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Fracture measurements on cement paste

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This paper describes an investigation into the fracture behaviour of hardened cement paste. Notched specimens of the material were tested to failure in flexure and tension. In the initial flexural tests on beams of fixed overall depth, the stress intensity factor at failure as calculated from linear-elastic fracture mechanics appeared to be a material constant. However, further investigation showed that this factor varied with specimen size, and suggested that linear-elastic fracture mechanics and the concept of fracture toughness are not readily applicable to hardened cement paste, which would appear to be a relatively notch insensitive material whose strength is not greatly reduced by the presence of flaws. A "tied crack" model explains semi-quantiatively the observed behaviour.

1. Introduction

Concrete is an extremely cheap, readily available material with many desirable properties. One very undesirable property is its low tensile strength, normally around $5\,\mathrm{MN\,m^{-2}}$, which compares very poorly with materials like steel (tensile strength $\sim 1000\ \mathrm{MN\,m^{-2}}$). Even relatively weak materials such as wood have tensile strengths $\sim 100\,\mathrm{MN\,m^{-2}}$. Since experience with other materials (e.g. steel and glass) has shown that a good understanding of the processess involved in the failure can often suggest means by which the strength can be significantly increased, the failure of concrete in tension was considered a worthwhile subject for investigation. In order to reduce somewhat the complexity of this investigation, it was decided to concentrate initially on hardened cement paste, with the intention that once this simpler system was more fully understood, then the work could be extended to the effect of aggregates.

In general the measured tensile strength of materials is one or two orders of magnitude below the value theoretically estimated. This discrepancy between the theoretical and observed strength is normally attributed to the stress concentrating effect of inherent flaws. Materials are not physically perfect; structural imperfections arise either as a result of the nature of the material as in crystal defects, or as a result of mechanical handling or fabrication procedures, for example cracks and scratches. These imperfections locally affect the stresss distribution in the body and result in the low observed strength. Thus the measured tensile strength is determined both by the material properties and by the imperfections present in the specimens tested.

The size of the largest flaws inherent in a material may be deduced from the normally measured strength provided there exists a theory for the material which relates strength to flaw size. Possible theories may be tested by loading to failure specimens containing artificially introduced flaws of known size and if found applicable may hopefully be assumed to apply also to the smaller inherent flaws. The artificially introduced flaws must be larger than the inherent flaws if they are to affect the strength.

Several papers in the literature [1–3] claim that linear-elastic fracture mechanics can relate strength to flaw size for hardened cement paste. However, there has been a certain amount of controversy surrounding this claim and so it was felt that a careful evaluation of the applicability was in order before proceeding to estimate inherent flaw sizes.

The theory of fracture mechanics has been extensively treated elsewhere [4] and so only a brief discussion is given here. Linear-elastic fracture mechanics is a study of the stress and displacement fields near the tip of a sharp crack in an ideal homogeneous elastic solid. In a homogenous linear-elastic body, the stress and displacement field in a region round the crack tip can be expressed in terms of one parameter, the stress intensity factor K, which is a function of load and specimen configuration only. As the load on the body increases, K increases until it reaches a critical value K_C at which rapid unstable growth of the crack occurs. This critical stress intensity at which the crack propagates is a material property called fracture toughness.

For a sharp crack length $2a$ under a tensile stress σ in an infinitely large body,

$$K = \sigma\sqrt{(\pi a)}$$

i.e. failure occurs when σ increases to a value at which

$$K_C = \sigma\sqrt{(\pi a)}.$$

Since K_C and π are constants the theory predicts that in this case the strength is inversely proportional to $\sqrt{a}$. In practice the length of the crack may be comparable with the specimen size, and this significantly affects the stress field round the crack so that, although failure still occurs at the same critical value of K, there is no longer a simple unique relationship between strength and crack size. It is, however, still possible to deduce the size of the strength controlling flaws from K_C and the normally measured strength.

Real materials differ from the ideal in that they are inhomogeneous and also behave in a non linear-elastic manner at the high stresses induced close to the crack tip. The validity of the above failure criterion to real materials depends upon the extent to which the stress around the crack tip is disturbed by non-elastic behaviour or inhomogeneity. If the region of stress disturbance is small relative to the specimen and flaw size, then the stresses outside this small region will be substantially in accordance with the ideal stress distribution. Under these conditions linear elastic fracture mechanics will apply, i.e. its concepts are most applicable to brittle homogeneous materials in which the inelastic region and the scale of inhomogeneity are small compared to the specimen and flaw dimensions.

In order to test its applicability to hardened cement paste, suitable specimens were tested and the stress intensity factor at failure calculated, by assuming it to apply. If it is indeed applicable, then the same critical value should be obtained at failure over a wide range of specimen sizes and configurations, and this critical value is the fracture toughness.

2. Experimental

A single large batch of ordinary Portland cement stored in sealed tins was used in the preparation of specimens. A weighed quantity of this cement was thoroughly mixed with distilled water in a plastic bag, poured into a mould and then consolidated on a vibrating table for 1 min. The mould and contents were cured in a 100% r.h. fog room at 20° C, the mould being stripped after 1 day.

In the first of a series of experiments, notched beams of hardened cement paste were tested to failure in three-point bending (see Fig. 1). Specimens nominally 9 cm long by 1.4 cm deep by 2.5 cm wide were cut from blocks of hardened cement paste (0.3 water/cement ratio) which had been cured for at least 1 month. Slits were then introduced with a thin diamond impregnated saw (thickness 0.015 cm). The plane of the slit relative to the direction which had been vertical during moulding was varied, in case segregation had led to anisotropy in the material. However, no anisotropy was detected, although it was found that there existed on the surface which had been the top during moulding, a layer a few millimetres deep which was weaker than the remainder. This layer, which was probably caused by slight bleeding, was normally cut off and discarded.

Beams of cement paste show drastic strength reductions (~ 50%) [5], if allowed to dry out for periods as short as 1 min. Therefore, great care was taken to keep the specimens wet at all times. The cutting and slitting were carried out under a copious flow of water, and the specimens were completely immersed in water during the actual testing. Since cement paste has a small strain to failure (~ 0.05%), the bottom two rollers of the

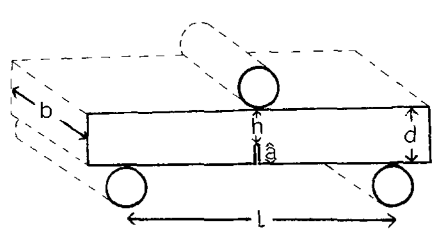

Figure 1 Flexural specimen.

testing rig were free to rotate slightly in a vertical plane through their axes, compensating for any slight lack of parallelism in the specimens. These rollers whose spacing was 7 cm were also free to roll, since restraining them increased the failure load slightly (~ 10%). The specimens were tested to failure in an Instron testing machine with a cross-head speed of 0.05 cm min^{-1}.

Subsequently, the following variables were individually varied: (1) water cement ratio, (2) specimen age, (3) rate of application of load, (4) slit width, (5) specimen size.

A number of specimens were broken in direct tension. In tensile testing, it is difficult to avoid the bending stresses which occur when the line of loading is not concentric with the centroid of cross-sectional area, e.g. an eccentricity of loading of only $\frac{1}{2}$ mm in a specimen 1 cm wide produces a maximum stress on one side 30% higher than expected [6].

A magnesium specimen equipped with electrical strain gauges was used to investigate several methods of tensile testing. Magnesium was used because its elastic modulus is similar to that of cement paste. As a result of this investigation, it was decided to use pin loaded specimens (Fig. 2). Here the two sides parallel to the pins were always equally stressed provided they were accurately symmetrical about the pin holes. This symmetry could be achieved by grinding the specimens on a specially constructed rig. Since normally the stresses on the two sides perpendicular to the pins were unequal, waterproof strain gauges were mechanically clamped to these sides. Then by adjusting the specimen position in the testing rig while monitoring the gauges it was possible to equalize the strains (and hence stresses) on these two sides and thus achieve pure tensile loading.

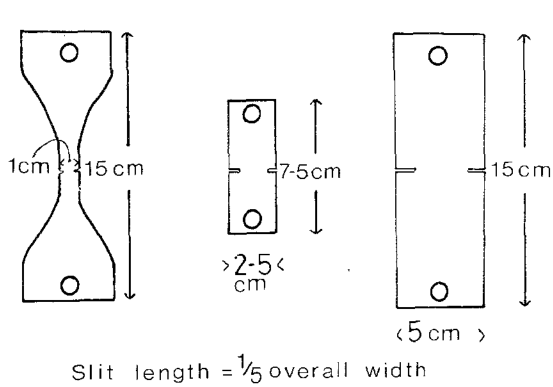

Figure 2 Tensile specimens.

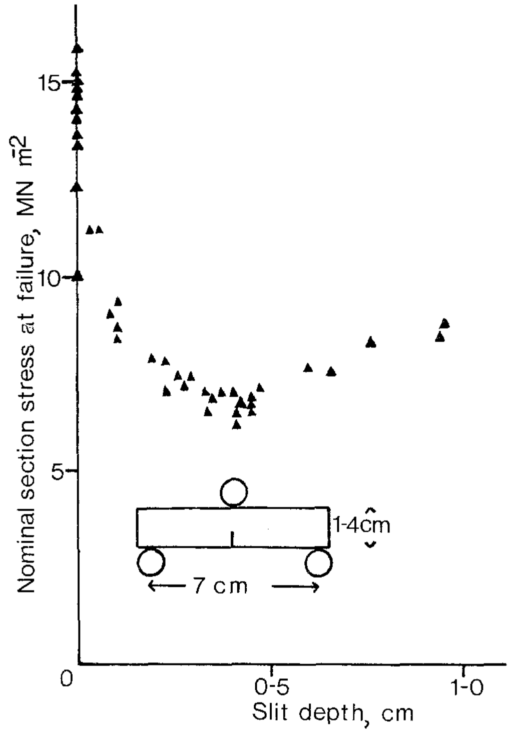

Figure 3 Nominal section stress versus slit depth.

Strips of perforated steel were used to reinforce the loading holes and the loading rate was adjusted so that failure occurred in the same time as with the flexural specimens. Once again the specimens were tested completely immersed in water.

3. Results

Fig. 3 shows the results obtained from the initial experiments in which beams having fixed overall depth but varying slit depth, were broken in

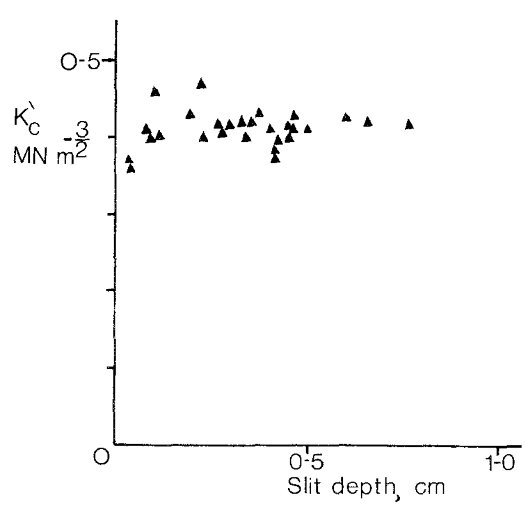

Figure 4 K'_C versus slit depth.

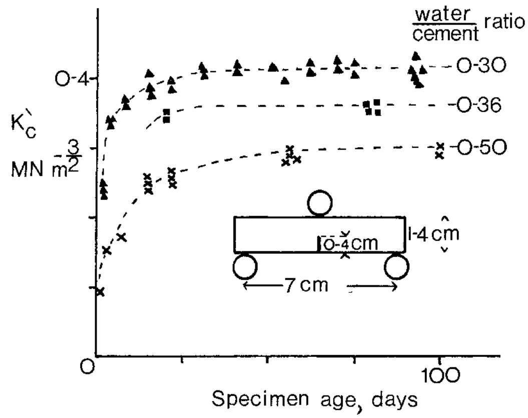

Figure 5 K'_C versus specimen age and water : cement ratio.

flexure. The nominal section stress σ_n is the maximum stress which would be present if there were no stress concentration due to the slit, and in flexure was calculated from the normal expression for modulus of rupture:

$$\sigma_n = \frac{3Pl}{2bh^2}$$

where P is the failure load and b, l and h are specimen dimensions (see Fig. 1). It is apparent from Fig. 3 that there is a stress concentration due to the slit, i.e. the material is "notch sensitive".

For a beam in three-point bending, the stress intensity factor K is given by [7].

$$K = \gamma \frac{3Pla^{\frac{1}{2}}}{2bd^2},$$

where $\gamma = 1.93 - 3.07\ a/d + 14.53\ (a/d)^2 - 25.11\ (a/d)^3 + 25.8\ (a/d)^4$ and Fig. 4 shows the previous results replotted as a graph of calculated

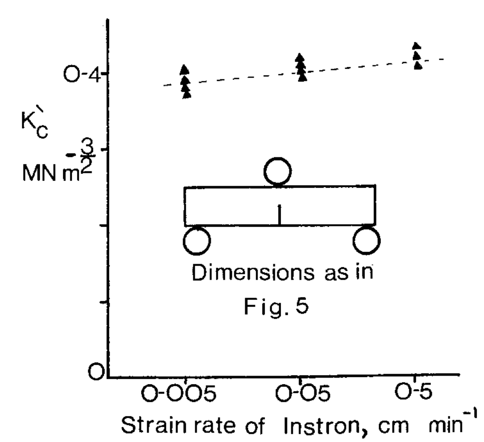

Figure 6 K'_C versus rate of loading.

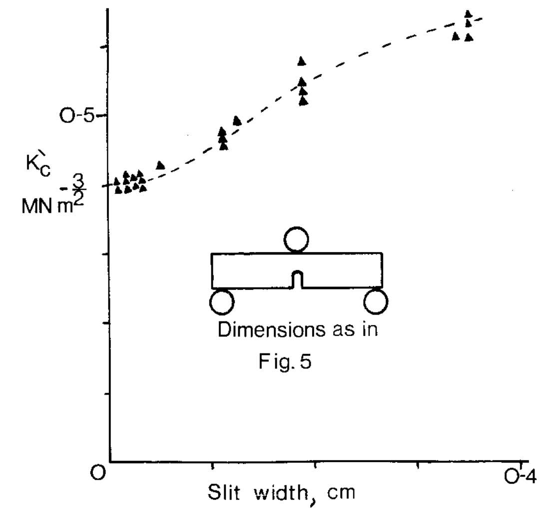

Figure 7 K'_C versus slit width.

stress intensity factor at failure K'_C against slit depth. From the graph this factor would appear to be a material constant.

The variation of the apparent (i.e. calculated) stress intensity factor at failure (K'_C) with water cement ratio, specimen age and rate of application of load is shown in Figs. 5 and 6, and was investigated to test the sensitivity of the results to the inevitable small experimental variations in these parameters. It was concluded that no significant error would be introduced by the experimental variation in these parameters. Fig. 7 shows how K'_C was affected by the width of the slit cut in the specimen. For wide slits, the failure load (and hence K'_C) decreased as the slit width decreased.

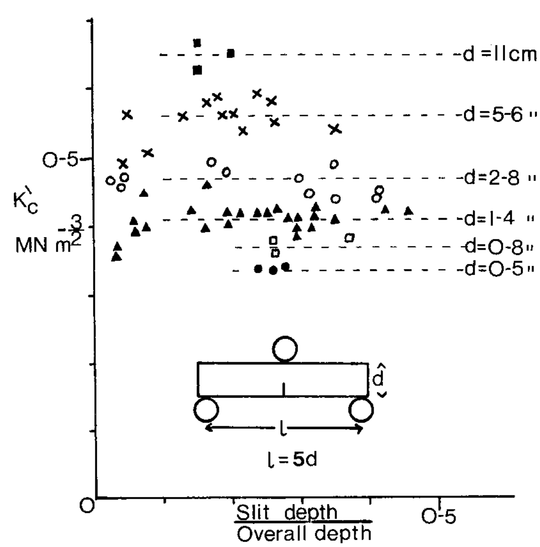

Figure 8 K'_C versus slit depth for varying specimen sizes.

However, for slit widths below about 0.05 cm no significant change in failure load with slit width was detected, and it was concluded that the slits cut by the thin diamond impregnated saw which were about 0.03 cm wide were sufficiently narrow to approximate to a sharp crack. All the following results relate to slits cut with this saw.

The initial experiments on beams of fixed overall depth had yielded a constant value of K'_C over a wide range of slit depths. However, when the overall beam depth was changed, K'_C also changed (see Fig. 8). Alterations in the roller spacing or the specimen width alone produced no change in K'_C.

In the direct tensile tests on the three sizes of specimen shown in Fig. 2, K'_C once again varied with specimen size, the values obtained (and their standard deviation) being respectively: 0.39 ($\pm$ 0.02), 0.44 ($\pm$ .02) 0.49 ($\pm$ .01) MN m$^{-3/2}$. Since K'_C calculated from the experimental results was not a constant, it must be deduced that simple linear elastic fracture mechanics is not applicable to hardened cement paste samples of the size used in the present study, i.e. the zone of stress perturbation round the slit tip is not small compared with the specimen and slit sizes. Although this means that we cannot use this analysis to estimate the size of the strength controlling flaws, it is still of interest to estimate the size of specimen required for the analysis to be applicable.

In the flexural tests, with notches between 1/6 and 1/2 of the overall depth, a relatively constant value of K'_C was obtained for beams of fixed overall depth (Fig. 8). If we plot this value as a function of overall depth (Fig. 9), we see that as the specimen size tends to infinity, so K'_C appears to tend towards a limiting value. Thus tests on large specimens with large slits should yield a constant value of K'_C (N.B. the slit length as well as the overall depth must be large). However, it is not possible to define any definite minimum size since the size at which it is no longer possible to detect an increase in this factor will be determined by the scatter in the experimental results and so the more carefully the experiments are performed, the larger the required minimum size will be. The largest beams used in the present study (60 cm long by 11 cm deep) were still too small for the experimental scatter to mask the change in the calculated stress intensity factor at failure with size.

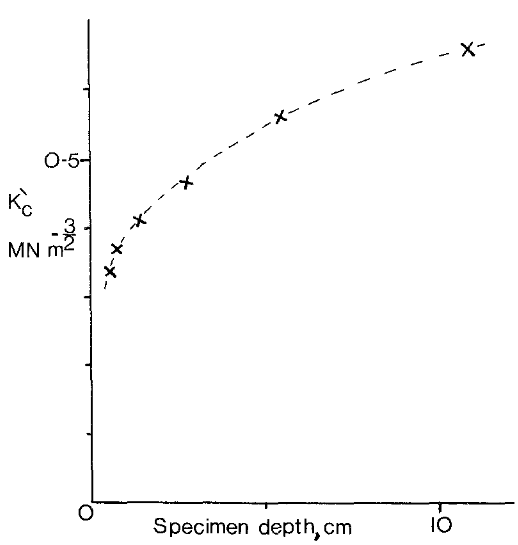

Figure 9 K'_C versus specimen depth.

A small number of tests on mortars showed that the addition of sand made the variation with specimen size even more pronounced, increasing as the size of the sand increased and so it would seem that to obtain constant values of the stress intensity factor at failure for concretes would require ridiculously large specimens. Thus for hardened cement paste and concrete, the size of specimen convenient for laboratory use is too small for linear-elastic fracture mechanics to be applicable, i.e. the zone of stress disturbance due to inhomogeneity or non-linear-elastic behaviour in these specimens is not small compared with the specimen dimensions.

So far the results have been presented in terms of the stress intensity factor K, a factor which tends to be rather confusing for those unfamiliar with it. This form of presentation was necessary because most of the slits were of such a length that they could not be considered small in comparison with the overall depth. In such cases, the proximity of a free edge substantially alters the stress field around the slit from that which would occur in an infinitely large body. It was considered useful to deduce the relationship between strength and slit length which would be obtained for slits in an infinitely large body. In theory, slits of constant size can be introduced into larger and larger specimens, and their effect on the strength examined. Then from the trend of the results it should be possible to deduce the effect that a slit of this size would have in an infinitely large body.

It is necessary to consider first how to define the strength of a body containing a flaw. In a simple tensile test the load at which a specimen

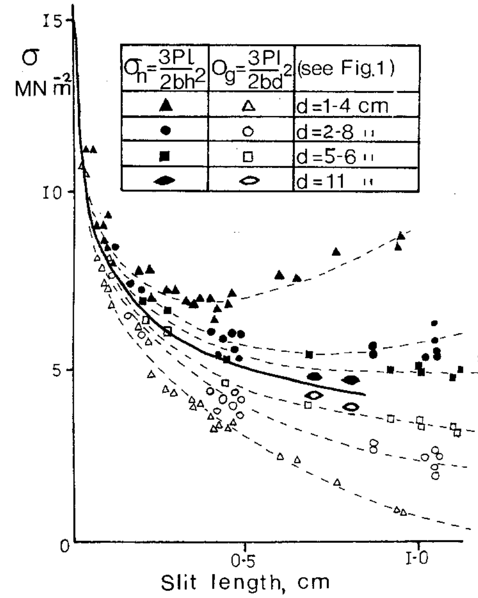

Figure 10 "Strength" versus slit length.

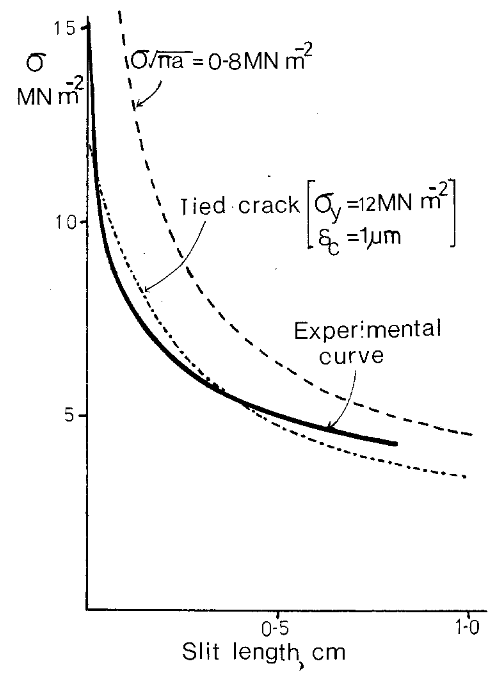

Figure 11 Comparison of fracture mechanics and tied crack predictions with experimental curve.

fails is measured and the strength is the applied stress at failure, i.e. the load divided by the cross-sectional area. However, the relevant cross-sectional area may be either the total area which gives the gross section stress σ_g or alternatively the flaw area may be subtracted from the total area, giving the net section stress σ_n. When the flaw size is insignificant compared to the specimen dimensions, this problem disappears since $\sigma_n = \sigma_g$ and it is possible to refer simply to the applied stress σ.

For the various sizes of flexural specimens tested, σ_n and σ_g at failure were plotted against slit length (Fig. 10). As the specimen depth increases relative to the slit size, σ_n and σ_g tend towards the same value, approaching it from different directions and it is possible to deduce the relationship between σ and slit length, which would be obtained for slits in an infinitely large body. This deduced relationship is shown by the solid line in Fig. 10.

As discussed previously K'_C appears to tend towards a constant limiting value for large specimens with large slits, and from Fig. 9 we may estimate this value to be about 0.8 MN m$^{-3/2}$. Fig. 11 compares the empirical relationship deduced in Fig. 10 with that predicted by fracture mechanics ($\sigma\sqrt{(\pi a)} = K_C = 0.8$ MN m$^{-3/2}$) and shows that for small slits there is a wide discrepancy between the two curves which reduces as the slit length increases. We may once again conclude that, although linear elastic fracture mechanics may be a good approximation for large slits, for the size of slits used in the present study it is inaccurate, i.e. the zone of stress perturbation around the slit tip is not small compared with the slit length.

4. Discussion

The empirical relationship between strength and slit size (deduced for the case where the slit length is small compared with the specimen dimensions) is fitted within the limits of experimental uncertainty by an expression:

$$\sigma_c = \sigma(1 + A\sqrt{a}) . \qquad (1)$$

The solid line in Fig. 10 and 11 is of this form with $\sigma_c = 15$ MN m^{-2} and $A = 28$ m$^{-1/2}$.

A similar expression may be derived by considering the classic case of a flaw with tip radius r in a large linear elastic body (see Fig. 12). The maximum stress occurs at the flaw tip and is given by [8]

$$\sigma_{max} = \sigma[1 + 2\sqrt{(a/r)}] . \qquad (2)$$

If we assume that failure occurs when σ_{max} increases to a critical value σ_c, the ultimate cohesive strength of the material, then we have a failure criterion:

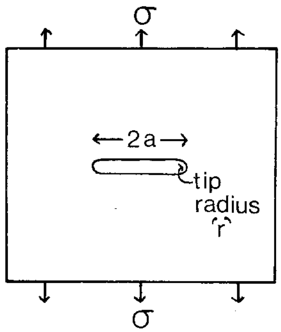

Figure 12 Flaw in an infinite body.

$$\sigma_c = \sigma[1 + 2\sqrt{(a/r)}] \quad (3)$$

which is identical to Equation 1 with $A = 2/\sqrt{r}$, i.e. $r = 0.5$ cm. This suggests that we may characterize the effect of slits in hardened cement paste by saying that they have the same "strength reducing effect" as would be produced in a linear elastic body by flaws of similar length but with tip radius 0.5 cm. However, this analogy, though a useful way of describing the sensitivity of the material to sharp flaws, is of little practical use in understanding the fracture process. The value of 0.5 cm for the effective radius of curvature probably has no significance as a length in the specimen. The fact that it is considerably larger than the actual radius of curvature (0.015 cm) simply suggests that slits in hardened cement paste do not concentrate stress nearly as much as would the equivalent slits in a homogeneous linear elastic body.

Equation 1, the empirical relationship between strength and slit length predicts a value of 15 MN m^{-2} for the ultimate cohesive strength in the absence of flaws. The greatest strength obtained in direct tensile tests on unnotched specimens was 12.5 MN m^{-2} which is consistent with an ultimate strength of 15 MN m^{-2}. In the flexural tests on unnotched beams, although most specimens failed at a modulus of rupture below 15 MN m^{-2}, a few showed greater strengths up to 19 MN m^{-2}. This is, however, not inconsistent with an ultimate tensile strength of 15 MN m^{-2} since the modulus of rupture is calculated from the applied bending moment and gives the maximum tensile stress which would occur if the material behaved in a perfectly linear-elastic manner. A slight deviation from linearity at high stress levels would be sufficient to produce a modulus of rupture of 19 MN m^{-2} without the actual stress in the body exceeding 15 MN m^{-2}, and although no non-linearity was detected in direct tensile tests up to the highest strength achieved (12.5 MN m^{-2}) it does not seem inconceivable that some non-linearity may occur at higher stresses.

The largest slits introduced in the present study appear then to have only reduced the strength of the specimens by a factor of about four (from 15 to 4 MN m^{-2}), in contrast to materials like glass, in which flaws only a fraction of a millimetre long can reduce the strength by a factor of 100, i.e. hardened cement paste is relatively notch insensitive.

Since the deduced ultimate strength and the observed unnotched strength are similar, it would appear that the inherent flaws do not lead to a significant reduction in strength, which suggests that there is no chance of producing large increases in strength by reducing the size of the flaws present, as has been done with other materials.

Various models have been considered in an attempt to explain the mechanical testing results in terms of the probable microstructure. The most promising, postulates that the initial crack growth from the slit is tied, i.e. there exists a residual attractive force between the two faces of the newly formed crack. This would be produced if particles bridged across the two new surfaces, for example failure may occur by slip of calcium silicate hydrate platelets past each other as suggested diagramatically in Fig. 13. The credibility of such a failure mode is supported by Fig. 14, a scanning electron micrograph of a hardened cement paste sample (water: cement ratio 3:1) which shows a

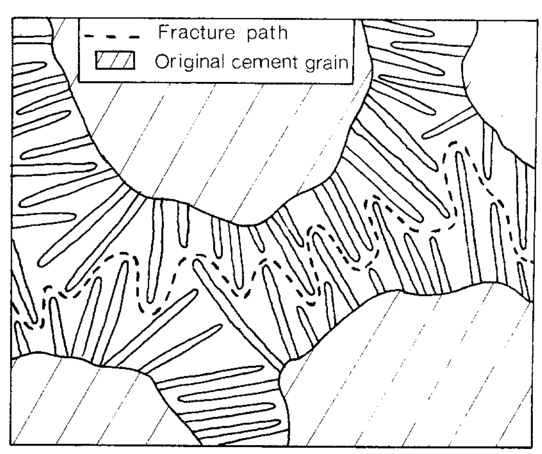

Figure 13 Proposed failure mechanism in which platelets slide past one another.

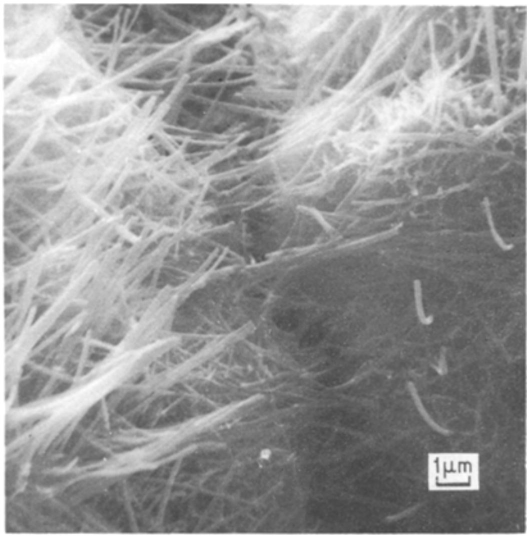

Figure 14 Scanning electron micrograph of hardened cement paste (3:1 water:cement ratio, hydrated 1 month).

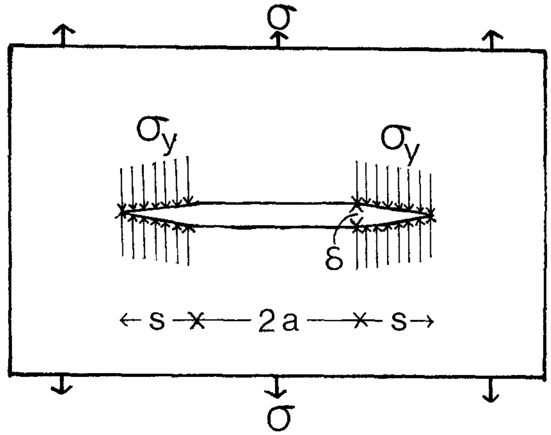

Figure 15 Dugdale model.

mass of interlocking fibres growing out from the original cement grains. While it must be admitted that such fibres grow best in mixes of high water: cement ratio containing unrestrained areas for growth and are not normally distinguishable in the low water: cement ratio specimens tested in the present investigations, it seems plausible to suggest that these specimens contain similar interlocking particles which are difficult to distinguish because of their close packing.

If this tied crack model is correct then, as the stress on the body increases, the length of the tied crack increases until a position of unstable equilibrium is reached at which the body suddenly fractures. Calculation of the expected fracture loads is difficult, since the attractive force between the newly formed surfaces will vary with their separation in a way that is difficult to predict. However, a simple analysis enables a rough estimate of the forces and distances involved to be made. This analysis is based on that of Dugdale (see [4] p. 67) who considered the case of a crack in a large linear elastic body. The ends of the crack are acted on by a constant force σ_y which tends to close up the crack, see Fig. 15. For our purposes, the slit is considered to have length $2a$, i.e. correspond to that portion over which no attractive force acts, and the tied crack is considered to correspond to that length s at the end of the slit over which the attractive force σ_y acts. It can be shown ([4] p. 67) that the crack opening displacement δ at the junction of the tied crack and the slit is given by

$$\delta = \frac{8\sigma_y a}{\pi E} \ln \sec \left(\frac{\pi\sigma}{2\sigma_y}\right).$$

Assume that the attractive force between the crack surfaces remains constant at σ_y until their separation increases to a critical value δ_c at which it drops to zero, then unstable failure will occur when the crack opening displacement δ increases to δ_c i.e. our fracture criterion is:

$$\delta = \delta_c = \frac{8\sigma_y a}{\pi E} \ln \sec \left(\frac{\pi\sigma}{2\sigma_y}\right).$$

Putting $\sigma_y = 12\,\mathrm{MN\,m^{-2}}$ and $\delta_c = 1\,\mu\mathrm{m}$ produces the relationship between strength and flaw size shown in Fig. 11, which although by no means a perfect fit to the experimental curve is close enough to suggest that it should be possible to fit the experimental curve exactly by means of a more sophisticated analysis in which the residual attractive force σ_y varies with the crack separation δ. However, the complexity of the mathematics makes such an analysis difficult and it was not considered to be justified while there existed no direct method of measuring the relationship between δ and σ_y.

The tied crack model postulates a plausible failure mechanism, and the Dugdale analysis provides an estimate of the forces and distances involved. It now remains to consider whether these forces and distances are realistic.

The value for σ_y ($12\,\mathrm{MN\,m^{-2}}$) is slightly less than the deduced ultimate strength ($15\,\mathrm{MN\,m^{-2}}$)

which is reasonable. The critical separation (1 μm) is around the normally estimated value for the length of the calcium silicate hydrate platelets [9] and agrees well with microscopical observations on stable crack growth in the material [10]. Therefore, such a model, in the absence of any evidence to discredit it, provides a useful working hypothesis to suggest further lines of enquiry. In particular, if failure in fact occurs by the pulling apart of interlocking platelets, it might well be possible to increase the strength of hardened cement paste by increasing the length of the platelets present, since long platelets would presumably be harder to pull apart than short ones.

5. Conclusions

(1) Tests on notched specimens of hardened cement paste failed to produce a single constant value of stress intensity factor at failure, and it appears that linear elastic fracture mechanics and the concept of fracture toughness are not readily applicable to laboratory sized specimens of this material.

(2) Extrapolation of the experimental results suggests that hardened cement paste is a relatively notch insensitive material whose strength is not greatly reduced by the flaws normally present.

(3) A tied crack model is consistent with the microstructure and can account semi-quantitatively for the experimental results.

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