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Determination of the planar distribution of glass fibres in opaque matrix composites

D. L. RAYMENT, A. J. MAJUMDAR
Building Research Establishment, Garston, Watford, Herts, UK

A scanning technique using a photodiode has been developed for estimating the glass fibre volume fractions in composites with opaque matrices. The information is derived from light transmitted along the reinforcing fibres by total internal reflection. The method described has advantages over a previous method based on photographic techniques and it is possible to gain information about the uniformity of fibre distribution within the composite. Results obtained with glass fibre reinforced cement and plaster specimens are described. The transmission of light through the composite is proportional to the volume fraction of glass fibres up to a limiting value of ~6% Inhomogeneous fibre distribution can be detected using this technique. For cement samples the curing history is important as the degree of interaction between the cement and the glass has a marked influence on the glass transmittance.

1. Introduction

As in all fibre reinforced composites, the mechanical properties of glass fibre cement and plaster are strongly dependent on the volume fraction (V_f) and the angular distribution of the fibre strands in the composite. Small variations in these parameters are reflected in changes in the tensile strength of the composite and its stress–strain behaviour [1]. In commercial products made from glass fibre cement (GRC), the volume fraction of the reinforcement is rather small rarely exceeding 5% [2], and it is therefore desirable, for quality control purposes, to develop a simple but reliable method for estimating V_f and assessing the variations in fibre distribution in these composite materials.

Hibbert [3] has shown that glass fibre strands in a thick (~ 7 mm) section of GRC composites are capable of transmitting quantities of light that can be recorded with simple equipment even when the incident light intensities are relatively modest. A contact print or a photograph of the dark face of the specimen records the numbers of the light transmitting elements, which can then be used to count the number of visible strands in a section of the specimen. Using such a technique Hibbert and Grimer [4] were able to find a correlation between the variations in the number of visible strands in several GRC samples and their fatigue properties.

In Fig. 1, a typical print obtained from a 50 mm x 10 mm GRC specimen of a nominal thickness of 7 mm is shown. The photographic method of recording the light transmitting elements suffers from the disadvantage that any over-exposure, which is often required to detect some fibre strands, removes the details of fibre geometry in others so that groups of strands that lie close to one another cannot be resolved. Also, fibre bundles formed as a result of the disintegration of a single strand will be counted as individual strands if their brightness or size exceeds a certain threshold value. The threshold values are arbitrarily selected and therefore can lead to inconsistent results. Furthermore, with the type of counting used in the photographic method no account is taken of the orientation or the mechanical integrity of the fibre strands which are reinforcing elements in the composite. Both these factors affect the transmission loss of the glass strand.

An alternative method in which the measure-

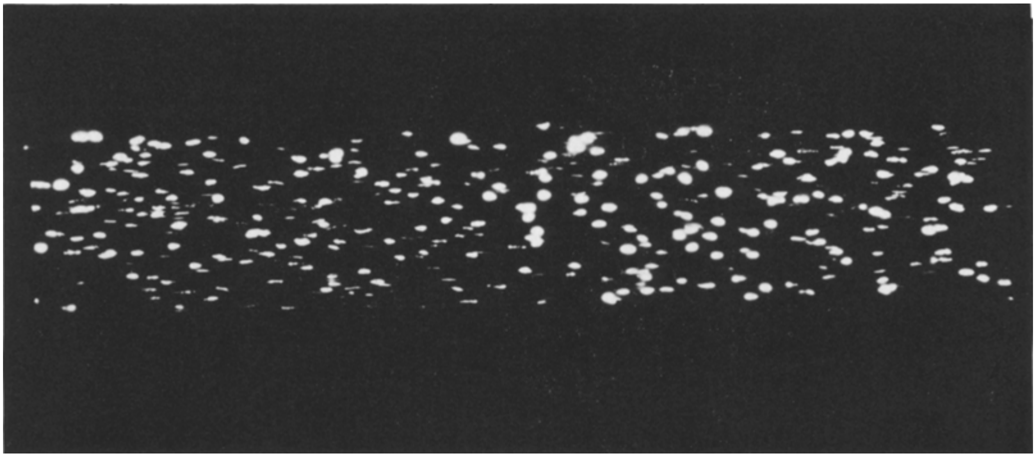

Figure 1 Contact print of a GRC section.

ment of the transmitted light is made using a photodiode scanning the specimen is described in this report.

2. Method

The principal features of the method are illustrated schematically in Fig. 2. Up to ten specimens are exposed simultaneously to a strong diffuse source of light. The light is transmitted through the essentially opaque specimen by the glass fibres by total internal reflection. The back surfaces of the specimens are scanned mechanically with a light guide/photodiode combination. The voltage output is amplified, then fed to a data logger and subsequently processed by a computer.

Fibre volume fractions, V_f, can be estimated directly from the print-outs after suitable calibration. Since in this analogue method the angular response of the detector assembly can be modified by changing the light guide or altering its position, the technique lends itself to an estimation of the angular distribution of the fibres in the composite. Any changes in the transmission efficiency of the fibres (say due to the ageing of the composite) can also be readily detected.

"Photoflood" tungsten lamps are used for

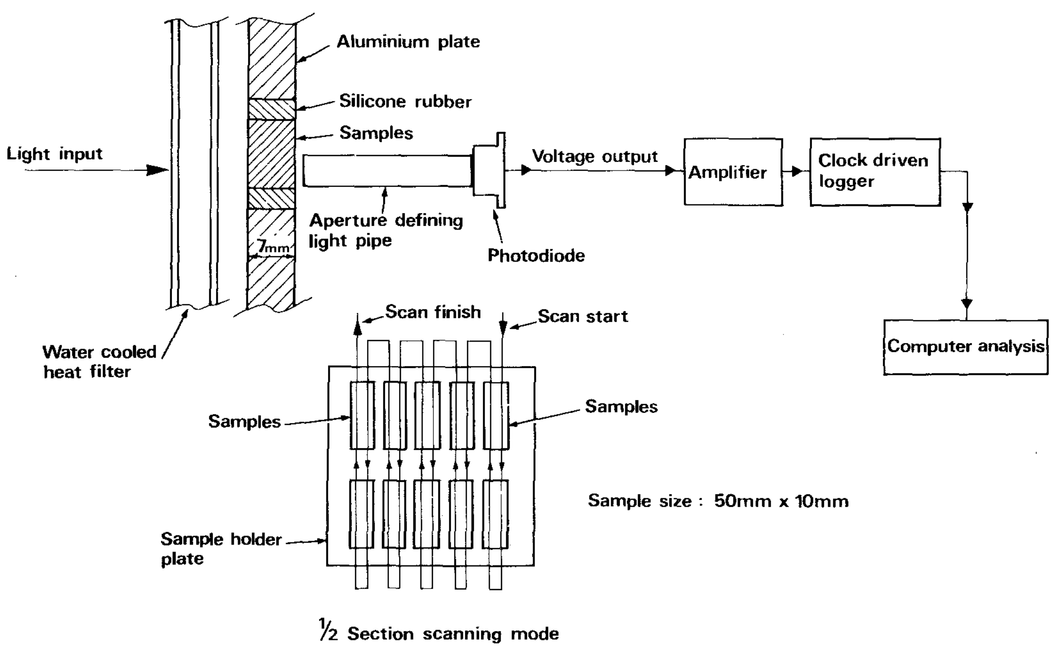

Figure 2 Schematic diagram of the photodiode scanning method.

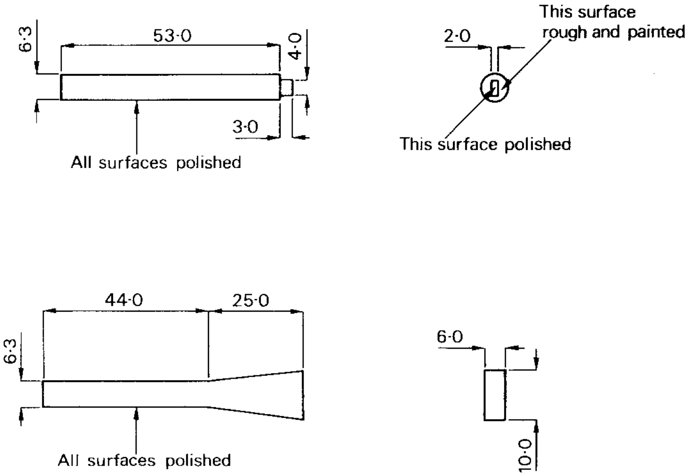

Figure 3 Large and small aperture light guide. All dimensions are in mm.

illuminating one face of the GRC specimen and the perspex light guide, which controls the defining aperture and the angular response of the light measuring system, is held at a distance of ~1 mm from the other face. Two of the light guides used in this study are shown in Fig. 3.

The light guide is optically coupled to a silicon photodiode having an active area of ~5 mm^2 and the output is amplified conventionally with a high impedance (>10^{11} Ω) FET d.c. amplifier. The photodiode can be operated in either the photoconductive mode with a 12 V bias voltage or in the photovoltaic mode. The mechanical scanner is driven at a single speed by a synchronous motor. The output from the photodiode is continuous but is recorded on paper tape for analysis. The rate at which it is recorded is synchronized with the scan rate so that a non-overlapping series of measurements can be made of the specimen transmission. Different light guides can be used to suit the requirements of individual experiments.

3. Experimental

Suitable specimens were prepared from 150 mm x 10 mm GRC coupons which had previously been tested for strength following their exposure to different environments for specified periods of time. The coupons were fractured first and integral sections with parallel faces were cut with a water fed diamond circular saw from as near the fracture path as possible and perpendicular to the long axis of the coupons. The specimens measured approximately 50 mm x 10 mm and their thickness (minimum section transmission length) lay in the range 7.12 ± 0.18 mm. They were stored in a CO_2 free atmosphere and allowed to equilibriate in the laboratory environment before examination.

The GRC coupons used in this study were obtained from composite boards produced in the laboratory by a spray-dewatering method [2]. In this method of production total isotropy in the plane of the board is not attained. This leads to variations in the board properties which depend upon the angle between the test and board axes. Inhomogeneities in the glass distribution through the thickness of the board result in "top and bottom" variations in the coupon properties [1, 5]. Unless otherwise stated, all GRC sections used in this work were cut and viewed in the same manner with respect to the facture zone, coupon base and saw direction.

3.1. Estimation of V_f

Two series of specimens, one prepared from coupons stored in air (40% r.h. and 20° C) and the other from those kept on the weathering site at Garston were investigated in the present work. The GRC coupons were three years old at the time of the examination and their nominal V_f ranged from 2 to 8% (nos. 1, 2, 3 and 4; Table I).

The GRC section was scanned over its entire width with a 6 mm thick light guide moving along the long axis of the section (see Fig. 2). In one traverse signals from 8 areas, each 6 mm x 9 mm could be collected although for the purpose of eliminating end effects only 6 inner areas were analysed. The end effects are those caused by the light guide aperture at the first and last measurement not being completely over the section. Allowing for a reversal in the scanning direction of the diode and a repeat of the run, 24 readings were

TABLE I Details of boards examined

Number	Description	V_f (%)	Fibre length (mm)	Curing	Remarks
1	OPC + AR-glass fibre	2.1	30	3 years in air and natural weathering	
2	OPC + AR-glass fibre	4.4	30	3 years in air	
3	OPC + AR-glass fibre	6.3	30	3 years in air and natural weathering	
4	OPC + AR-glass fibre	8.2	30	3 years in air and natural weathering	
5	OPC + AR-glass fibre	4.1	32	2 years in water	Coating applied on the fibre different from that of board nos. 1–4 and 8
6	OPC + AR-glass fibre	4.1	32	13 months in air	
7	OPC + AR-glass fibre	4.9	30	1 year in air	
8	OPC + AR-glass fibre	4.2	30	1 year in air	Two populations, one longitudinal and the other transverse
9	OPC + AR-glass fibre	6.2	32	7 days in air	
10	OPC			In air, water and natural weathering up to 1 year	MOR results from a large number of samples analysed
11	OPC				
12	Gypsum plaster + E-glass	5.4	22	In air	Age of the sample not important as fibres do not react with the matrix
13	Gypsum plaster + E-glass	5.4	43	In air	

OPC = Ordinary Portland cement
AR-glass fibre = Alkali resistant glass fibres supplied by Fibre Glass Limited. Not all the boards in the Table were produced from AR-glass fibres from the same batch.

recorded per specimen. An average of these readings were taken to represent the "transparency" of the section.

3.2. Estimation of composite anisotropy and inhomogeneity

3.2.1. Top and bottom effect

While determining the bending strength of GRC composites made by the spray-suction method it is frequently observed that the strength of the coupons varies systematically depending on whether the top or the bottom face of the composite is in tension during testing. The bottom face is defined here as that face which is in contact with the suction bed during fabrication while the top face is the one exposed to the air.

Coupons from 4 boards (nos. 5 to 8; Table I) were used in this study. Preliminary results had indicated that board no. 8 displayed "longitudinal and transverse" variations (see Section 3.2.2) in strength as well as a top/bottom effect and coupons from both these populations (longitudinal and transverse) were selected for examination.

The specimens were tested in four-point bending using a span of 135 mm. Half of the specimens belonging to any particular group were tested with their top face up (putting the bottom face in tension) and for the other half the bottom face was in compression as the specimens were tested bottom face up. The modulus of rupture (MOR) values were calculated using elastic beam theory.

Tested coupons from boards nos. 5 to 8 were selected in the standard manner as described previously. The light transmission from these sections were collected by a light guide having a 4 mm × 2 mm aperture. Two scans, covering the top and bottom halves of the specimen respectively, were made and each section was examined at least twice in this way. The light guide was placed with its edge as close to the section edge as possible leaving a narrow region in the centre of the sample not scanned. The MOR results from a large number of neat cement coupons were examined in order to determine whether the top/bottom variation was inherent in the fabrication method.

3.2.2. *Longitudinal and transverse effect*

In the mechanized version of the spray-suction method [2] of producing glass fibre cement and plaster composites, the spray head moves to and fro across the width of the board in a continuous fashion while the frame housing the spray head advances along the length of the board in discrete steps. Test coupons obtained from these boards often show variations in strength properties which can be related to their orientation in the boards. Transverse coupons (i.e. those where the long axis is perpendicular to the length direction of the board) are found to be in general weaker in bending and in tension than longitudinal specimens. Preferred orientation and possibly non-uniform distribution of fibres could account for these variations.

Gypsum plaster and cement coupons were used in this part of the study. The plaster coupon from boards 12 and 13 (Table I) had been tested in tension. The cement coupon from boards 8 and 9 (Table I) had been tested in bending. Coupons from board 8 showed a "top and bottom variation" (Section 3.2.1), and these were therefore divided into two populations, top up and bottom up. The two populations each contained 12 coupons, 6 from the longitudinal direction and 6 from the transverse direction. One section from each was examined.

Two coupons, one longitudinal, one transverse, from board 9 were available. These showed the greatest differences in MOR. Two sections were taken from either side of the failure zone. For the plaster boards one section from each coupon was examined. Five coupons, three longitudinal two transverse, were taken from board 12 and four, two from each direction were taken from board 13.

All the sections (MOR and tensile) were scanned in the $\frac{1}{2}$ section scanning mode, Fig. 2. The light guide had an aperture of 4 mm × 2 mm. Measurements were then made in the top and bottom halves of the sections. In the tensile sections all the measurements were added to give the tensile zone transmittance. In the MOR sections the "tensile zone" measurements were added to give the tensile zone transmittance. The ratios of the longitudinal and transverse tensile zone transmittance T_{ZL}/T_{ZT} are plotted in Fig. 6 as a function of the longitudinal and transverse strength ratios.

4. Results and discussion

4.1. V_f estimation

The relationships between the average transmittance of representative GRC coupons stored in air (40% r.h. 20° C) and natural weathering conditions over a period of 3 years at Garston and the nominal V_f of the composite boards they were derived from are shown in Fig. 4a. In Fig. 4b the ultimate tensile strength (UTS) of these coupons are plotted against V_f.

It can be seen that in air stored samples the coupon transmittance increased linearly with V_f up to 6% and there was very little change after that. The UTS values, on the other hand, showed a continuous increase up to 8%. For weathered specimens, the transmittance of GRC having 6 vol% fibre was found to be about the same as that containing 2 vol% fibre while the UTS of these specimens showed the expected trend of an increase with increasing V_f.

The low transmittance of the weathered samples reflects the condition of the fibre produced by the interaction between glass and cement over a number of years in a "wet" environment. In this environment cement hydration products are formed in large amounts and they are deposited on the surface of the fibre as well as inside the strands. It appears that the transmission efficiency of the individual fibre units is so very seriously reduced by this process as to be totally insensitive to changes in V_f of the composite. The changes in the transmission of light along glass rods embedded in cement has been the subject of a recent study by Leach and Ashbee [6].

The relatively low transmittance values from coupons having 8% fibre are not easily explained. It is known [1] that considerable difficulties are experienced in the production of GRC composites having more than 6 vol% fibre by the spray suction method. The fibre distribution in these composites is rather non-uniform and the attendant porosity is also higher than expected. For the 8% GRC composite it is conceivable that the local V_f in the sections of the GRC coupons examined were substantially less than the nominal 8% credited to the composite board. Alternatively, the increased porosity in the high V_f section may also be respon-

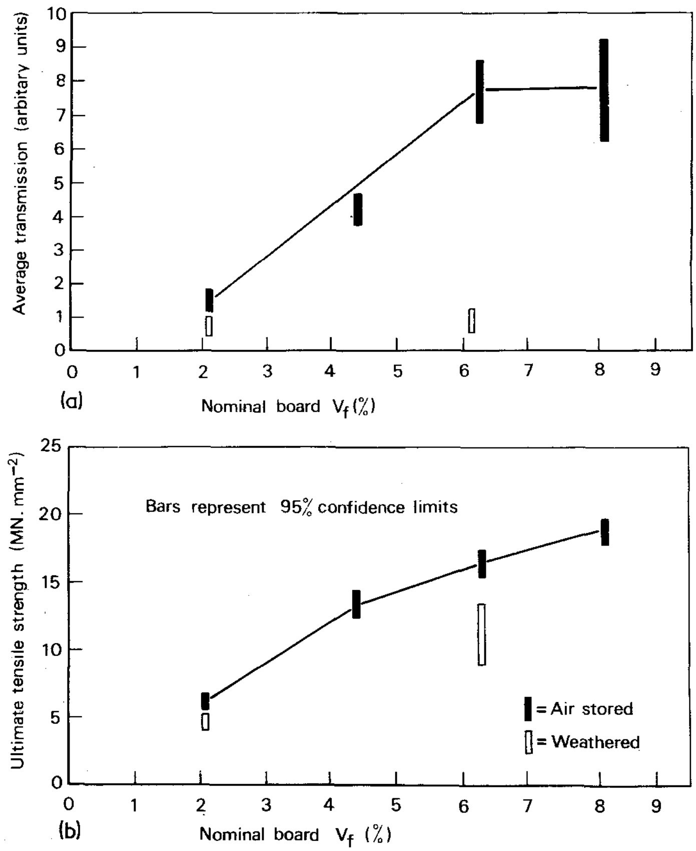

Figure 4 (a) Variation of section transmittance of GRC coupons with fibre content. (b) Variation of strength of GRC coupons with fibre content.

sible for the reduction in the transmission efficiency of the glass fibres. The supersaturated solution of $Ca(OH)_2$ formed by the hydration of cement fills the pores in GRC during the initial curing period and with increase in porosity it is likely that the interaction between this solution and the fibre surface will proceed to a larger extent. However, these are tentative suggestions and further work in this area is warranted.

The coefficients of variation in the average transmittance values for coupons having the same nominal V_f were large, 20 to 50%. They were also large for the transmittance data from the 6 mm x 4 mm area measurements, lying between 8 and 52%. The average transmission values recorded for the same section in different runs were less variable, the percentage difference between two runs for 24 specimens being 11.47 ± 3.37%. This is a result of misalignment between scanned areas. Large variations in both coupon and section transmittance are believed to be due to a non-uniform distribution of fibre. In order to obtain meaningful results a large number of samples would need to be analysed. Even with all these limitations it appears that the method here can be successfully used in the estimation of V_f in GRC provided a suitable calibration plot as that shown in Fig. 4a is available for the same type of material.

4.2. Composite anisotropy

4.2.1. Top and bottom effect

The information gathered in this study on the "top and bottom" effect is summarized in Fig. 5 in terms of two ratios, T_A/T_B and S_A/S_B. T_A and T_B were the experimental values of the average integrated transmission for the top and bottom halves respectively of GRC coupons belonging to the same composite boards. S_A and S_B refer respectively to the corresponding MOR values obtained when the top and the bottom faces of the coupons were in compression in the bend test.

For three boards, nos. 5, 6 and 7 the T_A/T_B were 1.90 ± 0.73, 1.37 ± 0.26 and 0.75 ± 0.17 respectively and the student *t*-tests showed that these values were significantly different from unity at the 95% level. The same was found to be the case for boards nos. 8a and b. A linear least-squares fit to the data gave $T_A/T_B = 1.88 - 1.67\, S_A/S_B$.

It should be noted that the regression line in

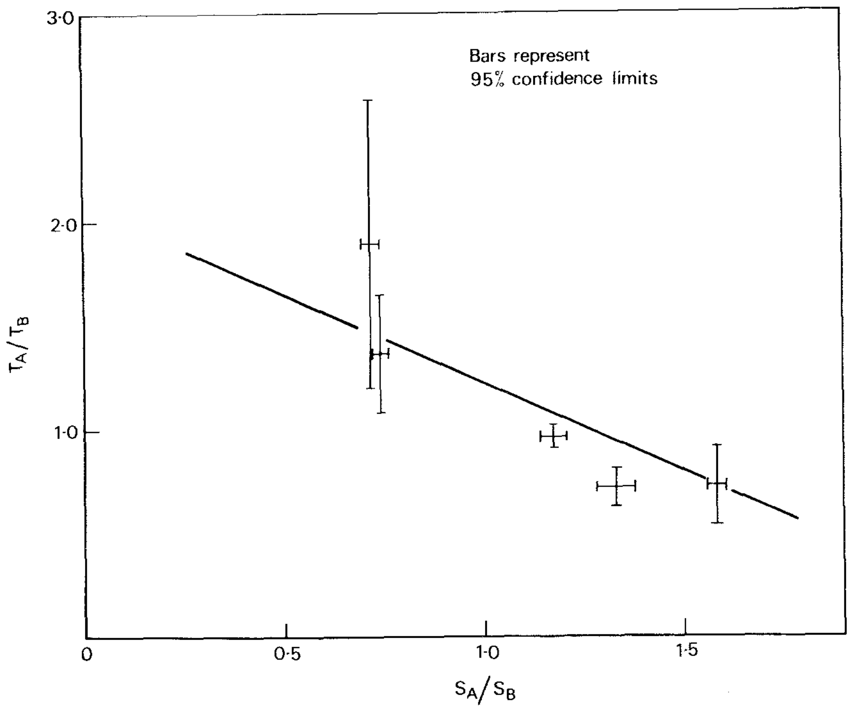

Figure 5 Top and bottom effect in GRC composites.

Fig. 5 does not pass through (1,1) as expected. This is believed to be due to subsidiary "top and bottom" effects caused by factors other than differences in V_f in the top and bottom halves of the GRC coupon. Experiments with plain cement coupons (19 groups of six coupons each from two boards; Table I) showed "top and bottom" differences in the MOR at the 95% significance level in only 3 out of 19 cases. By and large therefore the matrix in GRC does not contribute significantly towards these systematic "top and bottom" differences in the bending strength of the composite.

It can be seen from Fig. 5 that the 95% confidence limits are wide in some cases, the maximum coefficient of variation computed for the series being of the order of 35%. It is important to point out however that among the 43 average transmittance values used in the construction of Fig. 5 only 4 results were inconsistent with the trend in the measured values of S_A and S_B. It can therefore be concluded that the present method is capable of predicting the face which, when placed in tension, gives the highest MOR.

4.2.2. Longidutinal and transverse effect

The ratios of the measured average transmissions T_{ZL} and T_{ZT} for the longitudinal and transverse specimens of glass reinforced cement and plaster specimens are plotted against the corresponding strength ratios in Fig. 6. The strengths referred to

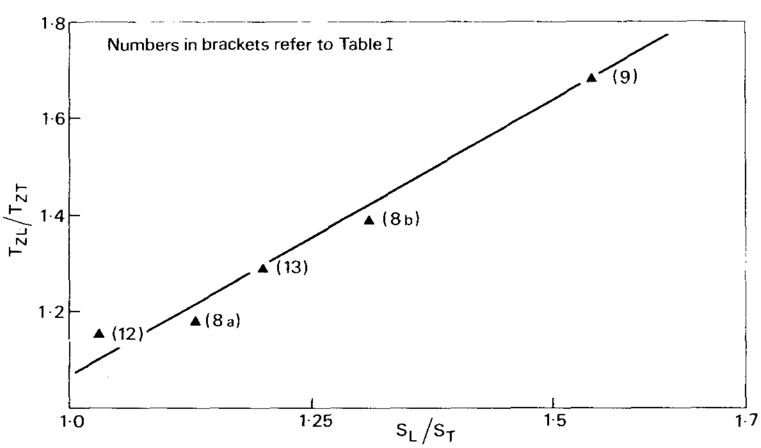

Figure 6 Longitudinal and transverse variations of GRC properties.

here are tensile for boards nos. 12 and 13 and MOR for nos. 8 and 9. The line of best fit can be represented by

$$T_{ZL}/T_{ZT} = S_L/S_T \cdot 1.07 + 0.009$$

It is not entirely clear whether the differences in the strength of longitudinal and transverse specimens of GRC are caused by increased apparent V_f due to an alignment of fibres in certain directions. Whatever the reason it appears that the present method is capable of detecting this anisotropy.

5. Conclusions

(1) The photodiode scanning technique of measuring the transmittance of glass reinforced cement and plaster sections can successfully replace the photographic method for obtaining quantitative information on fibre volume fraction in the composite. With the technique described measurements can be made independent of personal judgement.

The speed of measurement of the apparatus used for the work in this paper was limited by the data logger used. An advanced design of the apparatus has been constructed having an analysis time of 30 section min^{-1} in the $\frac{1}{2}$ section scanning mode. For cement composites the method as developed at present is restricted by the glass/cement interaction setting an upper limit on the age of wet or weathered samples that can be analysed. For air stored or young specimens this difficulty does not arise. From a practical point of view this is not a serious disadvantage as the principal application of this method of assessment is believed to be in the field of quality control during the production of components.

(2) The present method can easily detect certain types of anisotropy in composites that arise from significant local variations in the proportion and/or orientation of the fibre.

(3) Although the examples chosen here are those of cement and plaster composites it is believed that the method is applicable to any opaque matrix composite where glass fibres are used as reinforcement. For GRC a limit in the usefulness of the technique is implied by the results obtained in this study. This is not attributable to the method but to the characteristics of the glass fibre/cement system. With composites prepared from other matrices the usefulness of the technique may be extended both in the direction of high V_f and, since there are very few matrices as reactive to glass as Portland cement, to older samples.

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