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Properties of glass fibres in cement environment

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Fibres produced from a soda–silica–zirconia glass were reacted with Portland cement extracts at 20 and 65° C for various lengths of time and their strength and stiffness determined. The results indicate that these glass fibres resist the attack of cement extracts reasonably well at ambient temperatures. Fibre strengths of the order of 1200 to 1300 N mm^{-2} are obtainable after 2 years at 20° C, sufficient to reinforce cement, and there is no change in the Young's modulus of the fibre during this period. At higher temperatures both strength and stiffness are reduced but these temperatures are unlikely to be encountered in practice over extended periods of time. When fibres removed from cement composites containing commercially made alkali-resistant glass fibres are examined, it is found that fibre strengths depend very strongly on the environment in which the composites were kept. For air storage, fibre properties remain relatively unaffected but for composites kept under water continuously, an initial loss in fibre strength is observed. This difference in fibre strength is reflected in the relative strength of the cement composites.

1. Introduction

In recent years the reinforcement of cement and concrete by glass fibres has attracted attention [1, 2] and special alkali-resistant (AR-) glass compositions [3] have been developed for this purpose. Such a composition, containing a fairly large proportion of ZrO_2, is commercially available from Fibreglass Ltd under the trade mark Cem-FIL. The mechanical properties of Portland cement composites incorporating this particular fibre have already been subjected to detailed investigations [4, 5]; some information on their long-term behaviour and versatility in manufacture and use is also available [6].

In order to judge whether these cement composites will have an acceptable level of strength after say, 20 years, it is necessary to have some idea of the changes that might take place in fibre properties during this time. The present study was initiated to provide such information for AR-glass of the same type as that sold by Fibreglass Ltd using fibres which had been (a) in cement extracts, and (b) in cement composites. Some work was also done on asbestos fibres removed from weathered asbestos cement products.

2. Experimental

The experimental glass fibres used in the present work were of the composition (wt %); SiO_2 71, ZrO_2 16, Na_2O 12, and Li_2O 1. Single uncoated filaments were produced in the laboratory and these were reacted with aqueous cement extracts. Glass fibre-reinforced cement composites were produced by a spray-suction method [6] and kept in various environments for specified periods. The commercially produced AR-fibres were then extracted from the cement composites and their strengths measured at specified intervals.

2.1. Glass fibre drawing

Continuous filaments of the zirconosilicate glass were produced using the single-tip furnace assembly described by de Vekey and Majumdar [7]. Some new attachments were developed during the course of the present study which facilitated the production of fibres of uniform diameters in

separate batch operations. The most important of these attachments is an arrangement, operated by a tachometer circuit, which displays the fibre drawing velocity on a meter reading linearly upto 12 000 revolutions of the winding drum per minute. The circuit includes an uncovered transistor mounted beneath the winding drum shaft which is so machined that light from a source above it falls on the drum only during half of each drum revolution. To calibrate the meter a simple stroboscope is mounted on the end of the motor shaft. Knowing the circumference of the winding drum it is easy to calibrate the display dial in $m\,sec^{-1}$.

In monitoring the rate of glass flow, the size of the nipple is much more important than the head of glass in the crucible and by trial and error it was found that a nipple having an internal diameter of 2.5 to 3.0 mm was satisfactory for the production of glass filaments of approximately 10 μm diameter.

Fibres were collected on a 150 mm wide drum made from PVC and having a circumference of 356 mm. The drum was mounted on a trolley which could be driven to and fro every 15 sec by a reciprocating arm. This spread the glass fibre pulled over ~80 mm of the width of the drum, evenly distributing any diameter variations and enabling greater amounts of glass to be drawn in one run. This method was used to prepare fibres for the cement extract work. Without disrupting the normal drawing of the fibre, the reciprocating arm could be uncoupled thus converting to a fixed system. When uncoupled the machine could be used to produce a series of separate bands of uniform diameter by moving the drum laterally in short jerks.

To avoid the need for continuous observation of fibres for accidental breakage, an alarm system was designed. This consisted of three thermistors (mounted 200 mm below the glass melt) which could be moved close to the fibre when required. The thermistors are mounted in series with a relay, a variable resistance and an electric power source. As the fibre is pulled, warm air drawn along the fibre heats the thermistors. The circuit is so adjusted that the relay is maintained in a "just closed" position during the fibre drawing. When the fibre breaks, the temperature of the thermistors falls and the relay opens, setting off the alarm.

The diameter of a fibre of given density is related to the weight of the fibre pulled per minute and the fibre drawing velocity. For a fibre diameter of ~9 μm, a table linking these parameters was drawn up from which it was possible to judge the correct fibre drawing velocity for a given output rate (Table I). This table was based upon preliminary records of the glass flow rate through a particular crucible. An example of the rate of fibre production for a particular run is also included.

2.2. Corrosion experiments

2.2.1. With cement extracts

To study the effect of corrosion on glass fibre strength, uncoated continuous filaments having a nominal diameter of ~9 μm were reacted with the aqueous extracts of typical ordinary Portland cements. These extracts were prepared by rolling polyethylene bottles containing mixtures of

TABLE I Fibre drawing velocity and production rates

Weight of crucible plus glass (g)	Time (min) after full drawing velocity has been reached	Usual rate of flow of glass ($g\,min^{-1}$) based upon previous runs	Velocity ($m\,sec^{-1}$)* required to draw fibre of 9.15 μm diameter	Weight (g) of fibre in 1 min samples during a run
115.4				
113.3 approx	0	0.368	35.8	
	6			0.366
	10	0.365	35.6	
	20	0.362	35.3	
	21			0.361
	30	0.358	34.9	
	34			0.359
	40	0.354	34.5	
	50	0.350	34.1	
	54			0.347
94.3	55 Stopped			

* Calculated from the usual flow rate and a fibre density of 2.60 $g\,cm^{-3}$.

cement and distilled water for a period of 16 days. Two batches of cements were used (nos. 737 and 739), and reactions with the glass fibre were carried out at 20 and 65° C over extended periods of time. The details of the compositions of the cements and the preparative procedures for the extracts have been given elsewhere [8].

After digestion and removal of the bulk of the extract, the fibres were treated with 2 N acetic acid, washed with water followed by a further treatment with 5 N hydrochloric acid. Finally, the fibres were washed with water and dried at room temperature and stored in screw capped bottles prior to testing.

2.2.2. With fibres from the composite

Commercially made AR-glass fibres were extracted from cement composite boards which were made by the spray suction method and stored in different environmental conditions for investigating their long-term durability [9]. The fibres were obtained from a portion, about 50 mm x 35 mm, cut from the main test pieces and split centrally in the plane of the board to expose the fibre strands. These segments were placed in cold (about 2° C) hydrochloric acid solution (conc $HCl:H_2O = 1:2$) for 10 min, rinsed in distilled water for a further 10 min and then dried on paper tissues for 20 min. The strands from the split faces, which by this time could be peeled away from the matrix, were then mounted on polyethylene frames, rinsed in acetone and dried. Several filaments (at least 15) were then removed from the strand, their diameter measured and immediately afterwards they were tested for strength on the balance rig (see Fig. 2).

In addition to those extracted from the cured boards, fibres were taken from the "green" board immediately after spraying. A specimen of the roving used in the production of the board was also stored in the laboratory in a polyethylene bag to serve as the reference material. Fibres from this sample and others described above were tested after stipulated intervals of time.

Two weathered asbestos cement boards were selected for obtaining strength data on reacted asbestos fibres. One board had undergone 4 years of exposure on site before it was stored in the laboratory for a further period of 2 years. With the other board the corresponding periods were 7 and 2 years respectively. Experiments were also carried out with fibres from an unweathered asbestos cement board. It should be appreciated that these three boards were not produced at the same time and possibly also not by the same manufacturer.

By placing 100 mm x 50 mm pieces of asbestos cement sheets edgewise between the jaws of a press and applying pressure, it was possible to cleave the specimens in such a way that fibre bundles could be removed from the composite. This was achieved by careful manipulation with a sharp needle and a pair of tweezers with flat smooth faces under a binocular microscope. After the removal of the adhering matrix a quantity of fibre bundles, not less than 4 mm in length and having overall diameter in the range 15 to 40 μm, were selected for strength determinations.

2.3. Property measurements

2.3.1. Fibre diameter

The diameter of the glass fibres before and after corrosion was measured with the help of a microscope provided with a Watson image shearing eyepiece. A 10 μm graticule was used for calibration. For routine measurements, two 4 mm wide strips of double-sided adhesive tape were attached to a microscope slide longitudinally leaving a blank space at the centre across which several ~25 mm long fibres were mounted transversely. Fibre diameters were measured near the tapes where they were securely held.

The estimation of glass fibre diameters in air by the above procedure when only the graticule is used as the standard is subject to some inaccuracies arising from the large differences in the optical properties of the fibre *vis á vis* those of air. It is desirable to compare these measurements with those of other more accurate but slower methods in order to obtain appropriate calibration. The use of the scanning electron microscope for this purpose has been attempted [10] but in the present work two other methods were used.

It is easy to see that fibre diameters can be calculated if the density, weight and length of the fibre can be accurately determined. Single filaments of glass were pulled and wound on the drum of the fibre drawing apparatus described previously in five separate but consecutive bands, each band being the yield from 1 min pulling. During this exercise the furnace temperature just above the crucible was maintained at 1540° C and the drum was rotated at a constant speed of 6000 rpm. Utilising a value of 2.60 g cm^{-3} for the

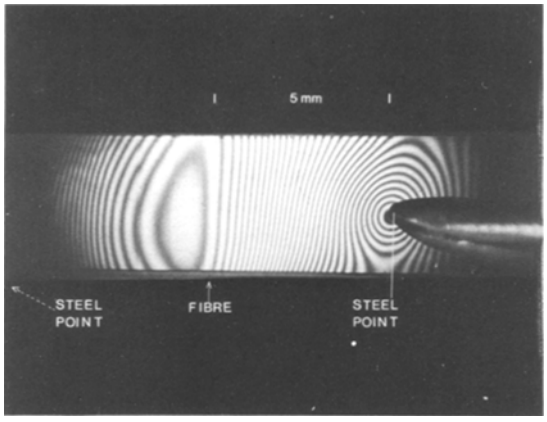

Figure 1 Interference fringe method of measuring fibre diameter.

density of the filaments, obtained by the density gradient column method [10], the diameter of the fibres were computed from the measured weight and length of each band. The coefficients of variation for diameter measurements ranged from 0.75 to 1.9% for individual bands.

The diameters of a large number of filaments from the above five bands were also measured using an interference fringe technique. The glass fibre was mounted transversely on a clean microscope slide and a 20 mm × 4 mm cover glass was placed on it with its length perpendicular to the fibre. A spring loaded pointed steel wire was screwed down on each end of the cover glass so that the cover glass arched over the fibre. The assembly was placed on the stage of a microscope and illuminated through the objective by light from a sodium lamp. A pattern of interference fringes was easily visible (Fig. 1). Ensuring that the coverglass was in contact with the fibre (and this was aided by placing the fibre non-centrally with respect to the two pressure points) the lesser number of fringes between the fibre and the pressure point was counted using a low power objective. The experiment was repeated several times and the diameter of the fibre was calculated from the formula $d = n\lambda/2$ where n is the smallest number of fringes counted.

For filaments taken from the five bands a mean value of 9.06 μm was obtained for the diameter by the density method and 8.87 μm by the optical method. An average of these two values was used in obtaining the calibration for the routine measurements with the Watson image shearing eye-piece. It is important to note that calibration by the graticule overestimated glass fibre diameters by about 10%.

The diameter of the asbestos fibre bundles was measured microscopically using the image shearing eyepiece. As the fibre bundle was very non-uniform in diameter along its length, it was necessary to design a special holder usable on the mechanical stage of the microscope, and provided with a facility by which the fibre bundles could be given a suitable amount of twist so that they had the appearance of a taut rope. Diameters were measured at selected points along the length of the fibre bundles where the visual edges were relatively free from imperfections. In general, six to ten measurements were made over a length of 2.5 mm.

2.3.2. Fibre breaking load

Breaking loads in tension were measured using the Tecam microtensile testing instrument [11] and the apparatus developed by Gillett and Majumdar

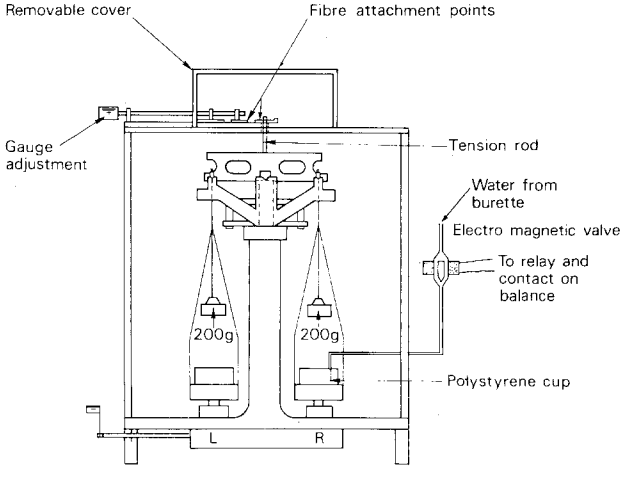

Figure 2 Fibre tensile testing apparatus.

[12]. Each test on the Tecam machine took more than 15 min to perform and the other method which was considerably faster (less than 2 min per test) was preferred when a large number of fibres had to be tested.

The essential features of the instrument developed by Gillett and Majumdar are shown in Fig. 2. A load applied to the balance pan is transferred by a rod set in the balance beam to the fibre mounted horizontally above the balance casing. The fibre is loaded by running water from a graduated burette through a flexible delivery tube into a polystyrene cup on the right balance pan. The rate of loading can be controlled by the jet size of the delivery tube when the burette tap is fully open. For the present work, a fibre gauge length of 5 mm was adopted and an electromagnetic valve was added which stopped the flow automatically when the fibre broke. When corroded glass fibres were tested using this apparatus, the tensile strength data showed a coefficient of variation of 5% or less.

The instrument was calibrated by mounting a glass fibre horizontally in the usual way with weights varying from 5 to 20 g suspended from it. This was achieved by leading the fibre over a free running pulley wheel mounted on the apparatus. This calibration agreed well with that computed from the relative moments about the pivot. The Tecam machine was calibrated in accordance with the procedure described in the manufacturer's handbook.

When glass fibres from the same batch were tested by both machines, it was observed that the strength results from the Tecam were consistently lower, by upto ~20%. It should be pointed out here that in the use of this machine the fibres have to be handled with great care and they are also exposed to temperatures exceeding 200° C when they are cemented to the anvils. Furthermore, compared to the other machine, the strain rate in the Tecam is considerably lower. It is likely that these factors are responsible for the observed discrepancy in the results obtained by the two methods.

The breaking loads for asbestos fibres were measured on the Tecam machine using the bundles which were previously employed in diameter measurements and which were still in the form of a taut rope.

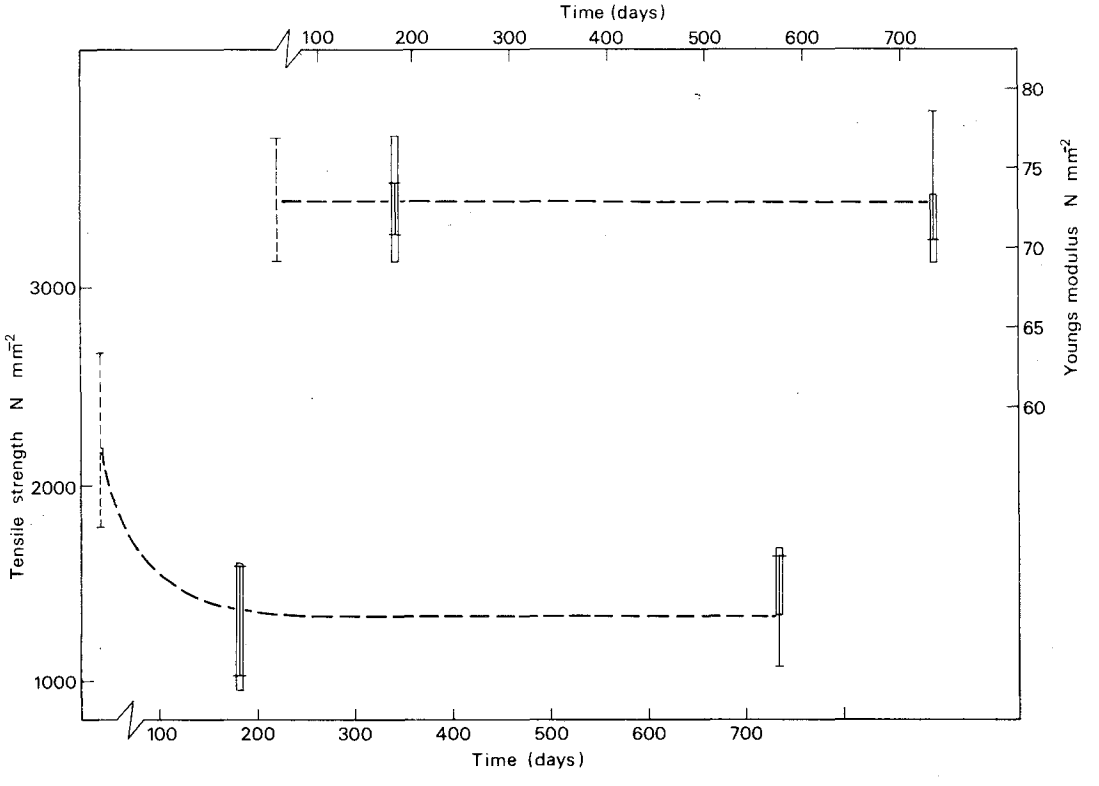

Figure 3 Tensile strength and Young's modulus of glass fibres reacted with cement extracts at 20° C. |– – –| pristine fibre; |——| fibre from OPC 737 extract; ▭ fibre from OPC 739 extract. The bars include ± one standard deviation.

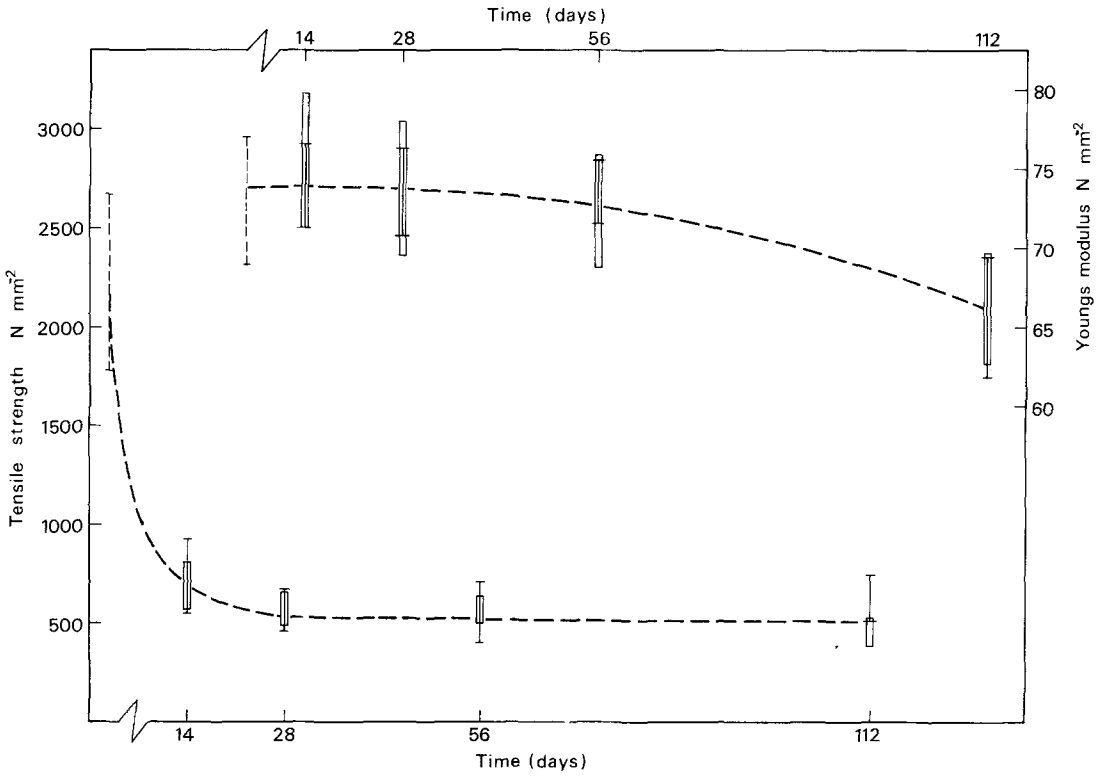

Figure 4 Tensile strength and Young's modulus of glass fibres reacted with cement extracts at 65° C. ⊢ – – ⊣ pristine fibre; ⊢——⊣ fibre from OPC 737 extract; ⊏⊐ fibre from OPC 739 extract. The bars include ± one standard deviation.

2.3.3. Young's modulus of glass fibres

With the Tecam machine it is possible to measure directly the extension suffered by the fibre under load. The Young's modulus values of glass fibres, both in the corroded and uncorroded state, were derived from the results obtained with this machine.

2.3.4. Composite modulus of rupture

The modulus of rupture of the composite was calculated, using homogenous beam theory, from the load data obtained with 150 mm x 50 mm x 9 mm specimens which were tested in four-point bending on an Instron machine.

3. Results and discussion

3.1. Glass fibre cement

The results of the various corrosion experiments relevant to the durability of glass fibre cement composites are shown in Figs. 3 to 6. From Fig. 3 it is seen that at ambient temperatures glass fibres lost significant proportions of their ultimate tensile strength (UTS) in course of the first 6 months when kept continuously in contact with Portland cement extracts. However, there is a firm indication that no further reduction in strength occurred up to 2 years at the end of which an UTS of $\sim 1300\,N\,mm^{-2}$ was recorded. The Young's modulus of the fibres remained virtually unchanged throughout the entire period suggesting that the structure of the bulk glass is not affected in a major way as a result of reactions with the cement extract.

At 65° C, a similar trend in durability is also observed (Fig. 4) but in this case, as would be expected, the initial reduction in strength was more pronounced and after 14 days digestion glass fibres lost nearly two thirds of their pristine strength. Again, there is a strong indication that a stable UTS value of the order of $500\,N\,mm^{-2}$ was reached after about a month. As expected, this value is considerably lower than the corresponding strength at ambient temperatures. The Young's modulus of the fibres also shows a slight reduction after prolonged exposure to the alkaline cement extracts at the higher temperature. It should be pointed out that the strength data presented in Figs. 3 and 4 were obtained with fibres which were

employed previously in chemical corrosion studies [8]. These fibres had undergone several stages of mechanical handling and chemical processing and it is conceivable that during these operations the surface of the fibres was "damaged" to a certain extent. This may partially account for the very substantial initial loss suffered by the fibres as depicted in Figs. 3 and 4. From these results, however, it is not possible to judge the time when such a reduction in strength first manifested itself as the earliest measurements of the strength of reacted fibres at 20° C were carried out after 6 months. It is interesting to note in this connection that Oakley and Proctor [5] have recently reported that the tensile strengths of strands of glass fibres of composition similar to that used in the present study are reduced from values in the range 1450 to 1750 N mm^{-2} to 1200 to 1300 N mm^{-2} after 24 h in a cement environment. Allowing for the expected differences in the strength of the strand compared to that of single filaments, these results are in accord with those shown in Fig. 3.

Cohen and Diamond [13] have recently reported that when placed in a "cement effluent solution" [3] at ambient temperatures, alkali-resistant glass fibre strand made in their laboratory was weakened only slightly over a period of some weeks. The authors speculated that the long-term strength of these fibres could be about 80% of the original values. These results are at variance with the results in Fig. 3 and the observation made by Oakley and Proctor [5] mentioned earlier with respect to the change in the strength of the glass fibre during the initial period of their life in the cement environment. It may be pointed out that the original strength of the fibre strand used by Cohen and Diamond was very low at 940 N mm^{-2} and it is certainly arguable that a much higher pristine strength might have shown a trend similar to that of Fig. 3 bearing in mind that glass filaments in the latter case were not externally coated as must have been the case with the strands used by Cohen and Diamond.

Cohen and Diamond rightly point out that the rate of fibre strength loss at say 50° C cannot be used in any speculation on glass fibre properties at room temperature. As far as the zirconsilicate glasses (of the type discussed here) are concerned, a detailed recent study [8] has shown that even at ambient temperatures ionic species are leached out of the fibres at a measurable rate. Some proposals have also been made in relation to the mechanism of glass/cement interactions for these glasses. However, it is not possible to predict the strength of glass fibres from these kinetic data as this is controlled mainly by the statistics of the size, population and distribution of the flaws in the fibre.

That alkali attack can promote serious reductions in the strength of glass fibres is demonstrated in several studies on E-glass fibres [6, 13] and by the observations that fibres made from A-glass become powdery after prolonged exposure to an alkaline environment. Zirconosilicate glasses are inherently alkali-resistant and once these glasses had been fiberized it was discovered that this property could be utilized successfully for the reinforcement of highly alkaline cements.

The tensile strength of Cem-FIL glass fibres after their removal from cement composite boards stored in air and under water over various periods of time is shown in Fig. 5. It should be noted that

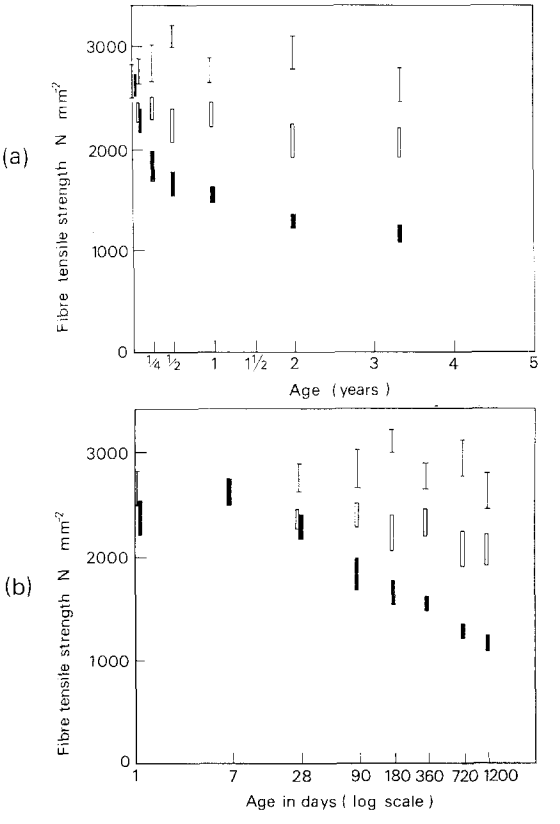

Figure 5 Tensile strength of alkali resistant glass fibres extracted from cement composites. |——| control (chopped fibre); ▭ fibres from air stored composite; ▬ fibres from water stored composite. (a) and (b) show the same strength results plotted against time and log time respectively. The bars represent 90% confidence limits.

in this experiment, some of the fibres remained firmly attached to the matrix and tended to break into shorter lengths during removal and, therefore, only a small proportion of these fibres were long enough for strength testing. The remaining fibres comprising mainly the interior of the fibre bundles were affected to a lesser extent in this respect.

For comparison, the modulus of rupture values obtained with glass fibre cement from which fibres were removed for testing, are plotted in Fig. 6, against the duration of storage of the composite specimens in the two environments mentioned above. The effect on fibre tensile strength of environment and time (Fig. 5) is reflected in the properties of the composite (Fig. 6). The physical basis for the strength time relationships, however, remains unknown for the time being and for this reason, and to avoid bias in interpretation, both logarithmic and linear time scales have been used to present the data in Figs. 5 and 6.

The Young's modulus of the fibres extracted from the composite boards was not affected significantly by the prolonged exposure to two different environments. There was also no noticeable effect of these factors on the diameter of the fibre. In these respects the properties of the fibres removed from composites were very similar to those of fibres reacted with Portland cement extracts at ambient temperatures.

Cohen and Diamond [13] have also carried out

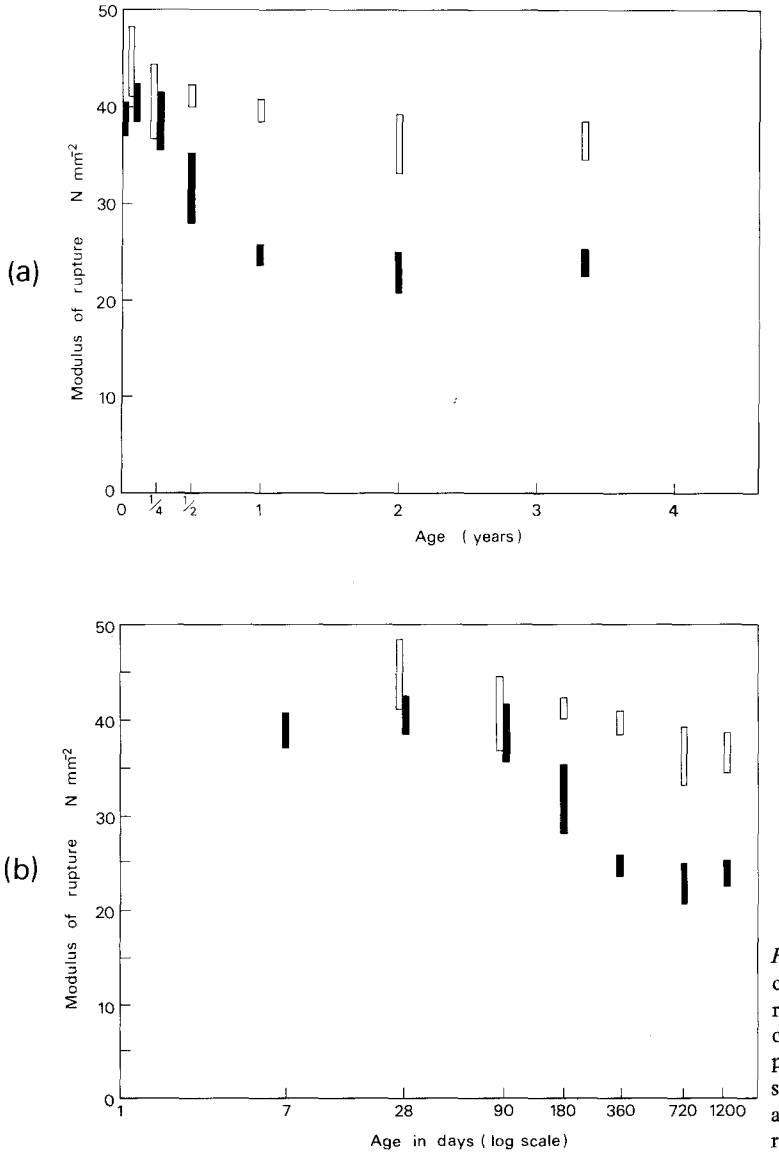

Figure 6 Modulus of rupture of cement composites containing alkali-resistant glass fibre. ▭ air stored composite; ▬ water stored composite. (a) and (b) show the same strength results plotted against time and log time respectively. The bars represent 90% confidence limits.

similar experiments with cement composites using alkali resistant fibres from an American source. They removed fibres from composite specimens stored in air at 22° C and 50% r.h. and measured their strength after various periods of ageing and observed that no reduction in strength had taken place. Unfortunately, not all their results were obtained from the same composite board. The corresponding results of the present investigation also obtained with specimens kept in air (but at 20° C and 40% r.h.), show a small reduction in the strength of the fibres over a period of 3 to 4 years. Therefore, it can be said that there is a qualitative agreement between the two sets of results although the magnitude of fibre strengths measured in the two laboratories are very different. The long-term flexural strengths of the composites studied by Cohen and Diamond are, however, rather low, $\sim 5\,N\,mm^{-2}$, and arguably this value is not much higher than that of the matrix. In that case it is difficult to see how the constancy of strength at this level with time can be cited as evidence for the lack of deterioration in the fibre.

In the present study, fibres extracted from water-stored composites showed a considerable reduction in strength with time (Fig. 5). When stored under water the cement phase undergoes continuous hydration releasing, proportionately, much higher volumes of alkaline solutions as well as crystalline $Ca(OH)_2$ and C–S–H "gels" than is possible during curing in air of normal humidities. (In the latter case, even after several years, much of the cement remains unhydrated.) Thus the observed reduction in fibre strength may well be due to the combined action of the solution and the solid phases.

Finally, it should be mentioned that work in this laboratory suggests that in water storage conditions, composites made from alkali-resistant glass fibres are more durable with aluminous and other cements less alkaline than their Portland cement counterparts.

3.2. Asbestos cement

The strength values for asbestos fibre bundles presented in Fig. 7 are considerably lower than those reported by other workers. For instance, Aveston [14] gives values of 3000 to 4400 $N\,mm^{-2}$ for the UTS of chrysotile asbestos. Klos on the other hand, has quoted a range of 560 to 750 $N\,mm^{-2}$ in a recent review [15] for chrysotile used commercially in the asbestos cement industry. As mentioned in a previous section the measurement of diameter of asbestos fibre bundles poses very serious problems. Aveston estimated the diameters of the "fibres" from the known density of the fibre and weighing a quantity of it on a very sensitive microbalance. Such a method could not be used in the present case for the following reasons:

(1) the fibres used in the commercial boards were not of one type but consisted of a mixture of blue and white asbestos,

(2) the fibres could not be freed completely from the adherent matrix and,

(3) the length of the fibre used on the Tecam

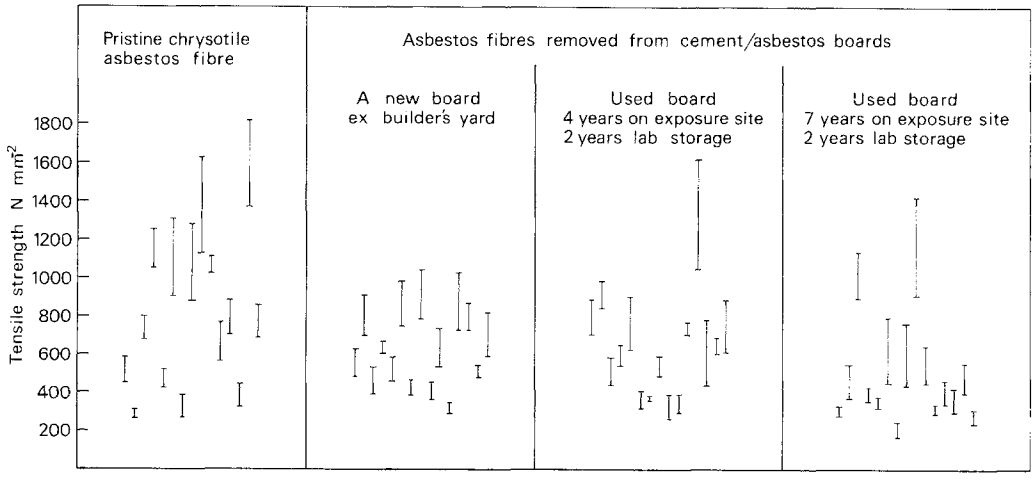

Figure 7 Tensile strength of asbestos fibres. Fibre bundles twisted to form a "taut rope" before measurement of diameter. Bars represent values based on minimum to average diameters.

machine was so short that a microbalance of very great sensitivity would have been required for weighing the fibre lengths. Such an instrument was not available. Instead an attempt was made at obtaining a realistic figure for the diameter of the fibre bundle by direct measurement.

In the present study, the change in the strength of the fibre bundles with time, on a relative rather than absolute basis, was the more important information to gather. In this respect, the values in Fig. 7 suggest that as the asbestos cement gets older in use, the fibre strength might show a small decrease. The scatter in the data in Fig. 7 is such that it is not possible to be precise about the extent of this decrease. From these values of asbestos fibre strength at various ages, it will be expected that the tensile and flexural strengths of asbestos cement sheets do not suffer significant reductions with age. Such a conclusion was reached by Jones [16] with respect to the modulus of rupture of asbestos cement roofing sheets exposed to weathering.

Very recently, Opoczky and Pentek [17] have examined some very old (up to 58 years) asbestos cement sheets by mineralogical methods such as petrographic microscopy, differential thermal and thermogravimetric analyses. They have concluded that some chemical reactions between asbestos fibrils and hydrated cements take place under natural weathering conditions. There is no indication, however, that these "corrosion" reactions which are aided by atmospheric CO_2 have any influence on the tensile strength of the fibre or the composite. The present work adds support to this view.

4. Conclusions

Glass fibres lose a proportion of their pristine strength when placed in a Portland cement environment. The factors responsible for this reduction remain unknown in precise terms but chemical attack by alkalies must be one of the more important ones. Recent studies confirm that the superior performance of zirconosilicate glass fibres as reinforcement for cements *vis á vis* other glasses is linked with the inherent alkali-resistance of this glass. At ambient temperatures glass fibres of this type are likely to possess long-term tensile strengths of the order of 1200 to 1300 $N\,mm^{-2}$ when placed in cement environments.

Present evidence suggests that there is no significant reduction with time in the strength of the asbestos reinforcement in asbestos cement products.

Acknowledgement

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