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The effect of elevated temperatures on the mechanical properties of B-Al composites

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The mechanical properties of an aluminium alloy reinforced with 20 and 50 vol % boron fibres have been obtained from specimens in the "as-received" condition and after heating to 600° C for various periods of time. Heating the specimens caused a reduction in the load-bearing capacity of each specimen and the eventual growth of a reacted layer at the fibre-matrix interface. As-received specimens fractured in a sudden brittle manner; however, those specimens heated for 4 h at 600° C slowly pulled apart. The variation in the static strength of the as-received and the heated specimens is explained in terms of upper and lower bounds calculated from a knowledge of the statistical strength variation exhibited by individual fibres.

1. Introduction

It is known that the mechanical properties of a metal–matrix composite are sensitively dependent upon the thermal history of the material. In particular, high-temperature reaction products have been observed at the interface between boron and aluminium [1, 2]. Also, heating to a temperature of 538° C for short periods of time has been shown to reduce the tensile strength [3]. In the present work, the mechanical properties of a 6061 aluminium alloy reinforced with 0.102 mm diameter boron fibres, determined after holding specimens for various sustained periods of time at a temperature of 600° C and cooling in air, are compared to the as-received properties.

2. Experimental procedures

The test material was fabricated by the Marquardt Corporation* using a proprietory diffusion-bonding technique. In general, the process consisted of applying a pressure of several thousand pounds per square inch to foil-filament arrays at a temperature of about 500° C. The ashless organic binder, which was used to maintain the integrity of the original arrays, was burnt off during the diffusion-bonding operation. After fabrication, the material was cooled in air by placing the composite panel on a large aluminium heat sink.

Each panel, nominally containing 20 or 50 vol % boron fibres and measuring 25 × 25 cm, was cut by mechanical shears to produce specimens 10 cm long, 1.3 cm wide, and 0.05 cm thick. The elastic modulus of each specimen was then obtained by applying a load, at a crosshead speed of 0.05 cm per min, in an Instron tensile test machine. In order to minimize any effects caused by misalignment, a spirit level was used to insure that the rigid grips were accurately aligned before each test. Also, each specimen was oriented such that the longitudinal axis of the reinforcing fibres was parallel to the load axis of the machine.

The strain produced in each specimen by the applied load was obtained directly by monitoring the output of a conventional extensometer attached to the specimen sides with spring-loaded clamps. Specimens were loaded elastically, and the elastic modulus was computed in the as-received condition and after each heat-treatment. Each specimen was then cut into 10 cm long pieces, and a reduced test section, 1.3 cm long, 0.8 cm wide was produced in the centre of each piece by shearing away the excess material

*This material was supplied in 1969. Since that time, the company has sold its fabrication facilities to Amercom, Inc.

from the specimen sides with a paper punch.

Individual boron fibres were extracted from specimens reinforced with 20 vol % boron by dissolving away the matrix in a sodium hydroxide solution. However, since boron fibres tended to crush when gripped directly, it was necessary to glue each fibre to a cardboard support using an epoxy setting at room temperature. This type of specimen was placed in the Instron, the sides of the support were cut with scissors, and the load necessary to cause failure of the fibre was then obtained.

3. Results and discussion

3.1. Properties of composites

The tensile strengths of as-received specimens were reduced markedly by subjecting them to increasing periods of time at 600°C. Most of the strength values, shown in Tables I and II, fall within 10% of the mean values of each group. Therefore, in this respect, the variation in the strengths are similar to those obtained from unheated borsic-aluminium composites by Kreider and Marciano [4]. The same data shown in graphical form in Fig. 1 indicates the magnitude of the strength decrease. It can be observed that the initial pronounced drop in strength was followed by a more gradual decrease until after 4 h at a temperature of 600°C the mean tensile strength had decreased from an initial 60.75×10^3 psi* to 23.2×10^3 psi for the specimens containing 20 vol % boron, and from a maximum of 138×10^3 psi to 57×10^3 psi for specimens containing 50 vol % boron in the same time period. In contrast, the modulus of both materials was unchanged. However, in one extended test carried out by heating a specimen for a period of 120 h, it was found that the modulus had decreased from the initial 18.6×10^6 psi to 13.4×10^6 psi and from 28.2×10^6 psi to 19.5×10^6 psi for composites containing 20 and 50 vol % boron respectively. During this time period, a reaction product had formed at the interface between the boron fibres and the aluminium that could be easily observed using simple metallographic techniques. This is shown in Fig. 2. A reaction product of this magnitude was not observed in specimens heated for only 4 h; however, the recent observations of Klein and Metcalf [2] using the scanning electron microscope indicate that reaction between boron and 6061 aluminium occurs at an early stage in the heat-treatment.

TABLE I The strength of aluminium-20 vol % boron composites subjected to various periods of time at 600°C

	Heating time, (h)					
	0	0.25	0.5	1	2	4
Tensile strength $\sigma \times 10^{-3}$ psi*	65.0	40.0	49.0	48.6	35.0	22.0
	62.0	35.0	42.0	38.3	21.6	17.6
	68.7	43.8	40.0	42.0	25.0	30.0
	63.3	45.3	51.5	29.0	18.6	
	54.5	44.0	43.4	26.6		
	60.4		43.3			
	58.6		33.3			
	55.0					
	57.5					
	61.5					
Mean value $\sigma \times 10^{-3}$ psi	60.75	41.62	43.0	36.9	25.0	23.2

*1000 psi ≡ 6.895×10^6 N m^{-2}.

TABLE II The strength of aluminium-50 vol % boron composites subjected to various periods of time at 600°C

	Heating time (h)					
	0	0.25	0.5	1	2	4
Tensile strength $\sigma \times 10^{-3}$ psi	138.0	—	80.0	65.0	60.0	57.0

*1000 psi ≡ 6.895×10^6 Nm$^{1-2}$

In contrast to the insignificant effect produced in the elastic modulus, heating for 4 h at 600°C produced a dramatic reduction in the strength of the composite and the mode of failure appeared to change.

Fig. 3 is a stress-strain curve derived from a load-strain curve originally obtained from a specimen reinforced with 20 vol % boron, heated for 4 h at 600°C. It is to be noted that the elastic portion of the curve extends to an elastic strain of about 0.001. The decreased tangent modulus that is then exhibited results, presumably, from plastic flow of the matrix. We therefore assumed that plastic flow occurred when the stress, on the matrix, σ_m, produced an elastic strain of 0.001, i.e., $\sigma_m = 10 \times 10^3$ psi.

For the unheated composite, separation of the specimen occurred in an abrupt, brittle manner. In contrast, failure of the heated piece occurred progressively with the fracture surfaces slowly pulling apart. Also, the heated specimen was still able to support a large fraction of the applied stress even after the maximum load had been applied, for the load could be removed and

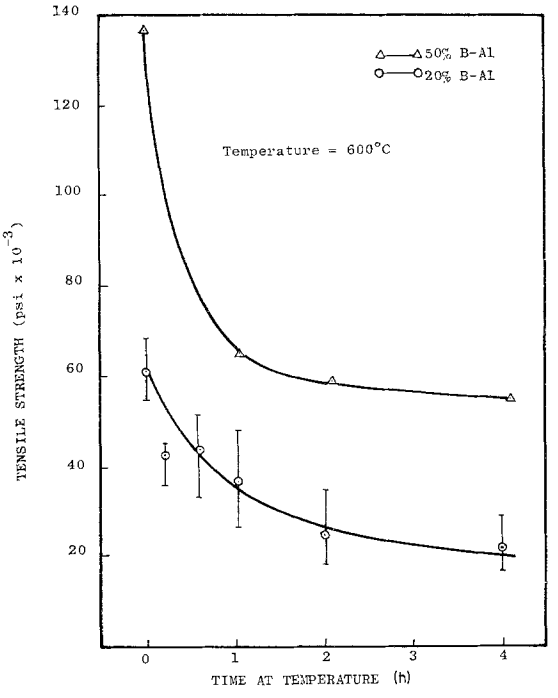

Figure 1 The effect of heating time on the tensile strength of B-Al composites.

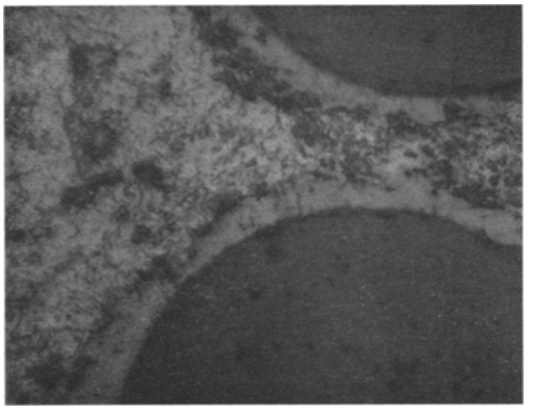

Figure 2 Reaction product layer in a 20 vol% B-Al composite heated for 120 h at 600°C.

reapplied at any point in the stress-strain curve as indicated in the figure.

As the stress was increased to the ultimate tensile strength of the composite, a zone of deformed metal, that was perpendicular to the applied load, formed on the surface of the specimen. This became progressively more intense, and final separation occurred along this line. The actual fracture surface of the specimen, as shown in Fig. 4, indicated that an appreciable

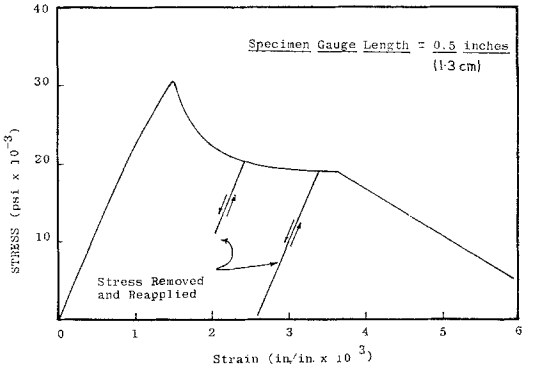

Figure 3 The stress-strain curve exhibited by a composite containing 20 vol % boron after heating at 600°C for 4 h.

amount of fibre pull-out occurred. Thus, it appears reasonable to assume that fibre pull-out accounts for part of the work of fracture of heated fibre composites. If all of the fibres pull-out then the total work done, γ, can be calculated using the expression provided by Kelly [5] and Beaumont and Phillips [6]:

$$\gamma = \frac{V_f \sigma_f l_c}{24},$$

For the heated specimen, $V_f = 0.2$, $\sigma_f = \sigma_{fB} = 104 \times 10^3$ psi, and $l_c = 0.686$ mm (see later discussion).

Thus, the maximum contribution of fibre pull-out to the total work of fracture is about 23 lbs-in in^{-2}.

3.2. Properties of the reinforcements

The mean tensile strength, $\bar{X}$, standard deviation, s, and coefficient of variation, C, for groups of 100 fibres of different gauge lengths extracted from as-received specimens and specimens heated at 600°C for 4 h are shown in Table III. The variation in the mean strength with fibre length is illustrated graphically in Fig. 5. It can be observed that the shorter fibres tend to exhibit higher strength values in agreement with the earlier work carried out on similar boron fibres by Herring [7]. It is also apparent that the heat-treatment resulted in a marked degradation in the strength of the fibres contained in the composite.

Herring indicated that a Weibull distribution could be used to describe his data and more recent work by Wright and Wills [8] has confirmed the applicability of the expression to 0.0053 in. (135 μm) diameter boron fibres. In this work a Weibull distribution was assumed

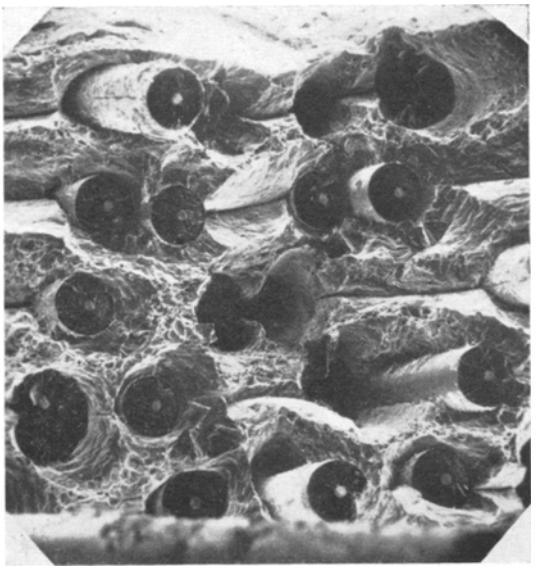

Figure 4 Photomicrograph taken with the scanning electron microscope of a composite containing 20 vol % boron fibres showing fibre pull-out, × 100.

the Weibull distribution can be performed. However, in the work reported here, a bundle efficiency factor ϵ, defined as the ratio between the strength of a bundle and the mean strength of the individual fibres, was obtained for the appropriate values of C by inspection of Fig. 6. The mean strengths of large bundles of fibres was then calculated from,

$$\bar{\sigma}_B = \epsilon \bar{X}. \quad (1)$$

Finally, the lower bound of strength of a composite specimen was obtained by multiplying the strength of a bundle of length equal to the gauge length of the specimen, i.e. 1.3 cm, by the volume fraction of fibres in the composite, V_f. The results were then assumed to be the lower strength bounds for the composites. They represent the lowest strengths that would be obtained from a composite if the matrix carried no load and transferred no stress.

Using the concept of the ineffective length, δ, and the weakest link theory, as applied to composites by Rosen [11], upper strength bounds were also calculated. In this case, the presence of the matrix causes the effect of broken fibres to be localized. Thus, in contrast to the situation in a bundle, a broken fibre can still carry load at some distance from the break. Obviously, the load in the fibre is zero at the break but it builds up rapidly until at a distance, $\delta/2$, it closely approaches that load carried by the surrounding fibres. The composite is therefore assumed to be composed of many small bundles of such fibres arranged in series. Failure of the composite occurs when one of the bundles fails.

TABLE III The effect of gauge length on the mean strength of boron fibres extracted from as-received specimens and from specimens heated 4 h at 600°C

Fibre length (in.)*	Mean strength $\bar{X}$ (10^3 psi)	Standard deviation S (10^3 psi)	Coefficient of variation C	Bundle efficiency factor, ϵ	Bundle strength $\epsilon \bar{X}$ (ksi)
3 (AR)	299.00	42.86	0.1433	0.73	218.27
2.5 (AR)	284.86	43.44	0.1525	0.71	202.25
2 (AR)	301.10	43.05	0.1430	0.73	219.80
1 (AR)	310.00	57.00	0.1851	0.70	217.00
0.5 (AR)	325.00	39.98	0.1230	0.75	243.75
0.5 (H)	125	Extrapolated		0.64	80.00
1 (H)	111.85	27.75	0.24	0.64	71.58
1.5 (H)	73.27	17.95	0.24	0.64	46.82
2.0 (H)	95.12	21.49	0.22	0.65	61.82

AR = As-received.
H = Heated 4 h at 600°C.
*1 in. = 2.54 cm.

and, using the theories of Daniels [8] and Coleman [10], the strength expected from a bundle of these fibres was calculated. It is important to note that some of the individual fibres present in a bundle of brittle fibres fail at low loads; thus, the strength of a bundle is always less than the mean strength. Essentially, the theory allows the mean strength, $\bar{\sigma}_B$, of a large bundle of fibres to be calculated provided both the mean strength, $\bar{X}$, and the coefficient of variation, C, in the strength of the component filaments are known. Actual calculations based on the values of the parameters that characterize

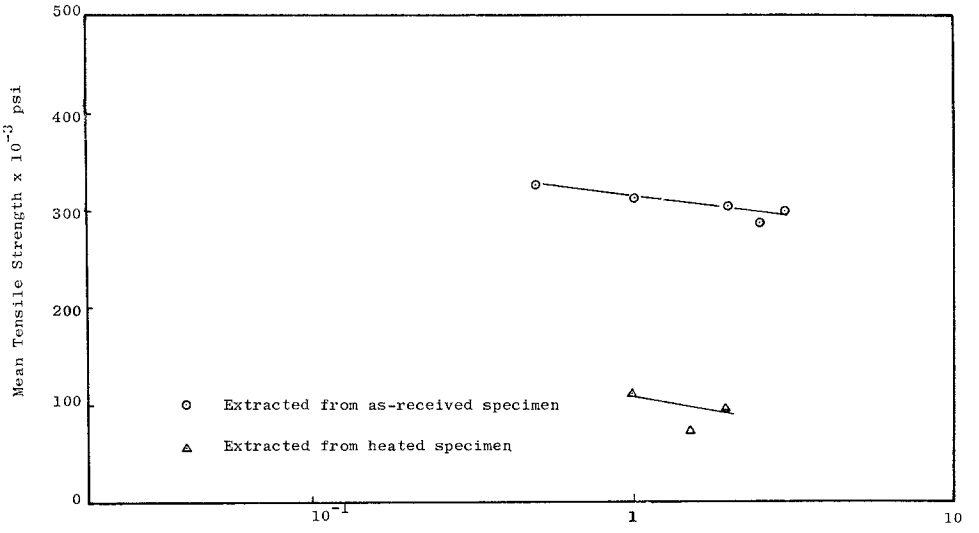

Figure 5 The effect of gauge length on the mean strength of boron fibres.

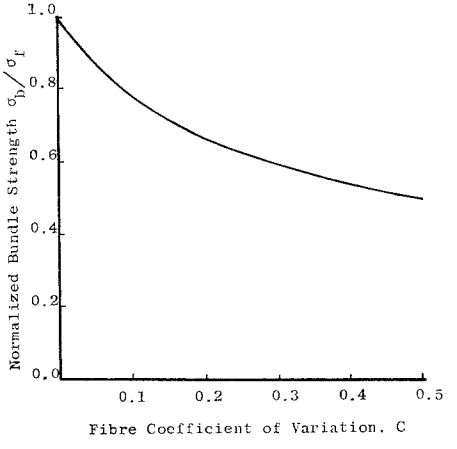

Figure 6 The bundle efficiency factor, ϵ, presented as a function of the coefficient of variation, C. (After Coleman)

For this work, the matrix was assumed to be a rigid-plastic material; thus, δ was assumed to be given by [12]:

$$\delta = \frac{\sigma d}{2\tau} \qquad (2)$$

where σ is the stress in the fibres at the load of interest; d is the diameter of the fibre, 100 μm; and τ is the effective shear strength of the matrix, assumed in this case to be 12×10^3 psi [13]. Unfortunately, since the strength of the fibres is length-dependent, the stress in them at failure of the composite σ_f, depends on the value of δ selected. However, both δ and σ_f can be calculated by substituting the above values of τ and d into Equation 2 and rearranging to give:

$$\delta/\sigma_f = 1.66 \times 10^{-7} \text{ in. psi}^{-1}. \qquad (3)$$

Accordingly, the mean strengths, $\bar{X}$, shown in Table III, were divided into the respective lengths of the individual fibres. The resulting ratios are shown plotted as a function of fibre length in Fig. 7. It can be observed that the appropriate value of the right-hand side of Equation 3 is obtained at a fibre length of 0.076 in. (0.19 cm) and 0.027 in. (0.07 cm) for the as-received and heated specimens respectively. The mean strength of fibres of these lengths is, from Equation 3, 458×10^3 psi and 163×10^3 psi.

By assuming that the bundle efficiency factors obtained for the longer fibres, as shown in Table IV, apply to these shorter lengths, bundle strengths, σ_{fb}, were calculated using Equation 1.

The upper bound of strength is given by adding the stress carrying capacity of the matrix at failure of the composite to the stress necessary to cause failure of a bundle with length equal to the ineffective length. Thus, in this work, the upper strength bound for the composite, σ_{cu}, was calculated by substituting the appropriate values into the rule of mixtures, i.e.,

$$\sigma_{cu} = \sigma_{fb} V_f + \sigma_m(1 - V_f) \qquad (4)$$

where V_f is the volume fraction of the fibres and σ_m is the stress in the matrix at the failure strain of the fibres. For this discussion work hardening of the matrix was considered negligible; thus,

TABLE IV Upper and lower strength bounds of Aluminium containing 20 and 50 vol % boron fibres

Vol % boron fibres V_f	Lower bound (10^3 psi)	Upper bound (10^3 psi)	Experimental mean value (10^3 psi)	Specimen condition
0.2	48.75	76.7	60.75	AR
0.2	16.0	28.86	23.2	H
0.5	121.87	176.75	138.0	AR
0.5	40.0	57.16	57.0	H

AR = as-received; H = heated 4 h at 600°C.
Upper and lower strength bounds for the 50 vol % material were obtained from fibres extracted from 20 vol % material.

$\sigma_m = \sigma(\text{yield}) = 10 \times 10^3$ psi (from previous discussion).

The upper bounds calculated using Equation 4 represent the strengths of bundles of fibres of length equal to the theoretical ineffective length, δ, plus the load-carrying capacity of the matrix. Since the bonding in a composite is sensitive to a variety of fabrication parameters, temperature, pressure, interfacial cleanliness, etc., the experimental value of δ would be more than the theoretical value. Thus, the values of the composite strength determined by experiment would be expected to fall between the upper and lower strength bounds.

A comparison of the mean strengths of as-received and heated specimens with the upper and lower strength bounds is shown in Table IV. It can be seen that the strengths of the majority of the as-received composites fall between the prescribed bounds. However, the strength of the as-received material containing the higher volume fraction of fibres approach the lower bound and the strength of the material containing the lower volume fraction approach the

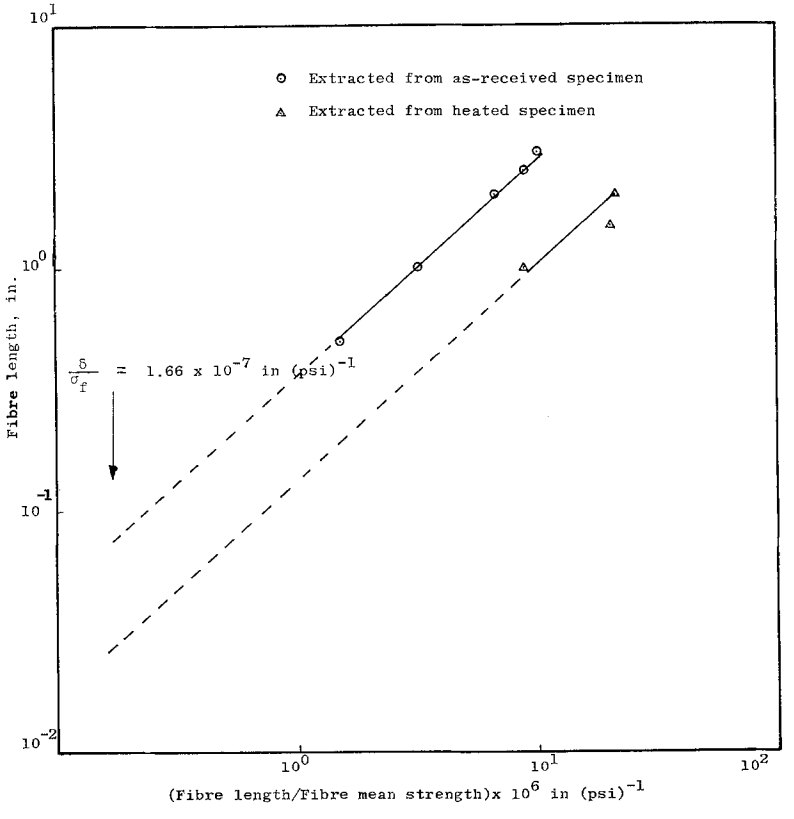

Figure 7 Variation of fibre length/fibre strength with fibre length.

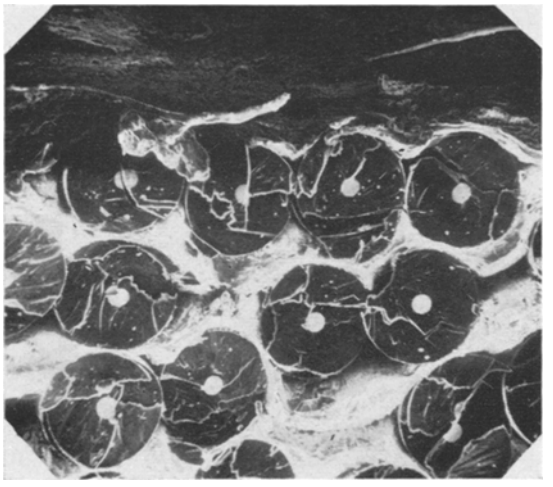

Figure 8 A 50 vol % B-Al composite showing discontinuities at the fibre-matrix interface, × 100.

upper bound. We believe that these results reflect differences in the fibre-matrix bond strength. Indeed, microscopic inspection of the material containing 50 vol % fibres indicated that the integrity of the bond was suspect. As shown in Fig. 8, the aluminium matrix did not completely surround each fibre, and many voids and points of fibre-fibre contact were present. In contrast, the matrix of the material containing the lower volume fraction of fibres appeared continuous and completely surrounded each fibre (see Fig. 4).

The strengths of heated composites also fall between the bounds of strength expected. Thus, it was concluded that the degradation in the strength of heated composites was, in the main, a direct result of a reduction in the strength of the reinforcement.

Also, for the low vol % material, at least, the experimental values obtained appear to approach the strength of a bundle plus the contribution of the matrix. The stress transfer function of the matrix was minimal. Thus, the strength of the bond between the matrix and fibre would appear to be degraded by the heat-treatment.

Acknowledgements

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