STRENGTH, FRACTURE AND DEFORMATION BEHAVIOUR OF PORTLAND CEMENT PASTE

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ABSTRACT

The effect of strain rate on the strength and fracture toughness of cement paste has been studied at room temperature. At strain rates up to 10^{-3}/s cement paste exhibits microductility, with rising strength and an activation volume of $1.5 - 2.0 \times 10^4$ Å^3. At strain rates above 10^{-3}/s cement paste behaves in a brittle manner with constant strength and a marked fall in fracture toughness. This transition to brittleness is interpreted in terms of micro-shearing and slow crack growth. The critical strain rate corresponds to the fastest relaxation process found in a stress-relaxation test.

KEYWORDS

Cement paste; strain rate; strength; fracture toughness; stress relaxation; slow crack growth.

INTRODUCTION

The understanding of the strength and fracture behaviour of Portland cement paste is fundamental to the production of improved cementitious materials. In recent years the applicability of fracture mechanics to cement paste has been examined. A detailed study of the effect of external variables on the fracture toughness of cement paste has been carried out by Higgins and Bailey (1976). An interesting conclusion drawn by these authors is that the stress-concentrating effect of notches in cement paste is small in comparison to metals, although Ziegeldorf, Müller and Hilsdorf (1981) have shown that it is large compared with mortar and concrete.

Many fracture mechanics studies on hardened cement paste may be criticised on the grounds that the notches were not sharp enough, or that corrections were not made for slow crack growth. Although a sharp crack may be produced by slow controlled loading (Hillemeier and Hilsdorf, 1977), the problem of accurate measurement of crack length remains. This difficulty is further compounded by the possibility of micro-cracking ahead of the main crack making it difficult to define the crack tip unambiguously. Hillemeier and Hilsdorf (1977) determined their fracture toughness values using compact tension specimens with sharpened cracks that were produced by slow stable crack growth. However, the advantage of using sharply cracked specimens may have been lost by using the compliance calibration curves determined from ordinarily notched specimens. They obtained a fracture toughness of 0.31 MPa $m^{\frac{1}{2}}$ (Table 1) and this is close to the value obtained by other workers using the same water/cement ratio with sawn or cast notches.

In the present work we have investigated the effect of strain rate on the strength and fracture toughness of cement paste. An effort has been made to relate these results, in conjunction with relaxation and SEM studies, to the sub-microstructural features of hardened cement paste. Wittmann (1974) has shown that the creep mechanisms in cement paste are thermally activated and that the Arrhenius rate equation applies. Our work extends this treatment to much higher strain rates.

EXPERIMENTAL

Specimens, in two different shapes (12 x 110 x 220 mm plates and 40 x 80 x 320 mm beams), were prepared from ordinary Portland cement with a water/cement ratio of 0.5. After vibration, the mould and its contents were kept in a fog room for one day before stripping from the mould. The specimens were cured wet in lime-saturated water until the time of testing.

(i) Plate specimens used for double torsion (DT) testing were grooved along their length with a 1.0 mm thick diamond saw to a depth of ∿1/3 of the thickness of the specimen. A notch was introduced at one end of the groove with the same saw except for two specimens which were notched with a 0.3 mm saw. All DT specimens were 75-78 days old at the time of testing and were kept wet during grooving, notching or testing. The stress intensity factor K was determined using the equation of Williams and Evans (1973).

$$K = Pw_m\left[\frac{3(1+\nu)}{Wt^3 t_n}\right]^{\frac{1}{2}} \tag{1}$$

where P = total applied load, w_m = length of each moment arm, W = plate width, t = plate thickness, t_n = plate thickness in the plane of groove and ν = Poisson's ratio.

(ii) Bend specimens cut from plates, 75-100 days old, were 12 x 25 x 100 mm (with a test span of 64 mm) and those cut from beams, 35-40 days old, were, unless stated otherwise in the text, nominally 20 x 40 x 160 mm (with a test span of 140 mm). Nearly half the number of beams from the latter group were broken without a notch and the flexural strength, σ_f, of these specimens was determined from the expression

$$\sigma_f = 3/2\ Pl/bd^2 \tag{2}$$

where P is the load, l the beam span, b the thickness, and d the depth of the beam. Notches to act as crack-starters were cut into specimens used for single edge-notched bend (SENB) testing with diamond saws of 0.3 mm and 1.0 mm thickness. Specimens were kept wet during cutting, slitting and testing, and the slits were introduced one day before testing. The stress intensity factor K for SENB tests is given by Brown and Srawley (1969) as

$$K = Y\frac{3Pl\ a^{\frac{1}{2}}}{2Bd^2} \tag{3}$$

where Y is a geometrical factor and a is the notch length.

(iii) Relaxation tests were conducted in three point bending on two, 40 day old, unnotched beams (40 x 40 x 160 mm) cut from a large beam. Each specimen was loaded to a predetermined load P_o and, after a desired period of load relaxation, a repeat relaxation test was carried out by reloading the specimen to P_o and allowing it to relax again. For thermally activated processes the relaxation test results can be represented (Klug and Wittmann, 1974) by

$$\Delta\sigma \equiv \sigma_o - \sigma = \frac{kT}{V}\ln(1 + At) \tag{4}$$

where σ_o and σ are stresses at times zero and 't' respectively, k is the Boltzman constant, T is the absolute temperature, A is a constant, and V is the activation volume. For large times, equation 4 reduces to a simple form

$$\Delta\sigma = \frac{kT}{V}(\ln A + \ln t) \tag{5}$$

which can be used to determine the activation volume. In the determination of activation volume from equation 5 and equation 6 we have used the maximum tensile flexural stresses, σ, rather than the more usual shear stress τ.

RESULTS

In agreement with Nadeau, Mindess and Hay (1974) the values of K_{IC} obtained from DT and from SENB tests in this work were found to be very similar at cross-head speeds up to 10^{-5} m s^{-1}.

TABLE 1 Fracture Toughness of Hardened Cement Paste

Reference	Method	w/c	Age (days)	K_{IC} MPa $m^{\frac{1}{2}}$
This work	3-BD	0.5	75-100	0.34
This work	DT	0.5	75-100	0.30-0.36
1	3-BD	0.5	78-99	0.32
1	DT	0.5	78-99	0.29-0.34
2	4-BD	0.47	14-84	0.33
3	CT	0.4	>50	0.31
4	3-BD	0.5	75-100	0.30
4	3-BD	0.3	75-100	0.40
5	4-BD	∿0.3	41	0.49-0.66

1 = Nadeau, Mindess and Hay; 2 = Brown and Pomeroy; 3 = Hillemeier and Hilsdorf; 4 = Higgins and Bailey; 5 = Mindess, Lawrence and Kesler.

The high values for DT were calculated from the maximum load and the low ones from the load at instability. The results from the DT tests were independent of the length and thickness of the notch, whereas for SENB tests K_{IC} increased with notch thickness from 0.3 mm to 1.0 mm but was independent of notch length in the range a/d = 0.2-0.5. All SENB results reported below are for a/d = 0.4 and notch thickness 0.3 mm. The latter value lies well within the range of thickness in which Higgins and Bailey (1976) found that the stress intensity factor was independent of notch thickness. The high values of K_{IC} of Mindess, Lawrence and Kesler (1977) (Table 1), appear to be due mainly to the large thickness (3 mm) of their notch, since Higgins and Bailey (1976) also found $K_{IC} \cong 0.6$ at w/c = 0.3 with a notch 3 mm thick. The width of the damage zone ahead of the crack tip in cement paste appears to be ~0.4 mm and any notch thicker than this cannot be treated as a sharp crack. If we treat the notch as an elliptical crack, with 3 mm as the radius at the apex, then for K_{IC} values of 0.49-0.66 MPa $m^{\frac{1}{2}}$ (Mindess, Lawrence and Kesler, 1977) and a crack length of ~40-50 mm, we obtain fracture strengths for unnotched beams equal to 11-15 MPa. These are in good agreement with the experimental strength of beams with w/c = 0.3, measured by Higgins and Bailey (1976). The Table shows that, for sharp notches, there is an increase in K_{IC} at lower w/c ratios but the effect is small.

The effect of a wide range of cross-head speeds, $\dot{X}$, and thus of maximum flexural strain rates, $\dot{\varepsilon}_{max}$, on the toughness of 75-100 day old paste measured using the SENB test is shown in Fig. 1. The maximum flexural strain rate was determined from the equation of Terwilliger, Bowen and Gordon (1970)

$$\dot{\varepsilon}_{max} = (6d/l^2)\dot{X}$$

where l is the beam span and d the beam depth. The K_{IC} values increase to a maximum at a cross-head speed of about 5 x 10^{-5} m s^{-1} and then sharply decrease. A similar strain rate dependence for K_{IC} has been reported for alumina at high temperatures by Kromp and Pabst (1980). An increase in toughness with increasing cross-head speed in the lower range of speeds was reported by Higgins and Bailey (1976).

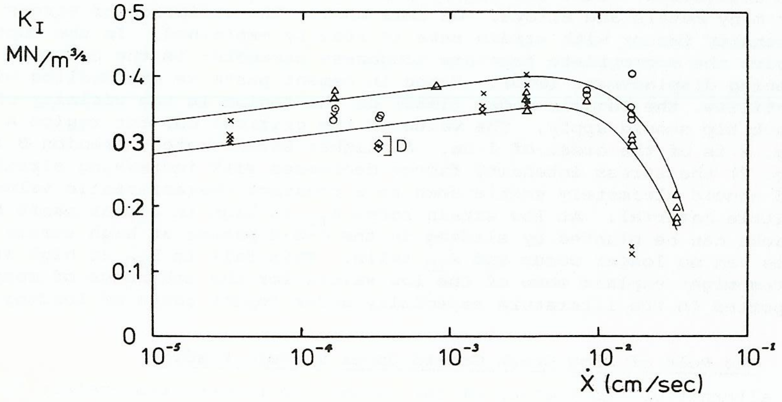

FIG. 1. Variation of K_{IC} with cross head speed $\dot{X}$
x = 75-85 days old, O = 90 days old, Δ = 100 days old,
◇ = 100 days old and dried for 5-10 min.

To test the effect of drying on fracture toughness, two SENB specimens were taken out of water and allowed to dry at room temperature for 5 and 10 minutes respectively. The specimen surfaces looked almost completely dry but the stress intensity factor of these specimens was reduced only by 15% (Fig. 1). Although this confirms the view that specimens should be kept wet during testing, the effect of short times of drying at least on 75-100 day old pastes appears to have been exaggerated by Higgins and Bailey (1976).

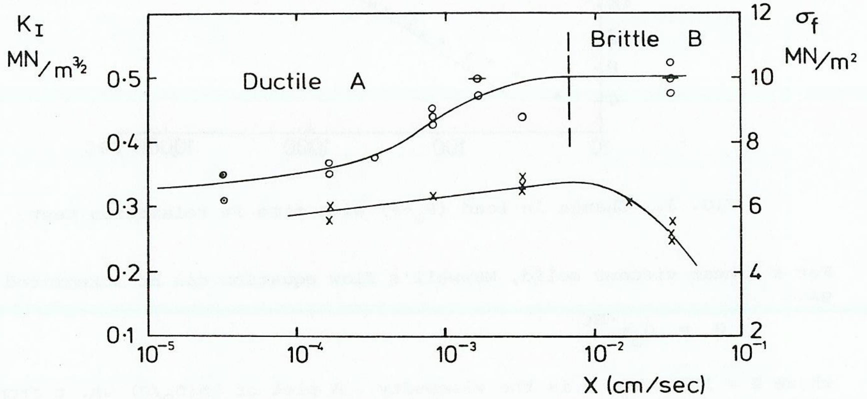

FIG. 2. Variation of K_{IC} and σ_f with cross head speed $\dot{X}$ for specimens aged 35-40 days.

Figure 2 shows the SENB fracture toughness and the flexural strength of unnotched specimens for 35-40 day old beams of 20 x 40 x 160 mm size. The effect of cross-head speed on the fracture toughness is similar to that shown in Fig. 1, while the flexural strength increases with cross-head speed markedly at about 10^{-5} m s^{-1} and then levels off. An increase in the size of unnotched beams to 40 x 40 x 160 mm did not affect the flexural strength significantly. The fracture toughness of cement paste increases only slightly with age after 35-40 days of hydration; a similar effect was observed by Higgins and Bailey (1976) for pastes with water/cement ratio = 0.5.

A typical result of the change in load obtained from the relaxation tests is shown in Fig. 3. Equation 5 is obeyed at long times and the activation volume is ~2 x 10^4 Å^3. The activation volume can also be determined from the strain rate dependence of the flexural strength (Gibbs, 1969), as

$$V = kT\,\partial \ln\dot{\varepsilon}/\partial\sigma_f \qquad (6)$$

At cross-head speeds below the sudden increase near 10^{-5} m s^{-1}, this yields a value of ~1.5 x 10^4 Å^3. Two bend specimens were allowed to dry in air at room temperatures for about 30 days and then broken in bending at different strain rates; these gave an activation volume of 3.7 x 10^4 Å^3, in good agreement with the values found by Klug and Wittmann (1974).

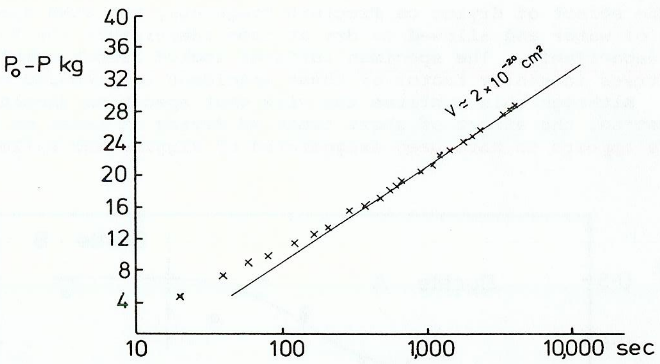

FIG. 3. Change in load (P_o-P) with time in relaxation test.

For a linear viscous solid, Maxwell's flow equation can be integrated to give

$$\sigma = \sigma_o e^{-Bt} \qquad (7)$$

where $B = 1/3\eta$, and η is the viscosity. A plot of $\ln(\sigma_o/\sigma)$ vs. t from the relaxation tests, Fig. 4, fails to give a straight line, suggesting that the flow of cement paste is non-linear. Equation 7 may be applied for cement paste only by replacing the term Bt by $\int_o^t \frac{dt}{3\eta}$. A non-linear behaviour for cement paste is also suggested by the repeat relaxation tests, in which the magnitude of stress relaxation in the second relaxation cycle was smaller than that in the first cycle at corresponding times.

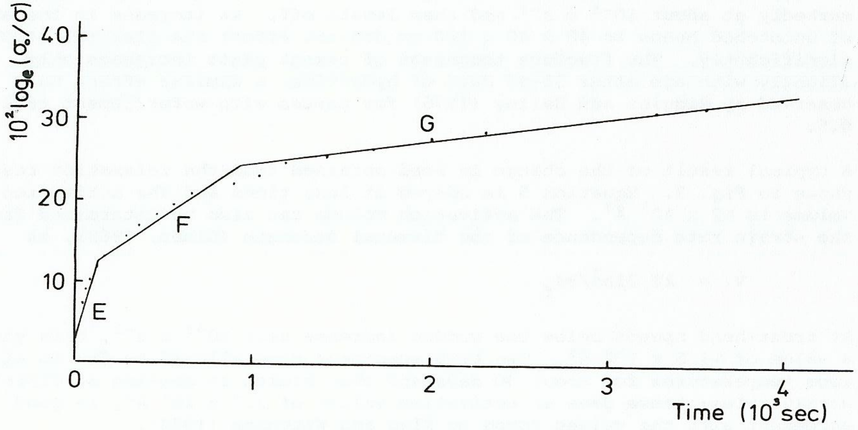

FIG. 4. Variation of load ratio σ_o/σ during relaxation test.

DISCUSSION

1. The Ductile-Brittle Transition

The activation volumes measured at strain rates in the ductile region may be interpreted in terms of a thermally activated shear process, occurring between sheets of calcium silicate hydrate (C-S-H) gel. For displacements of the order of the Ca-Ca spacing found by Taylor (1979), these activation volumes correspond to the shear of sheets of gel of the size formed in the later stages of hydration. The presence of water molecules between the sheets would aid the shear process in wet specimens, while shear would be more difficult in the dry specimens. These activated shear processes were observed both during relaxation tests and during measurements of flexural strength at various strain rates. The possibility of a relaxation process occurring at a given rate of deformation depends upon the relaxation time for that process. If the relaxation time is too large, the process cannot occur fast enough to maintain the externally imposed strain rate. Under these conditions another faster process must come into operation or brittle behaviour will intervene. In equation 7, B is equal to the relaxation frequency (i.e. the inverse of the relaxation time). From Fig. 4 and equation 7, the relaxation frequency for the fastest relaxation process found in cement paste (region E in Fig. 4) is 10^{-3} s^{-1} which corresponds very well with the strain rate above which brittle behaviour is observed in Fig. 2. This suggests that the distinction between the ductile and the brittle regions does have physical significance.

2. The Effect of Strain Rate on Toughness

So far we have considered the relaxation process responsible for the ductile regions to be one of thermally activated shear. The increase in strength with strain rate in region A is similar to that observed for ductile metallic materials, while the constant strength at high strain rates in region B is characteristic of brittle materials. Such a transition from ductile to brittle behaviour with increased strain rate has been reported by Maddin and Masumoto (1972) for a micro-crystalline Pd-Si alloy. Furthermore, the activation volume for cement paste is of the same order of magnitude as for many metals and alloys. On this basis, the variation of stress intensity factor with strain rate is readily explained. In the ductile region the appropriate fracture toughness parameter is the critical crack opening displacement (COD). Since in cement paste we are dealing with micro-ductility, the Dugdale model (1960) of the region in the vicinity of the crack tip should apply. The value of the critical COD for region A of Fig. 2 is of the order of 1 µm. At higher strain rates, (region B in Fig. 2) the stress intensity factor decreases with increasing strain rate and should ultimately settle down to a constant characteristic value for a brittle material. At low strain rates K_{IC} is high in cement paste because cracks can be blunted by sliding in the C-S-H phase; at high strain rates this can no longer occur and K_{IC} falls. This fall in K_{IC} at high strain rates might explain some of the low values for the toughness of concrete reported in the literature especially under impact rates of loading.

3. The Role of Slow Crack Growth Under Dynamic Loading

An alternative explanation of the relaxation process responsible for the micro-ductility is the formation and growth of stable micro-cracks. Evans (1974) has shown how the parameters of sub-critical crack growth can be determined from the strain-rate dependence of the flexural strength for brittle materials. In particular, a plot of log σ vs. log $\dot{\varepsilon}$ should have slope 1/1+n, where n is the slope of the log V - log K_I plot, determining slow crack growth. Plotted in this way, our flexural strengths from Fig. 2 reveal all of the features of slow crack growth at low strain rates, reverting to fully brittle behaviour at strain rates above 10^{-3} $\sec^{-1}$. From our limited results the value of n for cement paste lies between 20 and 40; this extreme range includes the results of Nadeau, Mindess and Hay (1974) and Mindess and Nadeau (1977) of ≅35 and 34.2, obtained directly from their log V - log K measurements for cement pastes. Mindess and Nadeau's figure of 17.7 derived from the strain rate dependence of their flexural tests appears to arise from a rather particular interpretation of their data. A more general interpretation suggests a figure of ∿40, in closer agreement with their direct measurement of n. The relationship between micro-cracking and thermally activated shear in cement paste requires further consideration.

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