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THE FRACTURE OF C-S-H AND C-S-H/CH MIXTURES

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ABSTRACT

The fracture behavior of six well-characterized synthetic preparations of C-S-H and C-S-H/CH mixtures was investigated. The C/S ratios of the C-S-H ranged from 0.68 to 1.49. Log crack velocity vs. stress intensity (log V vs. K_I) curves were obtained for compacted double-torsion specimens prepared with a wide range of porosity values. The dependence of the critical stress intensity factor K_c on porosity, C/S ratio, and polymer/dimer ratio of the C-S-H was determined. The results indicate that an optimum mixture of short and long silicate chains is required for maximum fracture resistance of C-S-H. *© 1998 Elsevier Science Ltd*

Introduction

Fracture mechanics and crack growth of hydrated Portland cement systems have been extensively studied (1–3). The fracture behavior of individual cement minerals and their hydrates, however, is generally not well known. Furthermore, it is believed that the fracture characteristics of pure calcium silicate hydrate (C-S-H) phases have not been reported.

The C-S-H, the principal binding phase in Portland cement concrete, is a product of variable stoichiometry and is considered the major contributor to the engineering performance of concrete. The C/S and H/S ratios can vary with composition of cement, curing temperature, use of supplementary cements, hydration time, and numerous other factors.

The impact of computer and information sciences on concrete technologies is evidenced by the recent growth of computer-based simulation models, databases, and artificial intelligence systems (4,5). Microstructural and simulation models of cement hydration require basic information on material properties and behavior to maximize relevance and effectiveness. The development and maintenance of quality expert systems also relies on accurate input data.

The fracture behavior of pure C-S-H preparations is the focus of this work. The results of crack growth studies of compacted C-S-H specimens (covering a wide range of C/S ratios, 0.68–1.49) are reported. The objectives are to provide original data for simulation models and to further understanding of the fracture process in cement-based materials.

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TABLE 1
Chemical and physical characteristics of C-S-H.

C-S-H	C/S	H/S	Density	Surface area	Monomer	Dimer	Polymer
			g/mL	m^2/g		%	
1	0.99	2.49	2.05	110.7	50	15	35
2	1.26	6.69	2.02	40.2	36	11	53
3	1.49	5.53	2.03	30.7	35	15	50
4	0.68	1.91	2.00	111.9	85	3	12
5	0.87	2.01	2.05	56.6	67	6	27
6	1.17	2.58	2.07	68.2	50	13	37

Experimental

Materials and Methods

C-S-H. Six C-S-H preparations were prepared and supplied by Lafarge Coppée Recherche. A summary of the chemical and physical characteristics is given in Table 1. Details of C-S-H synthesis are provided elsewhere (6). The silica polymerization measurements were made according to the method of Parrott and Taylor (7). Determination of C/S ratio utilized flame spectrophotometry and X-ray microanalysis methods. Determination of H/S utilized the ignition mass loss at 1000°C assuming that the whole loss was due to water.

CH. Reagent grade calcium hydroxide was added to the C-S-H powders for selected experiments.

Porosity Determination. Porosity values of the C-S-H specimens were calculated from the specimen dimensions and mass using the density values determined from the helium pycnometry data.

Specimen Preparation. The C-S-H or C-S-H/CH (mixtures containing 25% by mass of CH) powders were compacted in a high-pressure mold (at pressures of 175, 350, and 525 MPa) to form thin plates. The plates were nominally 25 mm long × 12.5 mm wide and about 1 mm thick. A 2-mm slot was cut in the centre at one end of the plate. On the bottom side of the compact test specimen a scratch (approximately 0.1 mm deep) was made along the centre line to provide a path for crack propagation. All operations were carried out in a glove box flushed with nitrogen gas to minimize the effects of carbonation. Determinations of the extent of carbonation before and after testing indicated that this method was effective.

Fracture Testing. All crack propagation studies were performed in an environmental chamber mounted on the cross-head of an Instron testing machine. The tests were all performed at 11% relative humidity. The test procedure employing a "double-torsion" specimen has been described elsewhere (8).

A load-relaxation technique was used to determine the log V-K_I curves for the compacted

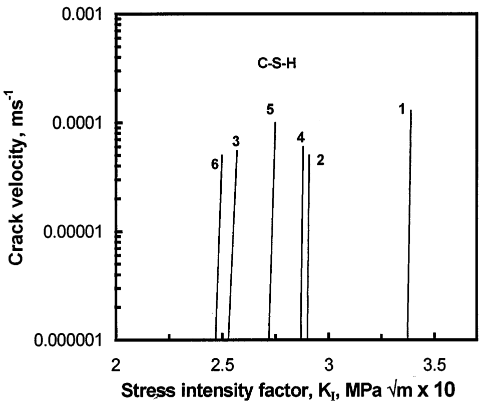

FIG. 1.
Log crack velocity vs. stress intensity factor curves for synthetic C-S-H compacted specimens prepared at 350 MPa (numbers refer to various C-S-H preparations described in the text).

C-S-H specimens (9). An initial cross-head speed of 8.35×10^{-5} mm s^{-1} was used. Crack velocity was determined from the relaxation curves using the following relation:

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

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 preparation at each compaction pressure. These curves are numerous and are not presented because the ratio A/B determined from them is usually $\ll a_i$ and can be neglected. Values of the derivative dP/dt were obtained from the load-relaxation curve.

The method of Williams and Evans (9) was used to compute the values of stress intensity factor K_I. The critical value of K_I or K_c was determined at maximum load. Multiple relaxation curves were obtained for each C-S-H preparation. Values of K_c reported are the average of at least six determinations.

Results

Selected log crack velocity vs. stress intensity factor curves for two sets of specimens comprised of C-S-H and mixtures of C-S-H and CH (25% CH by mass) are shown in Figures 1 and 2. The specimens represented by these curves were all compacted at 350 MPa. The curves having highest and lowest K_I values are represented by the same C-S-H materials for both sets of specimens, i.e., with and without CH. The curves for C-S-H alone have K_c values ranging from 0.25 to 0.34 MPa. Those for the C-S-H/CH mixtures have K_c values ranging from 0.25 to 0.33 MPa. The sequence (with respect to the type of C-S-H) of increasing K_c values is not the same for both sets of curves. It is noted that the porosity values for the pure C-S-H range from 29.8 to 37.2% and those for the C-S-H/CH mixtures from 25.5 to 35.5%.

The porosity dependence of the critical stress intensity factor, K_c, is described in Figure 3. The data for all the C-S-H and C-S-H/CH specimens appear to be best described by a linear

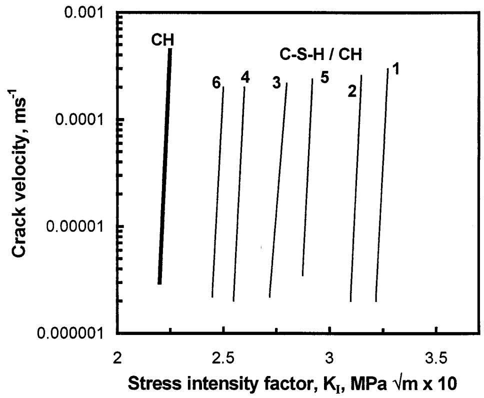

FIG. 2.

Log crack velocity vs. stress intensity factor curves for CH and synthetic C-S-H/CH compacted specimens prepared at 350 MPa (numbers refer to various C-S-H preparations described in the text).

relationship independent of C/S and H/S ratio. The regression equation is $K_c = 5.13 - 0.69$ (porosity) with a correlation coefficient of 0.86. The data for the pure CH system is also described by a linear relationship. The regression equation is $K_c = 5.18 - 0.118$ (porosity) with a correlation coefficient of 0.91. The K_c values for CH are lower than those for the C-S-H systems at equivalent porosity values. This is consistent with observations that K_c values of CH are lower than those of hydrated cement paste at the same porosity (10).

A plot of K_c vs. C/S ratio for specimens compacted at 350 MPa is given in Figure 4. The K_c parameter appears to be generally independent of C/S ratio for both pure C-S-H and mixtures of C-S-H and CH. It is noted, however, that the highest values of K_c are obtained for C-S-H preparations having a C/S ratio of 0.99. Close examination of the K_c vs. porosity

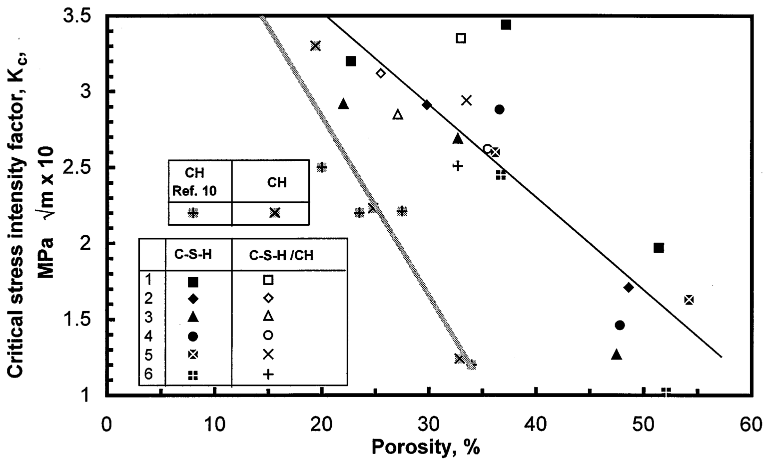

FIG. 3.

Critical stress intensity factor, K_c vs. porosity for compacted specimens of synthetic C-S-H, C-S-H/CH mixtures, and CH.

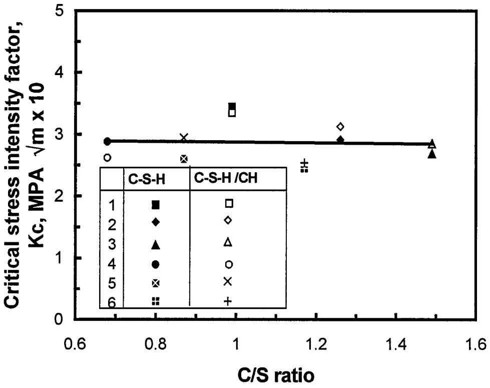

FIG. 4.

Critical stress intensity factor, K_c, vs. C/S ratio for compacted specimens of synthetic C-S-H and C-S-H/CH mixtures.

data (Fig. 3) indicates that the K_c values are the highest for the C/S ratio 0.99 preparations. This preparation contained the highest amount of dimer (similar to preparation 3) and an intermediate amount of polymer. A plot of K_c vs. polymer content (not shown) gives similar trends.

K_c is plotted vs. the polymer/dimer ratio for each of the C-S-H materials in Figure 5. The data indicate that there may exist a polymer/dimer ratio that gives a minimum value of K_c. The C/S ratio 0.99 preparation had the lowest polymer/dimer ratio and the highest K_c value. The other preparations were linearly related to the polymer/dimer ratios. Regression analysis gives the relation $K_c = 1.78 + 0.25$ (polymer/dimer ratio). The correlation coefficient is 0.95. The relatively low slope would indicate that the dependence is not strong. A maximum K_c value for the C/S ratio 0.99 preparation would not appear to be inconsistent with Sato and Grutzeck's model for C-S-H structure (11). Sato and Grutzeck described a model for C-S-H with C/S ratio >1 or <1. The C-S-H contains primarily short silica chains and dimer when

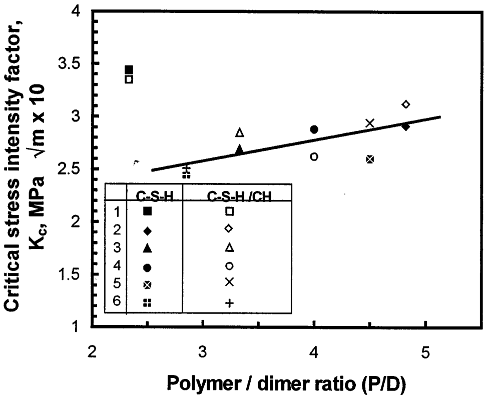

FIG. 5.

Stress intensity factor vs. polymer/dimer ratio of synthetic C-S-H in compacted C-S-H and C-S-H/CH systems.

the C/S ratio >1. The C-S-H with C/S ratio <1 contains long and cross-linked silica chains. It would appear from this work that there is an optimum mixture of short and long silicate chains for maximum fracture resistance.

The work of Crennan, El-Hamaly, and Taylor on autoclaved systems is relevant. They studied factors influencing strength of autoclaved lime-quartz materials (12). They determined the C/S ratios of the C-S-H formed in their systems to range from 0.83 to greater than 3.00. The preparation containing C-S-H with C/S ratio 0.83 had strengths of about 60 MPa. The compressive strength of the autoclaved materials then abruptly increased to a maximum and then decreased with an increase in the C/S ratio (up to 0.3) of the C-S-H. However, the data in the region of C/S ratio, 1 to 1.5, is highly scattered and there is no discernible dependence of compressive strength on C/S ratio. Maximum strength values (>100 MPa) were obtained for the preparations containing C-S-H with C/S ratios of 0.99 and 1.09. It was concluded that if the system is largely composed of C-S-H, the distribution of particle types within the C-S-H has a major effect on strength. These results would also appear to be consistent with those described above for the synthetic C-S-H materials as well as the Sato-Grutzeck model.

Conclusions

1. The fracture of C-S-H and C-S-H/CH mixtures is dependent on porosity for a wide range of C/S and H/S ratios.
2. The fracture resistance of CH is lower than that of C-S-H over a wide range of porosity.
3. The fracture of C-S-H and C-S-H mixtures was generally independent of C/S ratio and polymer content for most of the C-S-H preparations studied. The maximum fracture resistance, however, was observed for specimens prepared with C-S-H having C/S ratio 0.99.
4. There appears to be a value of the polymer/dimer ratio of C-S-H that gives a minimum fracture resistance. The C-S-H with highest fracture resistance had the lowest value of the polymer/dimer ratio.
5. There appears to be an optimum mixture of short and long silicate chains for maximum fracture resistance of C-S-H consistent with structural models proposed by Sato and Grutzeck.
6. It is apparent that computer simulations of hydrating cement systems need to identify the type and characteristics of the C-S-H present in order to input the appropriate engineering properties.

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