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Effect of Humidity on Subcritical Crack Growth in Cement Paste

by J.J. Beaudoin

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RÉSUMÉ

L'effet de l'humidité sur la croissance sous-critique des fissures dans la pâte de ciment a été étudié. Les courbes du rapport intensité des contraintes-vitesse de fissuration des échantillons ont été obtenues au moyen d'une technique faisant appel à la double torsion. Les données ont été utilisées pour déterminer les mécanismes possibles de propagation des fissures. La nature et la position relative de ces courbes dépendaient à la fois de l'humidité et de la porosité. On a comparé ces courbes à celles obtenues pour le verre soumis à divers taux d'humidité afin d'étudier les similitudes et les différences entre les deux matériaux.

EFFECT OF HUMIDITY ON SUBCRITICAL CRACK GROWTH IN CEMENT PASTE

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ABSTRACT

The effect of humidity on subcritical crack growth in cement paste was studied. Log crack velocity - stress intensity factor curves were obtained using a double-torsion technique. The data were used as a basis for assessing possible crack growth mechanisms. The character and relative position of these curves were both humidity and porosity dependent. Comparison was made of these curves with those obtained for glass at various humidities; similarities and differences are discussed.

Introduction

Hydrated portland cement paste is a moisture-sensitive material and many of its physical, mechanical and chemical properties, other than the hydration process itself, are humidity-dependent (1). Fracture mechanics techniques have been useful in studying the effect of environmental factors on fracture toughness and durability of glass and ceramic systems (2). Slow growth of cracks in a brittle material subjected to an applied stress less than the apparent failure stress can result in static fatigue or delayed fracture (3-5). This occurs when the flaw grows to the critical size for that stress. Slow crack growth in brittle materials is referred to as subcritical crack growth. Slow growth of cracks has been studied for numerous glass and ceramic systems and, to a limited extent, for the portland cement paste system (6).

The basic data used to estimate life expectancy or time to failure of brittle materials such as glass, are obtained from the log crack velocity-versus-stress intensity factor ($\log V-K_I$) curve. The objective of this study was to determine the effect of relative humidity on subcritical crack growth in cement paste, as characterized by the $\log V-K_I$ plot.

Experimental

Materials

Portland cement. The composition was as follows: $\text{SiO}_2 = 20.78\%$; $\text{Al}_2\text{O}_3 = 6.20\%$; $\text{Fe}_2\text{O}_3 = 2.23\%$; $\text{CaO} = 64.83\%$; $\text{MgO} = 1.84\%$; $\text{SO}_3 = 3.17\%$; $\text{Na}_2\text{O} = 0.05\%$; $\text{K}_2\text{O} = 0.40\%$. Blaine fineness was $300 \text{ m}^2/\text{kg}$, and calculated Bogue compound composition was $\text{C}_4\text{AF} = 6.7\%$; $\text{C}_3\text{A} = 12.7\%$; $\text{C}_3\text{S} = 51.4\%$; $\text{C}_2\text{S} = 20.3\%$ and $\text{CaSO}_4 = 5.4\%$.

Mixes and specimen preparation. Cement paste mixes were made at the following water/cement ratios: 0.25, 0.30, 0.35, 0.40, 0.45 and 0.50. Double torsion specimens were fabricated according to the following procedure. Mixes were cast in moulds to form $38 \times 76 \times 254 \text{ mm}$ prisms. The prism-shaped samples were removed from the moulds after 24 hours and moist cured for approximately 12 months. After this curing period, the prisms were sawn at 1-mm intervals along their length to produce test specimens $38 \times 76 \times 1 \text{ mm}$ thick. A centrally located guide groove $1.5 \times 0.5 \text{ mm}$ deep was saw cut along the length of each specimen. Starter cracks were notched in each specimen with a thin diamond blade (0.36 mm thick) to produce the final test piece, as illustrated in Figure 1. The specimens were then conditioned in desiccators over saturated salt solutions for up to 90 days at the following relative humidities: 100%, 95%, 66%, 32%, 11% and dry. The dry condition was obtained by heating the specimens under vacuum at 110°C for three hours. This procedure is equivalent to drying samples to equilibrium with the vapor pressure of dry ice at -78°C (D-drying) and corresponds to a relative humidity of approximately 0.003%. Five specimens were prepared for every test.

Porosity Measurement

Porosity of cement paste samples was determined using an Aminco Porosimeter capable of intrusion pressures up to 407 MPa.

Fracture Testing

An environmental chamber was mounted on the cross-head of an Instron testing machine. A rectangular, thermostatically controlled perspex bath was centrally located within the chamber. Temperature of the bath was controlled to $\pm 0.5^\circ\text{C}$. Tests in lime-saturated water maintained at 21°C were conducted in the bath. For non-saturation conditions, the humidity in the chamber was controlled by circulating conditioned air through the chamber. Saturated salt solutions were used to condition the air, which was pumped in a continuous

FIG. 1
Double-torsion specimen configuration

loop from an external source to the chamber. The chamber was also equipped with instrumentation to monitor the humidity. Carbonation in the chamber was minimized through the use of CO_2 absorbers.

Double torsion specimens, conditioned in desiccators at a particular humidity, were transferred to the test chamber environment, which was maintained at the same humidity. The specimens were placed on a small reaction frame similar to one described by Evans (7). The load was applied in the plane of the front two supports. The rear supports facilitated positioning of the specimen. Due to the double-torsion loading arrangement and geometry, the stress intensity at the crack tip is independent of the crack length and depends only on the load and fixed dimensions of the specimen and reaction frame.

A load relaxation technique was used to determine the $\log V-K_I$ curve for cement paste using an initial cross-head speed of $8.35 \times 10^{-5} \text{ mm} \cdot \text{s}^{-1}$ (8). The crack velocity was determined from the relaxation curves using the following relation:

$$V = da/dt = -(P_i/P^2) (a_i + A/B) dP/dt \quad (1)$$

The subscript i refers to the initial crack length, i.e., P_i is the load at the initial crack length a_i . A and B are the intercept and slope of the compliance - crack length curve. Compliance - crack length curves were obtained for each w/c ratio at each humidity. These curves are numerous and are not presented, as the ratio A/B determined from them is usually $\ll a_i$ and can be neglected. Values of the derivative dP/dt are obtained from the load-relaxation curve.

An expression given by Williams and Evans was used to compute K_I (8).

$$K_I = PW_m \left(3(1+\nu)/Wt^3t_n \right) \quad (2)$$

where P is the load, ν is Poisson's ratio and the other terms are dimensions depicted in Figure 1, which are dependent on the geometry of the test piece and loading configuration.

Results and Discussion

The results are presented in the form of $\log$ crack velocity - stress intensity factor ($\log V-K_I$) curves (Figure 2a-f). Each curve (for each water/cement ratio) was obtained from the results of five test specimens. The initial region of all the curves is linear. Some of the curves have a subsequent non-linear region and two curves (both at 32% RH) exhibit a second linear region.

The significance of three distinct regions in the $\log V-K_I$ curves for non-porous soda-lime glass (Figure 3, regions I-III) has been discussed by Wiederhorn et al (2). They concluded the following: in region I crack velocity is reaction rate limited, i.e., it depends on the chemical potential of water in the environment for a given stress intensity factor; in region II, crack velocity is not strongly dependent on stress intensity factor but is controlled by the rate of transport of water from the environment to the crack tip; in region III, crack velocity is independent of the amount of water in the environment. Discussion of the three regions in the curves for cement paste systems follows. Emphasis is given to region I, as not all curves contain regions II and III.

Region I

Significant differences in the way water interacts with glass and with cement paste (9) influence the relative position of the $\log V-K_I$ curves in Figure 2. The relative position of region I of the $\log V-K_I$ curves with

FIG. 2

Crack velocity - stress intensity curves* for cement paste tested at different humidities

respect to K_I is humidity dependent for all the cement paste preparations. Unlike non-porous glass, however, the median value of stress intensity factor (K_I)* in cement paste does not increase continuously as humidity decreases. In Figure 4, K_I for cement paste specimens generally increases to a maximum (at 11 or 32% RH) as humidity decreases. A decrease in K_I is then observed as the humidity approaches 0%.

*As the $\log V-K_I$ curves are steep for cement paste specimens, it is convenient to discuss the relative displacement of the curves along the K_I axis in terms of the median value of K_I for each curve.

FIG. 3
Crack velocity - stress intensity curves
for cement paste and soda-lime silica
glass. Silica glass data from Ref. 2

FIG. 4
Median value of stress intensity factor from region I of each $V-K_I$ curve
versus relative humidity for cement paste. Data for non-porous glass from
Ref. 2 (crack velocity = 10^{-6} m s^{-1})

The dependence of $\bar{K}_I$ on relative humidity is similar to the humidity dependence of the critical stress intensity factor (K_{IC}) (critical refers to the case when crack growth becomes unstable and fracture occurs) determined by the author in previous work with centrally notched beam specimens (10).

Three humidity dependent processes, which may modify the effect of applied stress on cement paste microstructure, are possible contributory factors to the humidity dependence of the stress intensity factor. These include the following: 1) meniscus rupture in capillary pores due to drying (35-20% RH), which releases the compressive stress on the solid due to meniscus forces; 2) aging phenomena, a term used to describe large changes in surface area on first drying accompanied by large irreversible shrinkages; 3) stress corrosion processes at crack tips involving hydroxyl ion attack on the silicate network.

Meniscus rupture in capillary pores should be more effective in higher w/c ratio specimens, which contain a larger amount of capillary pores. This is consistent with the observation of maxima at 32% RH in the curves of $\bar{K}_I$ versus RH (Figure 4) for specimens prepared at w/c > 0.40.

For specimens made at w/c ratios ≤ 0.35 , the maximum occurs at 11% RH. It is not clear why this maximum would occur at a lower humidity for these w/c ratios. There are, however, major changes to the microstructure of cement paste when the w/c is decreased to values ≤ 0.35 . These microstructural differences include lower values for surface area, hydraulic radius and solid density. In these lower w/c systems, meniscus rupture may play only a minor role.

Collapse of structure due to removal of interlayer water from C-S-H sheets, which comprise a significant portion of cement paste microstructure, occurs on drying between 11% and 0% RH (11). This process may contribute to the increase in stress intensity factor on drying the w/c = 0.40 specimen from 11 to 0% RH.

Evidence has been insufficient to establish whether a stress-corrosion mechanism is operative and if it contributes to the decrease in stress-intensity factor as humidity is increased from 32, or 11% RH to 100% RH in the cement paste specimens. A large amount of evidence, however, indicates that a stress-corrosion mechanism, involving the hydroxyl ion attack of the silicate network, is operative in glass systems (3). Evidence for a similar stress-corrosion mechanism in cement paste will be presented in another paper.

Region II

The existence of region II in cement paste systems has been suggested by Nadeau et al (6). However, no clear evidence for this was revealed in their studies, which involved the use of relatively thick double-torsion specimens prepared at w/c = 0.50 and tested at 100% RH. Their $\log V\text{-}\bar{K}_I$ curve is similar to the curve obtained in this work (Figure 2f); region II was not observed, in this study, for w/c = 0.50 specimens at any humidity, suggesting that if the porosity is sufficiently high (at w/c = 0.50, porosity = 36%), region II will not appear. The volume of capillary pores and permeability are highest at w/c = 0.50 and these characteristics facilitate the transport of water to crack tip sites in these specimens.

At 32% RH, region II is present in the $\log V\text{-}\bar{K}_I$ curves for all w/c ≤ 0.40 (Figure 5a). At 11% RH, region II was observed for the w/c = 0.35 specimen only (Figure 5b). An examination of the stress-intensity factor ($\bar{K}_I$) versus porosity curves for specimens tested at 32 and 11% RH (Figure 6) may be useful in understanding the humidity dependence of region II. For tests performed at

FIG. 5

Crack velocity - stress intensity curves for cement paste tested at 32% and 11% relative humidity.

32% RH, K_{II} increases to a maximum as porosity decreases from 36 to 22%. The maximum was obtained for specimens prepared at $w/c = 0.40$. For tests performed at 11% RH, K_{II} increases continuously as porosity decreases. The appearance of region II in a $\log V-K_I$ curve for cement paste appears to depend on both porosity and humidity. Meniscus rupture events that occur in the humidity region of 32% RH coincide with a high frequency of appearance for region II.

A well-defined region II was also observed in the $\log V-K_I$ curves for the $w/c = 0.45$ specimens tested in the dry condition.* Region II was observed for the non-porous glass sample at 0.017% RH (Figure 3).

Region III

Region III in the $\log V-K_I$ curve was observed only for the $w/c = 0.25$ and 0.35 specimens tested at 32% RH. The reason is not clear.

Conclusions

Sub-critical crack growth in cement paste is dependent on the applied stress-intensity factor and on the amount of water in the environment. It is also dependent on the water/cement ratio and, hence, on porosity of the paste.

There are some similarities in the $\log$ crack velocity - stress intensity factor ($\log V-K_I$) curves for cement paste and soda-lime silica glass. A non-linear region where crack velocity does not depend strongly on the applied stress intensity factor is present in some $\log V-K_I$ curves for both cement paste and glass systems.

*The actual humidity of the dry-state lies between 0.003 and 0.046% RH. The lower figure corresponds to the D-dry state; the upper figure is determined using the vapor pressure obtained with the magnesium perchlorate desiccant used to control the dry-state in the environmental chamber.

FIG. 6
Median stress intensity factor from region I of each $V-K_I$ curve versus porosity for cement paste; tests conducted at 32% and 11% relative humidity.

The non-linear region in the $\log V-K_I$ curves for cement paste is present most often for specimens tested at 32% RH and this may be related to meniscus rupture of capillary pore water, which occurs when cement paste is dried to about 32% RH.

A second linear region in the $\log V-K_I$ curve (which has been identified in glass systems as being environment independent) exists in cement paste made at $w/c = 0.25$ and 0.35 and tested at 32% RH.

Stress-corrosion processes at crack tips involving hydroxyl ion attack of the silicate network in cement paste may contribute to the decrease in crack resistance at humidities greater than 32% RH.

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