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Studies of the flexural properties of asbestos-reinforced phenolic composites

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Factors which may influence the flexural strength of asbestos-reinforced phenolic composites were investigated. These are: fibre-to-resin adhesion, the degree of fibre dispersion, the relative fracture strains of the fibres and matrices, and voids. Fibre-to-resin adhesion was promoted by pre-coating asbestos fibres with phenol-formaldehyde in solution state through *in situ* polymerization. Fibre dispersion was controlled by applying shear agitation to the mixture of fibres in solution during *in situ* polymerization. The ratio of the fracture strain of the fibres to that of the matrix was varied by using resins having different fracture strains. Voids were found to be present in all cases. The size of voids was not significantly affected by different processing conditions.

It is concluded that the flexural strength of the composites is largely controlled by voids in the moulded parts. Other factors have little effect on the flexural strength when they are varied over a range of practical importance.

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1. Introduction

Phenolics have been one of the most important engineering plastics in the plastics industry since they are inexpensive and have unusually stable thermal and mechanical properties when filled with either fillers or reinforcements [1]. Wood flour-filled general purpose grade, mica and asbestos-filled electrical grade, and fibre-reinforced engineering grade are examples. In all these areas of use, a composite with a higher flexural strength than currently available commercially is greatly desired. As is the case with other composite materials, the properties of phenolics vary widely (Table I) depending on the type of fillers or reinforcements used, and on the fabrication processes.

The purpose of this paper is to examine the relative importance of some of the variables which may control the flexural strength of a composite, based on the two-stage novolak resin and asbestos fibres. The investigation was started by examining the microstructure of commercially prepared composites and then determining the influence of the factors that were thought to be the most important.

2. Examination of commercial grade composites

The composite studied in this work consisted of asbestos filler (chrysotile), asbestos fibres (crocidolite), and phenol-formaldehyde resin. The exact composition is summarized in Table II. Lack of control over the orientation and the dispersion of fibres, and the non-uniform properties and geometry of the reinforcements make it practically impossible to assess the strength properties of this composite analytically. Therefore, preliminary measurements were made of the flexural and impact properties of commercial grade composites, followed by an examination of the resulting fracture surfaces. Table III gives the values of the flexural strength and modulus. The compression-moulded bars had an average flexural strength and an average modulus of 13.6×10^3 and 2.32×10^6 psi*, respectively, which are slightly higher than those of the transfer-moulded bars. The difference in the average impact strength was more significant: an average of 1.20 ft-lb in.$^{-1}$ † of notch

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* 10^3 psi ≡ 6.89 N mm^{-2}.
† 1 ft-lb in^{-1} = 53.4 Jm^{-1}.

TABLE I Mechanical properties of phenolics

Filler	Reference	Flex. strength (10^3 psi)	Modulus (10^6 psi)	Failure strain (%)	Impact strength (ft-lb in.$^{-1}$)
None	[10]	11.9	1.0	1	–
None	[11]	8.9	0.78	–	0.28
Asbestos	[12]	7–13	–	–	0.1–0.5
Asbestos	[13]	7–14	1.0–2.2	0.2–0.5	0.26–3.50
40% 7TF02*	[11]	13.5	1.44	–	0.20
60% 7TF02*	[11]	12.9	2.03	–	0.39
7T15*	[11]	13.7	1.03	–	0.32
Wood flour	[12]	7–14	1.0–1.2	0.4–0.8	0.24–0.60
Mica	[12]	8–12	–	0.1–0.5	0.27–0.38
Glass	[12]	10–60	2.0–3.3	0.2	0.3–18.0

* Asbestos fibre graded by the Quebec system.

TABLE II Composition of moulding compounds in wt %

Components		Type I	Type II		Type III	
		Commercial grade	Control	Pre-coated	Control	Toughened matrix
Resin:	Durez	42.1	42.1	35.3	–	–
	Borden RC-1000	–	–	–	42.1	–
	U.C. BRPA-8152	–	–	–	–	42.1
Long fibres:	Crocidolite H (as received)	33.0	33.0	–	33.0	33.0
	(pre-coated)	–	–	39.8	–	–
Short fibres:	Chrysotile 7RF-9	21.6	21.6	21.6	21.6	21.6
Lubricants:	metal stearates	2.6	2.6	2.6	2.6	2.6
Accelerator:	lime	0.7	0.7	0.7	0.7	0.7
		100.0	100.0	100.0	100.0	100.0
Water		3–4	3–4	3–4	3–4	3–4

TABLE III Flexural and impact properties of the commercial grade asbestos–phenolics composites

	Flexural strength (10^3 psi)	Flexural modulus (10^6 psi)	Impact strength (ft-lb in.$^{-1}$)
Compression-moulded	13.6 ± 0.5	2.32 ± 0.12	1.20 ± 0.16
Transfer-moulded	11.4 ± 0.6	1.75 ± 0.14	0.57 ± 0.08

Note: Data represent the average and the standard deviations of at least five specimens.

for the compression-moulded bars compared to an average of 0.57 ft-lb in.$^{-1}$ of notch for the transfer-moulded bars.

The fracture surfaces were examined using a scanning electron microscope (SEM). Scanning electron micrographs, shown in Fig. 1, revealed the following features:

(1) a high concentration of large unopened fibre bundles with diameters up to 0.25 mm;

(2) poor adhesion between fibres and resin. Fibres are pulled out of the matrix cleanly: the large bundles in particular show no evidence of being wetted by the resin;

(3) preferential orientation of fibre bundles. Compression-moulded specimens (Fig. 1a and b) show more fibre bundles parallel to the mould surface (i.e. perpendicular to fracture surface) than the transfer-moulded bars (Fig. 1c and d), where a large fraction of fibre bundles are oriented perpendicular to the mould surface (i.e. parallel to the fracture surface).

It is known that the reinforcing value of unopened asbestos fibre bundles is less than that of well opened and dispersed fibres [2, 3]. Because the adhesion of matrix to fibre is limited to only the outer fibres in the bundles, unless the inter-fibre adhesive strength is high, the contribution of the inner fibres to the flexural strength is insignificant. It is also known that orientation of the large fibre bundles is important. When adhesion is poor, fibre

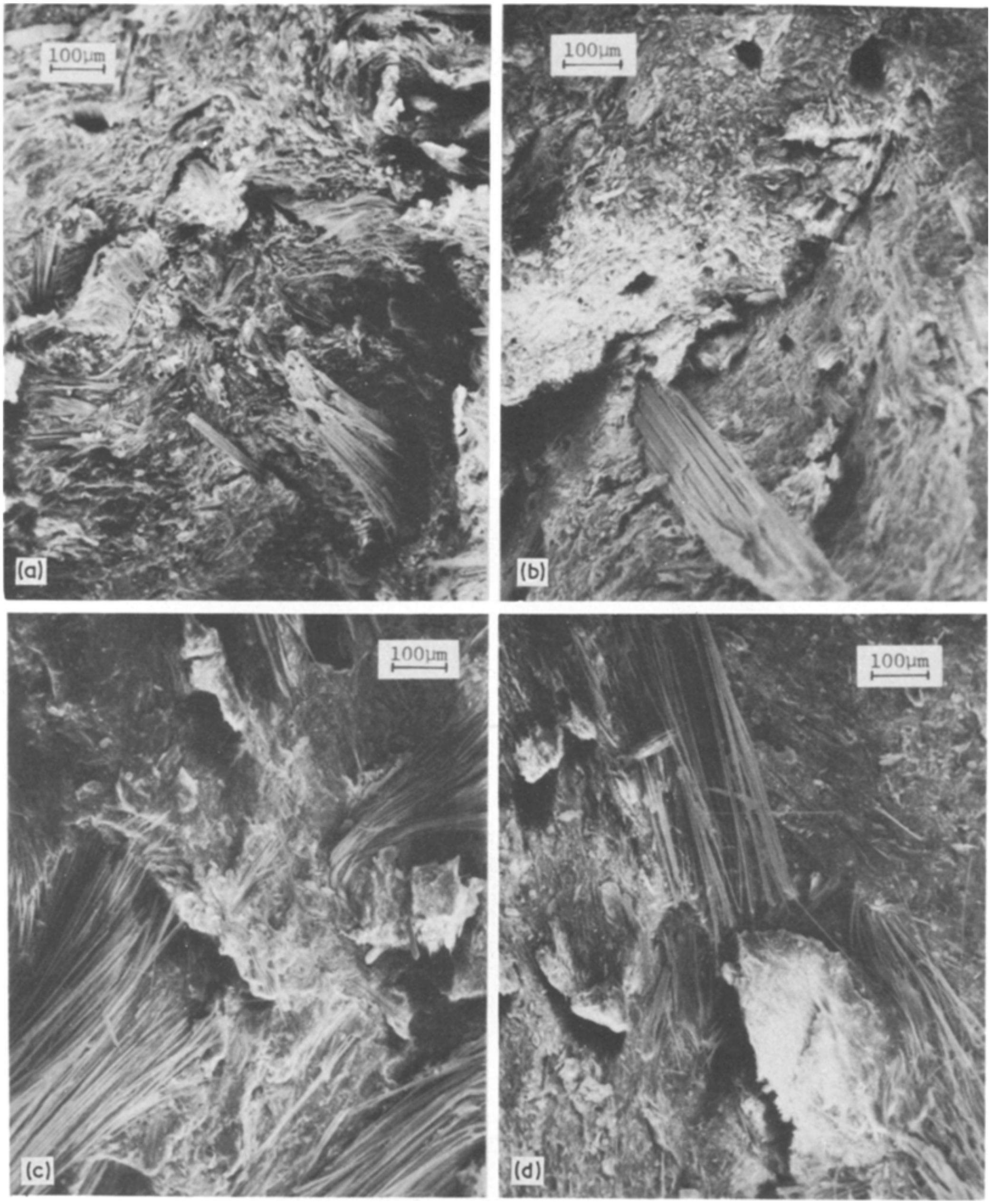

Figure 1 Scanning electron micrographs of the fracture surfaces of commercial grade phenolics: (a) and (b) compression-moulded; (c) and (d) transfer-moulded.

bundles lying perpendicular to the stress direction, as seen in transfer-moulded specimens (Fig. 1c and 1d), can act as stress raisers similar to large voids. Hypothesizing that this may be responsible for the lower impact strengths of the transfer-moulded specimens *vis-a-vis* that of compression-moulded ones, the flexural strength of the composite was investigated by making composites with fibres dispersed and pre-coated with phenolics in a solution state.

Both in flexural and in impact loading conditions, the commercial grade asbestos–phenolic composite exhibited a typical brittle fracture with fracture strains of about 0.6 to 0.8%. This is much lower than the fracture strain of the fibres (~ 1.5 to 2.0%) (Table IV). It is possible that the lower fracture strain of phenolic matrix compared to that of the reinforcing asbestos may have limited the utilization of high strength/modulus properties of fibres by premature fracture

TABLE IV Properties of some phenolic resins and asbestos fibres

Resin	Hexa content (wt %)	Flexural strength (10^3 psi)	Flexural modulus (10^6 psi)	Fracture strain (%)
Borden RC-1000	13.3	7.95 ± 0.52	0.72 ± 0.04	1.18 ± 0.05
Union Carbide BRPA-8152	13.0	11.33 ± 1.26	0.69 ± 0.03	1.81 ± 0.18
Fibres [15]	Length (mm)	Flexural strength (10^3 psi)	Flexural modulus (10^6 psi)	Fracture strain (%)
Chrysotile	–	281–436	23.2	1.5–1.9
	25	281		
	10	355		
	2	436		
Crocidolite	–	469–605	27.1	2.0
	25	470		
	10	540		
	2	603		

Note: Data on resin represent the average and the standard deviations of at least five specimens.

of the matrix. This possibility was investigaged by varying the fracture strain of the matrix.

3. Experimental

3.1 Moulding materials

Three different types of moulding compounds were investigated. One (type I) was a commercial grade phenolic prepared and supplied by Rogers Corporation (Rogers, Connecticut). The other two (types II and III) were prepared in the laboratory using the procedures described below. All types of moulding compounds contained approximately 33 wt % crocidolite asbestos fibres ($\sim\frac{1}{4}$ in. length), 22 wt % chrysotile asbestos fibres ($<\frac{1}{32}$ in. length) and 42 wt % of novolak resin. For each type II and type III formulation, control specimens of type I were also prepared under identical conditions to account for processing variables. Table II shows the complete compositions of these moulding materials.

3.2. Preparation of type II moulding compound (*in situ* polymerization of novolak/asbestos)

The type II moulding compounds are different from type I in that crocidolite fibres were precoated with phenol-formaldehyde resin by *in situ* polymerization before incorporating them into moulding compounds. The detailed steps employed are given below.

3.2.1. In situ polymerization

Approximately 810 g crocidolite fibres were charged into a 22 litre three-neck flask. 350 g formaldehyde (37 wt % aqueous solution; Allied Chemical) were then added, followed by about 46 litres distilled water to wet the fibre surface completely. 220 g phenol (88 wt %; Mallinckrodt) were then charged to give a 2:1 molar ratio of formaldehyde to phenol. The mixture was then diluted with an additional 4.6 litres of distilled water in order to allow adequate mixing of reactants by a teflon blade stirrer. After the mixture is mixed thoroughly, 4.3 g sodium hydroxide was dissolved in ~ 2.2 litres distilled water and added to the mixture. The total volume of the mixture was ~ 12 litres in which fibres were suspended. The polymerization reaction was carried out at 95° C, under reflux for about 1 h. A teflon blade stirrer was used to mix the mixture throughout the reaction. It was then terminated by neutralizing the reactants with sulphuric acid. The reaction mixture was then transferred onto a large screen and was dried by evaporating under forced air convection at 50° C. The difference in weight of fibres before and after coating was taken as the amount of resol coating. Fibres gained approximately 20 wt % of coating by this method.

3.2.2. Compounding

Type II moulding compound was prepared using coated crocidolite fibres by *in situ* polymerization. In order to maintain the overall composition of type II the same as those of type I compound, the amount of novolak resin was reduced by the amount of coating already deposited on the fibres. Compounding was carried out using a conventional laboratory scale sigma-blade kneader. Predetermined amounts of fibres, lubricant, and accelerator (lime) were charged into the kneader and mixed for 15 min without the resin and for an additional 5 min after the resin was added. Novolak resin, containing 20 wt% curing agent (hexamethylenetetramine), was added first in powder form, followed by the liquid resin form. Warm water (~ 65° C) was added to accelerate the softening of resin and the formation of agglomerates. After about 5 min, the mixture was cooled by adding iced water to prevent any premature advance of cure reaction. After a further 5 min compounding, the agglomerates were reduced to suitable sizes by a grinder. The compound was then discharged on to a screen and dehydrated in a forced-convection oven at 65° C until the moisture content was reduced to ~ 3 to 4 wt%. A control batch was prepared under identical conditions using uncoated fibres for comparative evaluation.

3.3. Preparation of type III moulding compound (toughened matrix composite)

The type III moulding compound was different from type I in that the resin matrix was substituted by a thermoplastic modified novolak (Union Carbide BRPA-8152). A control batch was also prepared using Borden Chemical's RC-1000 resin, which is equivalent to commercial grade Durez (type I). The properties of the two different novolak resins are summarized in Table IV for comparison. The compounding of the type III batch was carried out in the same manner as in the preparation of type II materials except crocidolite fibres were uncoated as in type I.

3.4. Moulding

The test specimens were moulded from all types of moulding compounds according to the ASTM Standards for flexural and impact tests. Flexural bars were 5 in. x 0.5 in. x 0.25 in. and impact bars were 5 in. x 0.5 in. x 0.5 in. Each impact bar, after being notched yielded two test specimens.

Moulding was done in the following manner. Predetermined amounts of moulding compounds were formed into a 1.25 in. diameter discs by application of 16 000 psi in a cold mould. The preformed disc was dielectrically heated to a temperature of 190° F immediately before being moulded. Both transfer and compression-moulding techniques were employed. The use of dielectric heating provided a quick and uniform pre-heating of moulding compounds which enhanced the flow of compounds within the mould as well as shortens the cure cycle. The mould temperature was 350° F, and the cure cycle was 5 min. The pressures were 6000 psi for the compression-moulding and 3800 psi for the transfer-moulding. Specimens were also compression-moulded using higher pressures up to 15 000 psi with various heating cycles in an attempt to minimize the size of voids.

3.5. Mechanical testing

Flexural properties were obtained by the three-point bending method according to the method described in ASTM D790. Impact properties were measured from notched impact bar with Izod tester using a 2 ft-lb scale according to ASTM D256.

3.6. Microscopy

Fracture surfaces of the composites were investigated using a scanning electron microscope (JEOL-2). Specimen surfaces were coated with gold using a vapour deposition method.

The microstructure of the composites was examined by optical microscopy using a well-polished cross-section of the composites. Both optical and scanning electron microscopes were used to study the surface characteristics of asbestos fibres before and after coating by the *in situ* polymerization process.

4. Results

Fig. 2 shows scanning electron micrographs of crocidolite fibres before (Fig. 2a and b) and after (Fig. 2c and d) *in situ* polymerization. The resin is clearly visible as a film on and between the opened fibres in Fig. 2c, which also shows that all the short fibres are encapsulated. This is in contrast to untreated fibres which typically have large bundles of fibres as well as small fibres (Fig. 2b). Although a significant improvement was achieved in fibre opening and dispersion, some fibre bundles remained unopened even after the *in situ* poly-

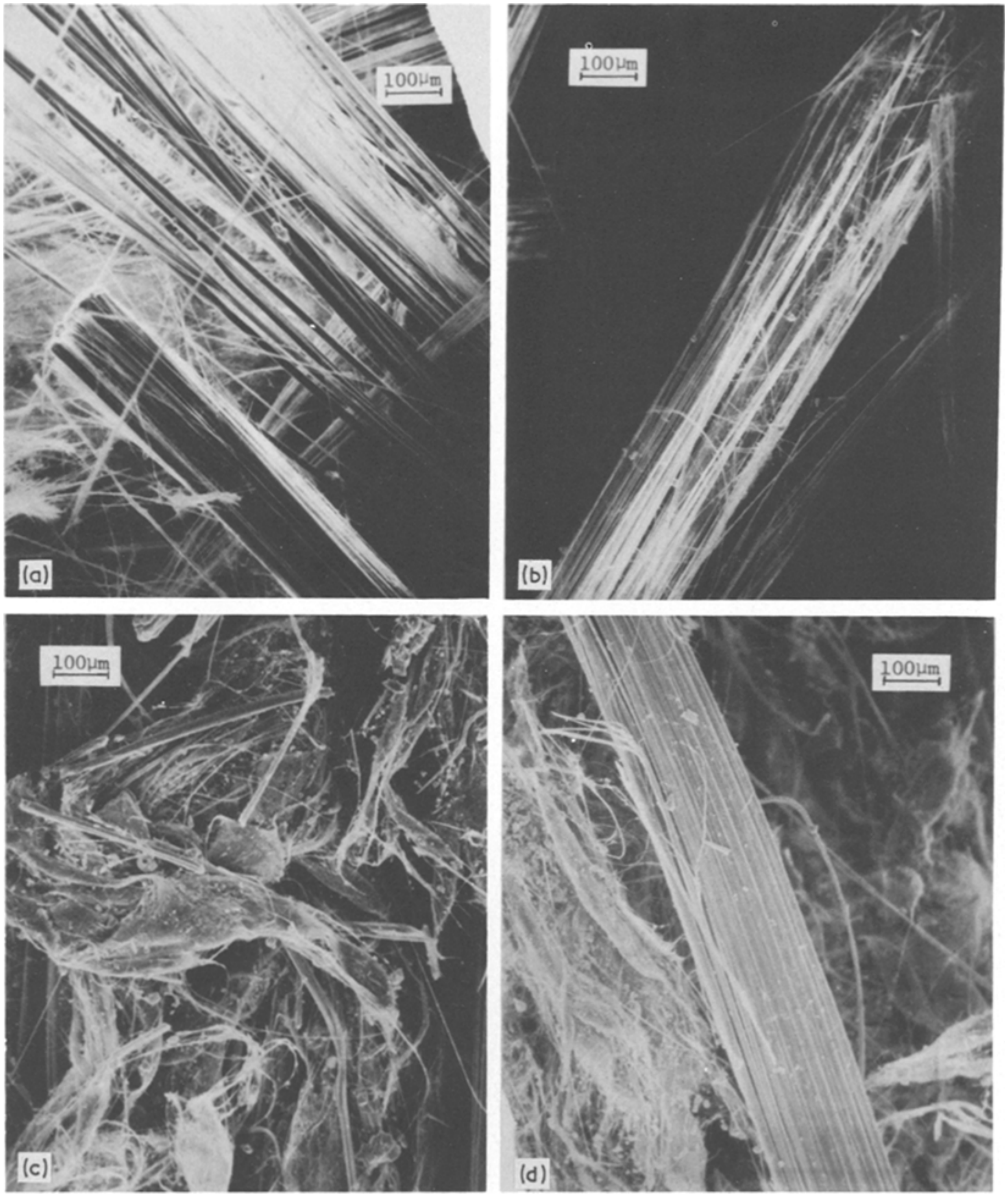

Figure 2 Scanning electron micrographs of asbestos fibres: (a) and (b) fibres as received; (c) and (d) fibres coated by *in situ* polymerization.

merization process. The unopened fibre bundles were also resin-coated, showing a smoother surface (Fig. 2d) than the uncoated fibre bundles (Fig. 2a and b). The penetration of the resin through the inter-fibre crevices within a fibre bundle is clearly seen in Fig. 2d.

The flexural and impact properties of composites containing precoated fibres (type II) are summarized in Table V and compared with the control that contains regular fibres. Improvements in flexural strengths were about 10% in both compression- and transfer-moulded specimens: 13×10^3 psi compared to 11.8×10^3 psi (of control) in compression-moulded and 11.9×10^3 psi compared to 10.7×10^3 psi (of control) in transfer-moulded, respectively. Modulus increases were also of the same order of magnitude: about 10% in the compression-moulded and 7% in the

TABLE V Flexural and impact strengths of type II (*in situ* coated) and control composites

		Flexural strength (10^3 psi)	Flexural modulus (10^6 psi)	Impact strength (ft-lb in.$^{-1}$)	Fracture strain (%)
Compression-moulded	coated	13.0 ± 0.9	2.46 ± 0.12	1.39 ± 0.12	0.60 ± 0.02
	control	11.8 ± 0.3	2.21 ± 0.15	1.47 ± 0.15	0.63 ± 0.03
Transfer-moulded	coated	11.9 ± 0.8	1.75 ± 0.18	0.56 ± 0.13	0.78 ± 0.03
	control	10.7 ± 0.9	1.64 ± 0.15	0.69 ± 0.09	0.74 ± 0.06

Data represent the average and the standard deviations of five to eight specimens each.

transfer-moulded. Impact strengths were, however, lowered slightly in compression-moulded (~ 5%) and substantially in transfer-moulded specimens (~ 17%). Fracture strain remained more or less the same at 0.6 to 0.63% in compression-moulded bars and at 0.74 to 0.78% in transfer-moulded bars.

Improvement in fibre dispersion was clearly observed in optical micrographs of polished cross-sections as well as in SEMs of fracture surfaces. Both showed a smaller number of large fibre bundles. Also the surface of the moulded parts was much smoother in type II specimens than in control specimens. During the compounding of type II moulding materials, the flying asbestos dust was greatly reduced compared to the compounding of control batches. The pre-coating technique employed in type II compound minimizes the health hazard problem in handling asbestos substantially.

The flexural properties of composites based on the thermoplastic modified matrix (type III) and those of control composites all moulded in compression are compared in Table VI. No significant improvements were obtained in the flexural properties of the composites in spite of the significant differences in flexural strengths (7.95 × 10^3 versus 11.33 × 10^3 psi) and failure strains (1.18% versus 1.81%) observed in the resins used. Only slight improvement in flexural strength (13.5 × 10^3 psi versus 12.4 × 10^3 psi) was obtained with no improvement in failure strain. The difference in modulus was insignificant and amounts to the scattering range of the data.

TABLE VI Comparisons of flexural properties of type III and control composites

	Flexural strength (10^3 psi)	Flexural modulus (10^6 psi)	Fracture strain (%)
Tough matrix	13.49 ± 0.40	2.14 ± 0.08	0.71 ± 0.04
Control	12.43 ± 0.60	2.05 ± 0.07	0.74 ± 0.09

Data represent the average and the standard deviation of at least five specimens.

Optical micrographs of the polished cross-sections of the unfilled phenolics and the composites are shown in Figs. 3 and 4, respectively. Voids, predominantly spherical in shape and varying in size up to 100 μm, are present in both regular novolak (Fig. 3a) as well as in thermoplastic modified novolak (Fig. 3b). Much larger voids (~ 0.5 mm) with irregular shapes were found in all composites of type I (a), type II (b) and type III (c) as shown in Fig. 4.

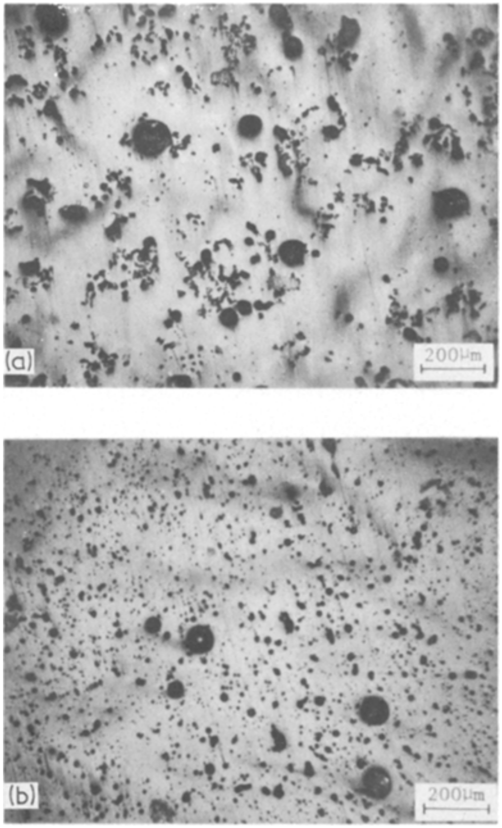

Figure 3 Optical micrographs of the cross-section of unfilled phenolics: (a) regular novolak; (b) toughened novolak.

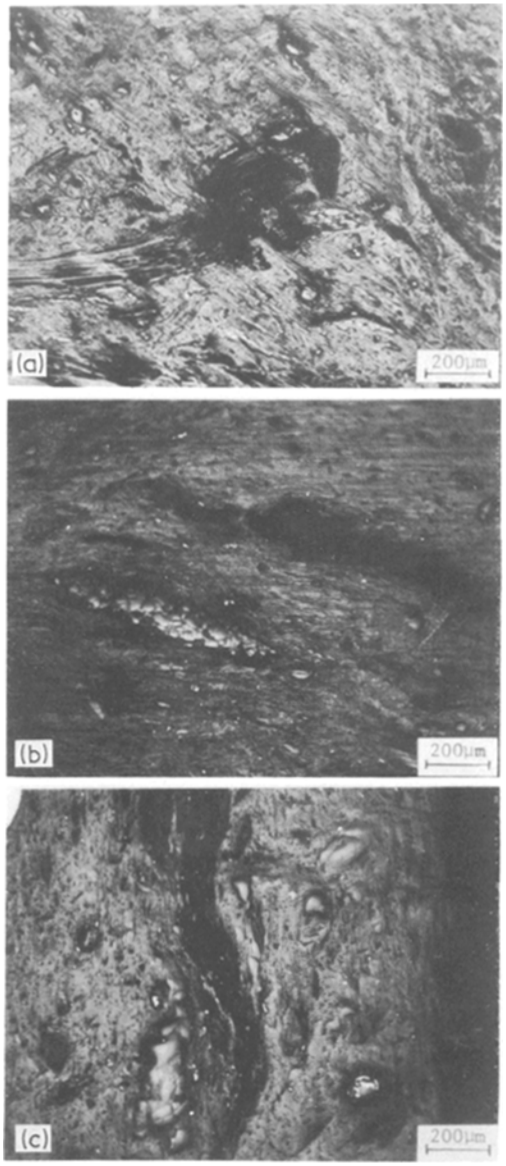

Figure 4 Optical micrographs of the cross-section of composites: (a) type I; (b) type II; (c) type III.

5. Discussion

It is clear that the *in situ* polymerization technique yielded fibres that were well dispersed and coated with phenolic resin (Fig. 2). However, the effect of this pre-coating on the flexural properties of the composite was rather insignificant, resulting in an improvement of only about 10% (Table V). The impact strengths decreased by a similar amount. Although the agitation applied to fibres during pre-coating could result in reductions in fibre lengths of the pre-coated fibres to some extent, such a change was not observed before or after the coating. Furthermore, both the pre-coated and regular fibres were subsequently subjected to the identical dry compounding operation where fibres were exposed to much more severe shear field. The extent of fibre damage would be governed primarily by the compounding process and, therefore, must be the same in both control and the type II moulding compound. Even though the effects are not substantial, the improved flexural properties combined with the reduced impact strength in the type II composites are indicative of the increased fibre-to-resin adhesion as shown in other composite studies [3–5]. It should be noted, however, that similar effects could result from improved fibre dispersion alone as was shown in asbestos/rigid-PVC composites by Crugnola and Litman [2]. They reported that a slightly increased tensile strength and reduced impact strength were obtained as a result of improving the openness of asbestos fibres. This decrease in impact strength contrasts with the findings of Williams *et al.* [4] on short glass fibre/PMMA composites, where the impact strength was reported to increase monotonically as the fibre dispersion was enhanced (by reducing the diameter of fibre bundles). The discrepancy may be due to the differences in inter-fibre adhesive strength and the aspect ratio of fibre bundles. The results obtained with type II composites in this study cannot differentiate the effect of improved adhesion from that of better dispersion.

The ineffectiveness of the pre-coating techniques in improving the flexural strength of composite is not caused by the fact that the failure strain of the matrix (~ 1%) is smaller than that of the reinforcing fibres (~ 1.5%). Increasing the failure strain of the matrix from ~ 1% to ~ 1.8% in type III composites gave no substantial changes in either the strength or the failure strain of the composite (Table VI). This result differs from the work done by Gaggar and Broutman [6] on randomly oriented glass fibre/epoxy composites. They also reported that the increased ductility of the matrix does not result in greater elongation-to-failure of the composite under tensile and flexural load. They noted that in a composite specimen, the matrix material is subjected to a complicated triaxial stress even when uniaxial loading is applied. Under a triaxial stress condition, the reduced yield strength of the matrix which was brought about in increasing ductility leads to a lower failure strain of the matrix in composite and thus does not increase the failure strain of the composites. This. however, is not the case in the type II composites in this work because the failure strain of the matrix was increased without reducing the modulus or the yield strength (Table IV).

Results obtained with types II and III composites strongly suggest that the initiation of the fracture process and, thus, the ultimate flexural strength are controlled by factors other than fibre–matrix adhesion, fibre dispersion, and fracture strain of the matrix. The presence of voids in matrix and composites shown in the micrographs (Figs. 3 and 4) appears to be critical. The role of voids in brittle fracture would be similar to that of a Griffith crack.

According to linear elastic fracture mechanics (LEFM), the introduction of an elliptical crack in a brittle material intensifies the stress at the crack tip. The maximum stress, $\sigma_{\max}$, is related to the applied nominal stress, σ_{app}, by

$$\sigma_{\max} = \sigma_{\text{app}} [1 + 2\sqrt{(c/\rho)}] \qquad (1)$$

where c is the half-length of the crack and ρ is the radius of the crack tip. Even though direct applications of LEFM to various fibrous composites are still in question [7–9], a generalized relation such as:

$$\sigma_{\max} = \sigma_{\text{app}}(1 + q\sqrt{c}) \qquad (2)$$

is applicable [8] when ρ is less than a critical value, ρ_0, of the material and q is the generalized stress concentration parameter. Thus, fracture will occur when

$$\sigma_{\text{app}} = \frac{\sigma_{\text{UTS}}}{1 + q\sqrt{c}} \qquad (3)$$

where σ_{UTS} is the ultimate tensile strength of the material. The value of the parameter q is not known for asbestos–phenolics composites, but is constant for any given material. Since σ_{UTS} is an inherent property of the material, for a given value of q, it is clear that the larger the crack (large c), the smaller will be the σ_{app} at fracture.

Some of the voids in the composites shown in Fig. 4 are as large as 1 mm in length, whereas those in unfilled phenolics range only up to 100 μm. The origin of the voids in matrix may be water present in the moulding compound as a plasticizer and by-product from the condensation reaction, ammonia from the decomposition of hexamethylenetetramine, entrapped air, and weld lines resulting from non-uniform heating. The large irregular voids in composites must be associated with the flow of the anisotropic fibre bundles during moulding which may either entrap air or develop resin starved areas.

The high-pressure moldings with various heating cycles were not successful in eliminating large voids or crack-like voids in particular. High pressure cannot eliminate crack-like voids if they form during unloading due to the residual stress. Application of a pure hydrostatic state of compressive stress during moulding may partially alleviate this problem. Such an attempt was made by Price and Lucy [14] to eliminate voids in asbestos–phenolic and glass–phenolic composites. The application of different combinations of vacuum, and unidirectional heating during moulding was reported to yield only about a 5–7% increase in flexural properties over conventionally moulded composites. This insignificant effect appears to be due to the fact that these methods are effective only in eliminating voids in an unfilled matrix.

6. Conclusions

(1) Pre-coating asbestos fibres by *in situ* polymerization in aqueous phase provides improved fibre–matrix adhesion and fibre dispersion, but provides only a limited improvement (~ 10%) of the flexural properties of composites.

(2) An increase in fracture strain of the phenolic matrix does not significantly affect the failure strain of composites and, thus, the strength of composites.

(3) Large irregular voids, which are absent in the unfilled matrix, are present in all the composites studied in this work. These voids are considered to act as stress raisers and become responsible for the initiation of the fracture, thus acting as the controlling factor in determining the flexural strength.

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