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The measurement of glass fibre strength in composites from studies of their fracture surfaces

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Scanning electron microscopy provides a convenient technique for the study of glass fibre-reinforced composite fracture surfaces. This work describes how a detailed examination and measurement of the "mirror zones" on the fracture surfaces of the fibres themselves can be used to evaluate the fibre strength at the time of failure of the composite. A mirror zone/fibre strength calibration for single E-glass fibres and alkali resistant (AR) glass fibres was made giving an inverse square root relationship for fibres down to 12 μm diameter and for strengths as high as 2000 MN m^{-2}. Using the calibration, fibre strengths *in situ* can be measured on failed composites and strength histograms compiled. The technique has primarily been used on fibre strength studies of glass reinforced cement composites and reinforced plastics.

1. Introduction

The study of fracture surfaces of brittle materials and the relationship of "mirror zone" measurements to the failure stress has had wide application, especially in the glass industry, for over 20 years. Originally "mirror zone" measurements and strength calibrations were made on flat glass, glass rods and thick "weak" fibres [1–3] using optical microscopy and the results used in investigations to estimate the strength of failed samples. The "focus" of the mirror zone was also used to locate the origins and causes of failure [4, 5].

While optical microscopy is suitable for measurements of large mirror zones on weak materials, high strength brittle materials have mirror zones too small to be measured accurately using the optical microscope. The scanning electron microscope (SEM), however, enables much more accurate identification and measurements of mirror zones and SEM techniques have been applied to studies of fracture surfaces of optical fibres [6–10]. In addition to measurements on optical fibres the present authors have been using the SEM to study the fracture surfaces of fine glass fibres (about 12 μm diameter) and, in particular, have used mirror zone measurements for the direct estimation of fibre failure strength in glass fibre-reinforced composite materials (e.g. glass fibre-reinforced cement and plastics).

This paper describes the calibration of the mirror zone/strength relationship for glass fibres of 12–20 μm diameter and its application to the measurement of glass fibre strengths in composite materials, with particular reference to glass fibre-reinforced cement.

2. Fibre strength – calibration

Levengood [1] showed that the square root of the mirror zone radius (r) is inversely proportional to the tensile breaking stress (S), i.e.

$$Sr^{1/2} = \text{constant}. \quad (1)$$

†It is with regret that, since the acceptance of this paper for publication, we have learnt of the death of Mr S. C. Simmens.

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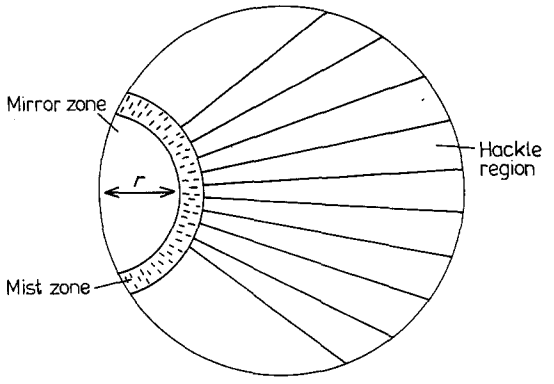

Figure 1 Schematic illustration of the fracture surface of a glass fibre.

The mirror zone radius (r) is measured from the fibre surface to the onset of the mist zone and is shown schematically in Fig. 1. Before any quantitative work could be carried out on either fibres or composites the mirror zone/fibre strength relationship had to be confirmed for fibre diameters down to 12 μm.

Single filaments of 12 μm Cem-FIL* AR glass fibres were made and short lengths mounted onto card frames for tensile strength measurements. At the centre of the 25 mm gauge length the fibres were purposely damaged by abrading with fine "Carborundum" paper to produce a range of fibre strengths. The abraded fibres were then tensile tested and the matching fractured ends mounted for examination in the SEM.

It was found that fibres with strengths greater than 2000 MN m^{-2} stored so much elastic energy that on tensile failure the fibres tended to break into dust so that original fracture surfaces could not be found. However, fibres of lower strengths often broke in such a way as to leave the complete length of fibre in two matching halves with matching "mirror zones". It was these fibres that were selected for the mirror zone calibration work. An example of a fibre fracture surface is shown in Fig. 2.

Mirror zone measurements were made on both matching halves at a magnification of 10 000×. At the same time the diameter of the fibre was measured and this value used in the fibre strength calculation of the tensile test result. An average value of mirror zone radius was calculated from both the matching halves of the fibre break. At a magnification of 10 000× the mirror zone radius was normally measured to within ±0.025 μm.

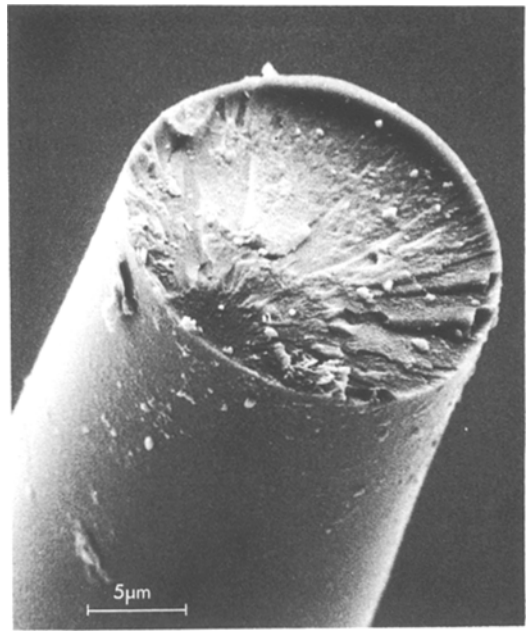

Figure 2 Fracture surface of single glass filament showing the mirror zone.

Slight loss of image resolution in determining the onset of the mist zone could increase this error to ±0.05 μm. Mirror zone radii measurements were made over a range of fibre strengths (from 660 to 1950 MN m^{-2}) to enable the calibration graph to be constructed. Using Equation 1 $Sr^{1/2}$ = constant, Fig. 3 shows a plot of S against $1/r^{1/2}$ on the original results obtained for Cem-FIL AR fibre; (for this work the fibre strength is calculated in MN m^{-2} and the mirror zone radius measured in microns (μm). The line drawn through the points is a linear regression fit with a correlation coefficient of 0.95. Such a good fit confirms that Equation 1 holds for small mirror zones on glass fibres of 12 μm diameter, as well as for flat glass, glass rods and thick fibres as in [1–3, 7, 10].

A calibration plot for E-glass fibres (normally used in reinforced plastics) is shown in Fig. 4, (strength range measured 360–1550 MN m^{-2}). Here the correlation coefficient is 0.99. The values of the mirror zone constant for these two glasses calculated from the results are: Cem-FIL AR Fibres (zirconia–silicate glass): 2.37 MN m$^{-3/2}$; E-glass fibres (alumina borosilicate glass): 1.47 MN m$^{-3/2}$. A similar calibration has been carried out on thicker (150 μm diameter) alkali–borosilicate glass fibres over the strength range 200

*Cem-FIL is the trade mark for alkali resistant glass fibre manufactured by Fibreglass Limited.

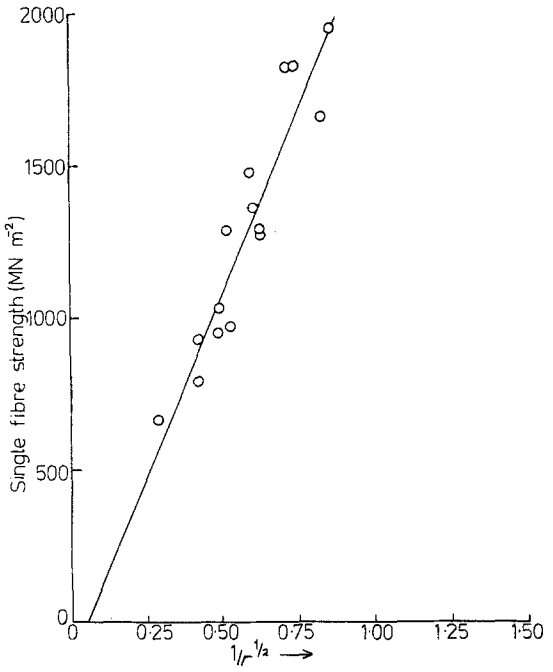

Figure 3 Cem-FIL AR glass fibre, mirror zone/strength calibration.

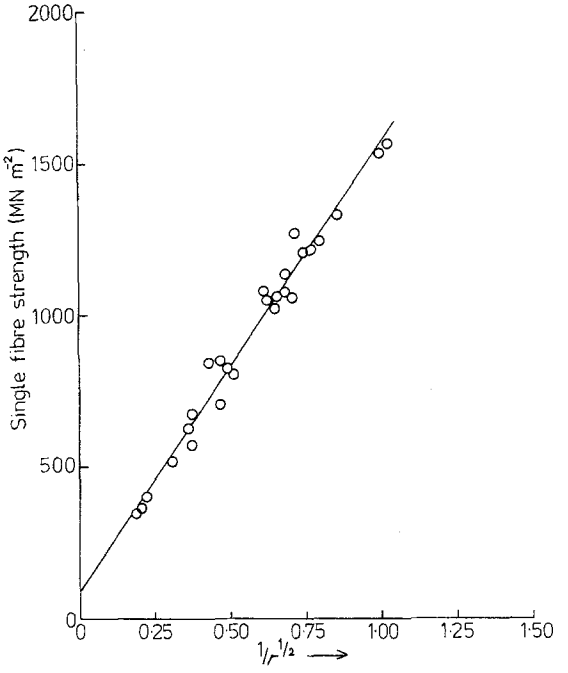

Figure 4 E-glass fibre, mirror zone/strength calibration.

to 830 MN m^{-2}. This gave a mirror zone constant of 1.33 MN m$^{-3/2}$ with a linear regression correlation coefficient of 0.99. In all cases the line does not pass through the origin.

These values of the mirror zone constant are lower than those previously published for similar glasses [11]. It is worthwhile here to consider some of the factors which may explain these effects.

The first one is of specimen size. Published results are generally based on relatively thick specimens compared with these results on thin fibres and the lower constants reported here may be due to specimen size effects.

The majority of the previous results calculate constants based on specimens broken in bending compared with these values calculated from tensile fractures. Lower constant values for tensile results have been reported and various explanations have been made to account for the differences [12–17]. The results here may reflect a difference between thick weak samples broken in bending compared with thin strong fibres broken in tension.

Physical property differences between the fibre and the bulk glass may be significant and should also be considered since glass fibres have lower densities and elastic moduli compared with bulk glass [18, 19], the effects of a lower modulus giving a lower mirror zone constant [20].

Most published constants are calculated from the mirror zones of relatively low failure stresses (i.e. <500 MN m^{-2}) and over a limited strength range. The mirror zone measurements in this work cover a wider and higher strength range and together with the high correlation coefficients can be considered to give more accurate mirror zone/strength values for high strength glass fibres than would be obtained with an extrapolation using low strength bulk glass constants.

Although these results confirm the inverse square root relationship, the linear regression fit through the results indicates a residual stress existing in the fibres. To account for this Equation 1 should be modified to

$$(S + a)r^{1/2} = \text{Constant} \qquad (2)$$

where a is the residual stress.

Orr, quoted by Johnson and Holloway [3], observed such an effect for toughened laths. Other authors have also modified Equation 1 to Equation 2 [21, 22], the validity of Equation 2 being later confirmed by direct measurement of mirror zones and photoelastic measurements on compressively clad glass rods [23]. In this work the value of a is calculated as below.

Cem-FIL AR Fibres: Residual stress $a = +137\ MN\,m^{-2}$
E-Glass Fibres: Residual stress $a = -81\ MN\,m^{-2}$
Alkali – borosilicate fibres: Residual stress $a = -44\ MN\,m^{-2}$

(+ = residual tensile stress, − = residual compressive stress).

These residual stresses could occur for a number of reasons, the most obvious being thermal stress due to differential rates of cooling between the surface and the interior of the fibre and stresses induced by "freezing-in" the fibre drawing tension. These factors have been considered for the drawing of optical fibres [24] and may be relevant to the glass fibres considered here.

Optical fibres rely on compositional differences between the fibre core and cladding. It has long been though that compositional differences exist as a thin skin on the surface of reinforcement fibres [25, 26]. The authors of this paper have, by direct electron microscope observation, seen that such a surface layer can exist (unpublished work) and other workers using surface analytical techniques have confirmed chemical compositional differences between the surface and the bulk of 12 μm diameter fibres [27]. These differences may contribute to the residual stress.

The generation of flaws on the fibre surface by mechanical damage may induce residual stresses into the glass surface. While not affecting the mirror zone relationship for 3 mm thick glass discs [28] there may be more significant effects on glass fibres. Thus all these effects either singly or combined may account for the residual stresses measured here.

3. Mirror zone studies in glass fibre reinforced composites

One of the most useful applications of glass fibre strength calculations from mirror zones has been in studies of composite materials and in particular examination of fracture surfaces of glass fibre-reinforced cement (GRC) composites.

The measurement of fibre strength in GRC composites has been limited previously to strength measurements of single acid extracted fibres [29, 30]. This extraction technique has four main disadvantages:

1. It is very laborious.

2. The exact location that the fibres had in the cement matrix cannot be easily determined.

3. In extracting fibres the possibility arises of preferentially selecting the stronger fibres only, since the very weak fibres would tend to break and stay in the matrix or be broken in handling.

4. The acid may attack the fibres and change their strength.

An examination of the fracture surface of a GRC test coupon (Fig. 5) shows that the individual position of each filament can be identified in relation to the fibre bundle and that the majority of fibres are capable of having their "mirror zones" measured in the SEM (Fig. 6). Thus it is possible to compare the fibre strength of fibres in different parts of the bundle.

Using the calibration graph and its extrapolation to high strengths (since many fibres, having mirror zones of 0.1 μm, can now have their fracture surface retained due to the damping effect of the matrix), it is possible to calculate the individual strengths for a number of fibres and to build up a histogram of results. Measurements can be made of the fracture surface of tensile, bend or impact test coupons, though allowances must be made for the different strain rates if an absolute value is required.

The results obtained from impact fracture surfaces of GRC under various stages of accelerated ageing compared with the strength histogram for a naturally weathered composite are shown in Fig. 7. From these histograms it is possible to study the change of strength distribution with time and the values of mean fibre strength can be used in composite strength calculations. In some instances, particularly where integral bundles of fibres are incorporated, it may be more appropriate to use a "strand" or "bundle" strength calculated from the histogram of results [31, 32]. This technique has made it possible to compare the strength changes in accelerated tests with those occurring in composites exposed to "real weathering". It has also been used to relate fibre strengths in large GRC components with smaller test coupons and with direct tensile strengths of strands in cement [33].

With a measured value of the fibre strength in the composite, together with a study of the bond interface [34–36] and knowledge of the general microstructural features of fibre filamentization, length, orientation and fibre volume content, it

Figure 5 Fracture surface of GRC composite showing fibre arrangement.

becomes possible to isolate the fibre strength when comparing different composites. An examination of the fractured fibre ends in the broken composites will establish whether they were fractured as the composite was broken, or if poor bonding has merely allowed the fibres to be pulled out without utilizing the available fibre strength. For example, if unusually low strengths are measured in GRC composites and the glass fibre content, fibre strength and fibre orientation is acceptable, then it is normally found that the fault lies in poor fibre/matrix bonding. Usually, this occurs because of inadequate cement curing and is evident as a high local porosity at the bond interface, rather than a smooth continuous high surface area of contact.

Although most of the studies over the past nine years have been made on glass fibre-reinforced cement composites, fibre strength measurements have been made on E-glass fibres in glass reinforced thermoplastic and thermoset materials, and strengths being calculated from the E-glass strength calibration graph (Fig. 4). This has been particularly valuable in identifying the amount of damage in composite manufacturing or extrusion processes, and detecting strength changes in GRP materials in corrosive environments.

4. Conclusions

By direct measurements an inverse square root relationship of mirror zone radius to fibre strength has been shown to be valid for glass fibres down to 12 μm in diameter and for strengths as high as 2000 MN m^{-2}.

Application of these measurements to glass fibres in composite materials and especially glass fibre-reinforced cement has enabled glass fibre strand strengths to be calculated *in situ* within the composite matrix and provides a means of gaining a deeper knowledge of composite properties and performance.

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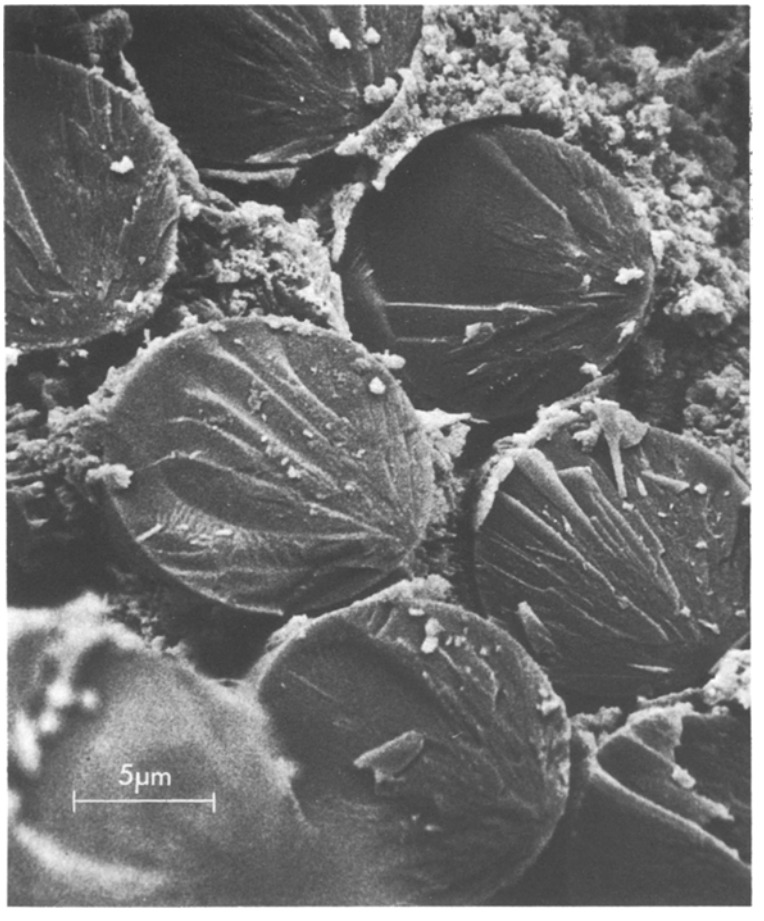

Figure 6 Mirror zones on individual fibres in GRC composite.

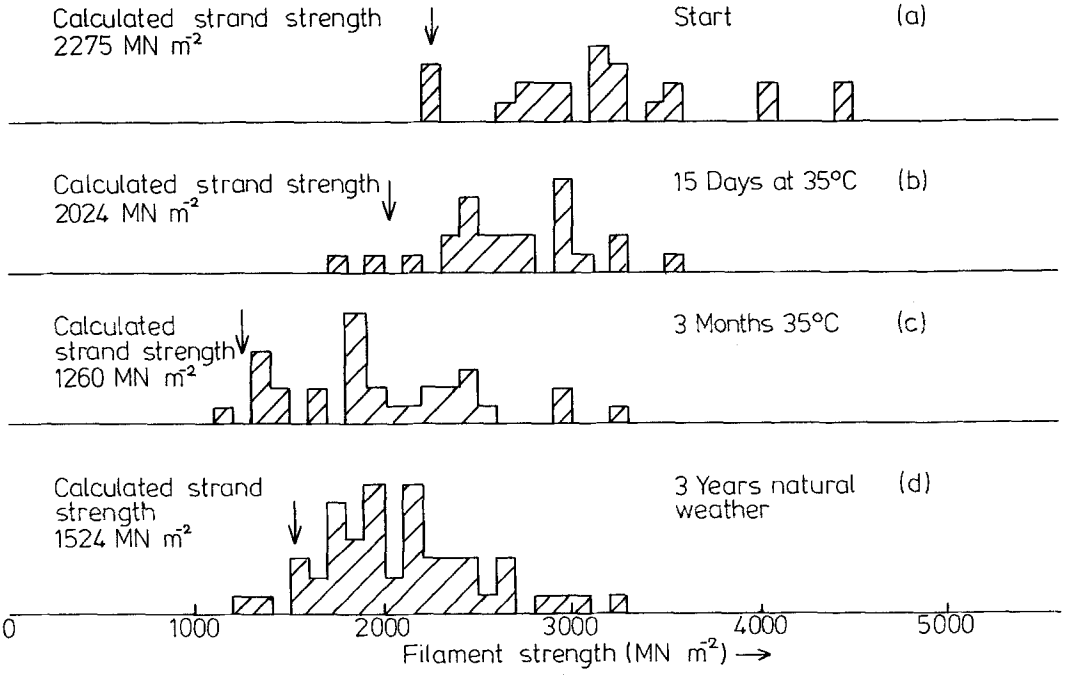

Figure 7 Strength histograms calculated from mirror zones.

Institute for the patient preparation of the SEM specimens. This paper is published with the permission of the Directors of Pilkington Brothers PLC and Mr A. S. Robinson, Director of Group Research and Development.

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