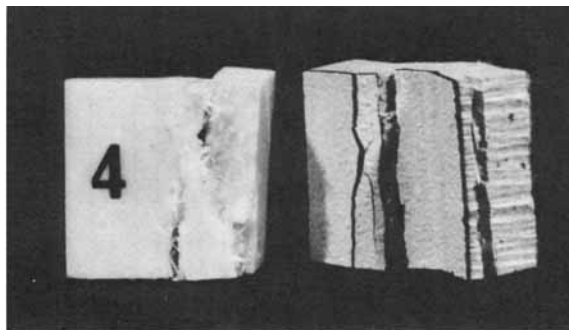


Fig. 9. Broken short-beam plane specimens cut from polyester (marked 4) and phenolic (unmarked) laminate samples.

The values of the interlaminar shear strength measured using specimens cut from the thin laminate samples were about 20% less than using plane specimens cut from thick laminate samples. This can probably be explained by the bulk effect which causes the latter specimens to be better cured due to a higher exothermal effect in thicker samples. For specimens cut from the thin laminate samples tested with surface finishing polyester gel coat/phenolic primer layers up, i.e., in contact with the single loading edge, the distribution of results contrasted with the normal distribution as shown in Figure 3. The results were also 12% higher than for the specimens tested with the surface finishing layers down. They were, however, 15% less than for the plane specimens cut from the thick laminate samples. The first difference can be caused by the fact that the gel coat also has some strength in compression, but is very weak in tension and will readily crack. This will produce stress concentrations, leading to failure of the proper laminate (see Fig. 10). It is not as good as the glass-reinforced resin even in compression and is still liable to crack and initiate failure.

In the case of polyester laminates the fracture of short-beam specimens as represented by point 5 in Figure 2 is of a double character. The interlaminar shear is combined with a tensile-type fracture in the most stressed cross-section as shown in Figure 11. The interlaminar fracture in the specimen's mid-plane often resulted in withdrawal of the reinforcing glass fibers from the polyester resin matrix as shown in Figure 12, although adhesion between the components is very good. Due to the described dual fracture mechanism the ultimate stresses decrease suddenly after the development of a fracture at stress levels of about 24 MPa as shown in Figure 2. Further increase of stress and the two peaks marked 6 and 7 in Figure 2 are related to the compressive damage of the specimens between the upper and lower loading tools.

The interlaminar shear strength of the polyester laminates calculated on the basis of the stress level marked 5 in Figure 2 is in a very good agreement with data published by other authors.^{5,16} The comparison of the two kinds of laminate, i.e., phenolic and polyester, seems, however, to be doubtful because of the above described differences in the fracture mechanism (see Fig. 13).

When the perpendicular specimens to give shear in a plane perpendicular to

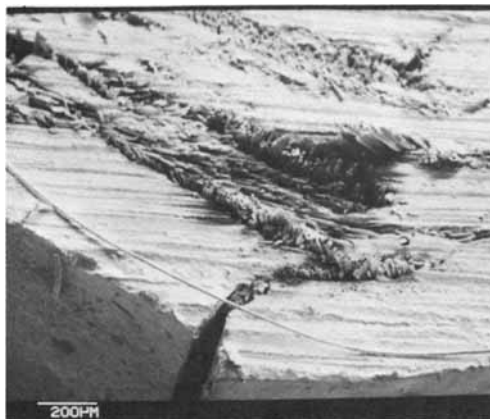


Fig. 10. Development of interlaminar shear cracks near the transverse cracks in the surface finishing layer as observed for short-beam plane specimens cut from the thin phenolic laminate samples.



Fig. 11. Combined tensile and interlaminar shear-type fracture of short-beam plane specimens cut from the thin polyester laminate samples.

the plane of the layers of the glass were used the fracture mechanism of the materials was the same or at least very similar as shown in Figure 14. In the case of phenolic laminates interlaminar cracks perpendicular to the specimen's plane also appeared at higher levels of stress. The stress/strain relationship was very similar to that of the plane polyester specimens but the stress levels were about twice as high. Also the differences between the different laminating procedures became more significant as shown in Figure 6.

From all previously performed tests^{2-4,13} it is evident that the phenolic laminates are strong but nonbrittle and that the polyester laminates are strong and brittle. Therefore it was expected that the delamination energies for the latter, especially when measured dynamically, should be lower than for the phenolic laminates. On the contrary, the tests have shown that more energy is absorbed in delaminating the polyester laminates. It should be noted that for both types of static and dynamic delamination energy tests the polyester and phenolic

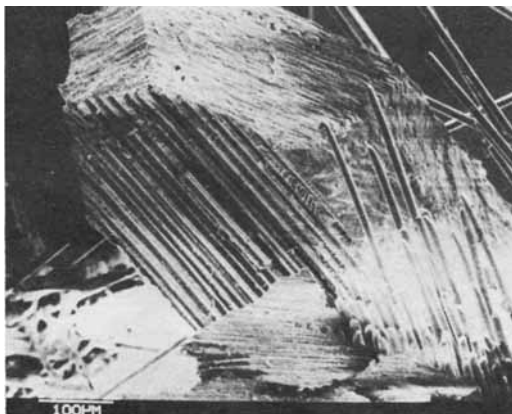


Fig. 12. Withdrawal of glass fibers from the polyester resin matrix due to the interlaminar shear forces in short-beam plane specimens.

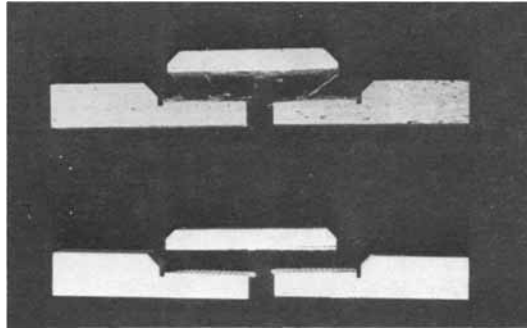


Fig. 13. Dynamically broken delamination energy specimens cut from thick phenolic (upper) and polyester (lower) laminate samples.

laminate specimens were always broken into three pieces as shown in Figure 13 and no other way of break was ever observed.

The same fracture mechanisms were observed for laminates with fire retardants as without for both plane and perpendicular specimens (see Figs. 5 and 6). The values of the interlaminar shear strength were, however, lower than for the standard phenolic laminates. The phenolic resins mixed with the fire-retardant catalyst are more viscous, so that the resulting laminates contained more air bubbles. These bubbles act as stress-concentrating centers so the mechanical properties of these laminates are decreased, as shown in Tables II and III, as well

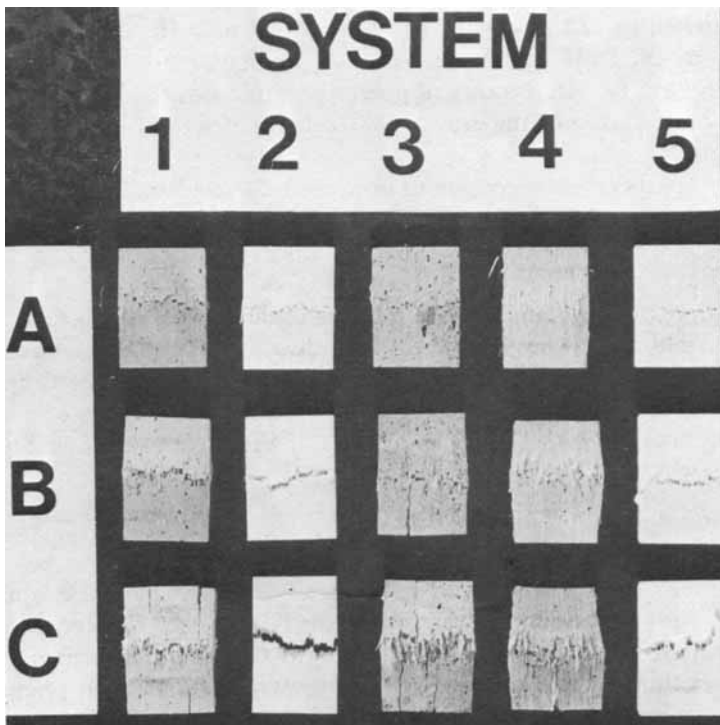


Fig. 14. Broken short-beam perpendicular specimens. (A) First cracks appear; (B) development of cracks; (C) final failure. Laminate system numbers as shown in Table I.

as the interlaminar shear strength. This is, however, compensated for by the very good fire resistance of these laminates as represented by very high oxygen index values as shown in Table III. The interlaminar shear delamination energy (static) absorbed by the specimens with fire retardants was found to be higher than for general purpose phenolic laminates and even higher than for polyester laminates as shown in Figure 7. This is probably due to higher elongation of the fire-retardant phenolic laminates or to higher interlaminar molecular friction in the resin matrix containing boron compounds. Higher adhesion between the resin and glass reinforcement caused by the above-mentioned fire retardants may also be the reason for such properties.

Fabric-Reinforced Laminates

During short-beam interlaminar shear strength tests with the plane specimens the behavior of both elevated laminate systems 4 and 5 were similar, but the actual strength values were very different as shown in Figures 5 and 6. As expected, in the case of the phenolic laminates the interlaminar shear strength values were even lower than for the laminates reinforced with chopped strand mats. The reason for this was obviously the poor adhesion between glass fibers and the phenolic resin. Poor adhesion is also confirmed by the relatively low flexural strength of these laminates as shown in Table II. The absorbed delamination energies were, however, relatively high, especially in the case of the dynamic tests. This can be related to the dissipation of energy by friction caused by the withdrawal of glass fibers from the phenolic resin matrix. Shear fracture of the thin resin layers between reinforcing glass fabric layers was also noted as well as thickening of the specimens under stress near the upper loading tool. This was observed still better when the perpendicular specimens were used as shown in Figure 14. In the case of polyester laminates the interlaminar shear fracture occurred in the same way as for the laminates reinforced with chopped strand mats.

When the perpendicular specimens were used the measured interlaminar shear strength values were the highest recorded for the laminate systems obviously due to the good adhesion between the fabric fibers and the polyester resin as well as due to the high glass content in the laminates.

During the static interlaminar delamination energy tests the phenolic laminate specimens reinforced with glass fabric were always damaged due to interlaminar shear and bending forces which was in contrast to the chopped strand mat reinforced specimens. This resulted in very high values of the interlaminar delamination energies (static). During the dynamic tests the specimens were, however, always damaged as shown in Figure 13.

CONCLUSIONS

(1) The measured interlaminar shear strength of the phenolic laminates is about 20% lower than that of the polyester laminates. The comparison is, however, doubtful because of differences in the fracture mechanism.

(2) The values of the interlaminar shear strengths of both phenolic and polyester laminates are relatively low compared to the values for advanced composites. Shear loads must be carefully minimized by design especially for thick items.

(3) The interlaminar shear strength of GRP is simply related to the adhesion between the reinforcement and the resin matrix.

(4) The use of perpendicular specimens in short-beam interlaminar shear strength testing is more discriminating for the comparison of different kinds of laminates than using plane specimens.

(5) Neither static nor dynamic interlaminar delamination energies of GRP are correlated to the interlaminar shear strength.

(6) The difference between static and dynamic delamination energies can be explained as the kinetic energy of the broken specimens leaving the Charpy impact testing machine.

(7) The dynamic method for measurement of the interlaminar delamination energy can be recommended for eventual standardization because of simplicity and good reproductivity of results.

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