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FRACTURE TOUGHNESS OF A SOLIDIFIED COMPOSITE RESIDUAL MATERIAL

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ABSTRACT

An experimental investigation was conducted to determine the fracture properties of a solidified composite produced from residual sludge of the steel industry. The purpose of this study was to investigate the causes of random cracking and spalling which were observed in the previous field tests. Fracture toughness (or stress intensity factor), K_{1c} values were determined using compact tension specimens which were prepared from concrete-like solidified material. These values were used as a gauge to determine the relative effects of various factors on the cracking behavior of this composite material, such as the mix component ratios. The specimens were prepared with different specifications and were cured at different environmental conditions. The best mix and the best suitable curing temperature were investigated.

Introduction

The feasibility of using solidified industrial sludge residue as a capping material for an existing site was investigated in the field. The test plots showed random cracking and spalling after one period of freeze-thaw. The objective of this study was to investigate in the laboratory the causes of the cracking and spalling of this solidified material. The composite material was a mixture of dewatered steel process sludge, (which will be referred to as "filter cake" (FC) from here on) cement, kiln slag and water. The solidification process was undertaken to utilize the product as a capping material for existing waste impoundment lagoon. Details of that particular work and field results have been published elsewhere (1, 2). In this paper, it is attempted to characterize the cracking behavior by relationships obtained between the unconfined compressive and tensile strengths versus the fracture toughness of the solidified material cured under different conditions.

The fracture toughness of the composite material was determined through linear elastic fracture mechanics (LEFM) theory was used. The LEFM theory has been used quite successfully in predicting crack propagation in metals for the last few decades. It also has been applied to predict fracture behavior of concrete and rock (3) and cement-based materials (4). Theories of fracture mechanics have been applied to concrete as early as 1961 (5). According to linear elastic fracture mechanics, the near-tip stresses around a crack are proportional to stress intensity factor of the material which characterizes the crack propagation. The stress

intensity factor is K_{1c} in units of MPa.$m^{1/2}$ in SI system. The composite material used in this work is a concrete-like material with aggregate and har dened cement paste. Therefore, it was considered a notch sensitive material. Based on this brief background the specific objectives of the investigation reported in here can be itemized as below: a) To develop a suitable test procedure for determining the fracture toughness or stress intensity factor K_{1c} of the solidified residual material described above; b) To study the effect of mix compound ratios, curing conditions, and curing durations of the mixture on the measured fracture toughness; c) To determine the relationships between fracture toughness of the material versus the unconfined compressive, and the unconfined tensile strengths, d) To determine the best mixture and curing condition based on the established cracking potential using the fracture toughness analysis.

Determination of Fracture Toughness

Compact tension (CT) specimens are frequently used to determine the fracture toughness of com posite materials (6, 7, 8). In this study the fracture toughnesses were determined using the manufactured CT specimens as shown in Figure 1.

Method of fracture toughness evaluation is based upon ASTM Standard practice that currently exists for the establishment of plane-strain fracture toughness, K_{1c}. In this study, ASTM standard test recommendations for plane-strain fracture toughness of metallic materials (E 399-83) and also the recommendations given in the (9) were used. The fracture toughness or the SIF values were calculated using the following formula:

$$K_{1c} = \frac{P(2W + a)}{\sqrt{\pi}h(W - a)^{3/2}} F\left(\frac{a}{W}, \frac{b}{W}, \frac{c}{W}\right)$$

where, P is the fracture load (kN), "a" critical crack length, "b","c","W","h" are specimen dimensions as shown in Figure 1. "F(a/W, b/W, c/W)" is a function of dimension parameters. This formula is given in the (11). Although, a slightly different is recom mended in ASTM Standard E 399-83, that equation was used because it incorporates all pertinent dimensions of

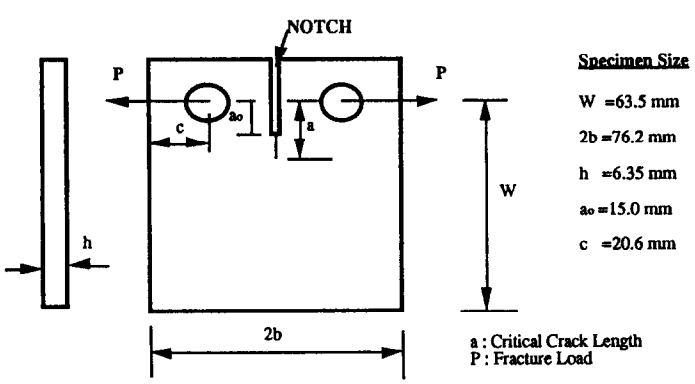

FIG. 1.
The sizes of the CT specimens.

TABLE 1
The Amounts of Main Components

Components (% by total mass)			
Water	Cement	Filter Cake	Slag
10	16	39	35
12.5	16	37	34.5
16	16	33	35

a CT specimen. The value of the function F(a/W, b/W, c/b) was obtained from tabulated solution of the function for CT specimens (11). This value was determined to be 1.465 for a/W = 0.236, b/W = 0.6, c/b = 0.54.

Test Program

As a capping material, the solidified product might be subjected to polluted water infiltration, therefore Type II Portland cement (C) was used as a precaution against moderate sulfate attack. The other components of the composite material were filter cake (FC), kiln slag (S) and water (W).

FC is a by-product of steel process sludge. It is mainly particulate material of average size 5-10 μ. It contains various oils and greases at about 4-8 % by total weight, Fe products by 24 %, CaO by 11%, and other inorganic species such as Zn, Al_2O_3 at smaller proportions. It's water content is 45 % by total weight and specific gravity is 1.56. The compact tension and tensile strength test specimens were prepared with portion of slag passing #28 mesh (less than 0.595 mm). This variation was done due to the specimen size limitation of the CT specimens, thickness of were 6.35 mm. Water content of slag is 9 % by weight of total weight and specific gravity is 3.57. The proportions of components used in the test mixtures are given Table 1.

The mixing water contents were varied as 10, 12.5 and 16 by percent of total mass, and the curing temperature was varied from -20 to 100°C. One set of the fracture specimens was prepared with air entrainment reagent AEA of increasing proportions with constant mixing water content. A similar set was prepared with increasing proportion of a plasticizer (superplasticizer) reagent at constant mixing water content. The humidity was controlled in one set of specimens. The speci mens were cured in a moist room at constant humidity of 95 %, and at constant temperature of 28°C. One set of specimens was left outside to be cured in ambient temperature and humidity which varied over time. The average of day and night temperature over the period of 28 days curing was estimated to be 20°C. The average relative humidity for the same period of time was about 65 %. One set of the specimens was left to dry in the laboratory which was air-conditioned. The average day and night temperature was around 25 °C and the relative humidity was around 20 %. The other set of specimens were cured in the oven at 40 and 100°C and in the refrigerator at -4 and -20°. The frozen specimens were left to thaw in room temperature for few hours prior to testing. A bench type laboratory mixer was used for mixing slurried composite. First slag and cement were put in the mixer then FC was added. After 3-4 minutes of dry mixing, the mixing water was added to the mixture. In preparation of samples with reagents such as the air entrainment and the plasticizer, these reagents were pre-mixed into the water. The notch was created by in serting a thin metal part

into the pre-cut groove on each mold section. The slurry set up in 3-4 hours, after being poured into the molds. They were then taken out of the molds and introduced into their respective environment for curing. All of the tests were conducted on triplicate specimens.

Test Results

Variation of SIF with Mixture Compound Ratio. W-C and W-FC Ratios: The relationships between the K_{1c} values and the W-C and W-FC ratios are shown in Figure 2. The maximum K_{1c} value obtained for all the specimens tested was around 0.04 MPa.m$^{1/2}$ at an approximate W-C ratio of 0.8. Also shown in Figure 2 is the variation of K_{1c} values with curing conditions. Compact tension specimens tested in this group were cured in five different environments.The specimens that were kept in random ambient temperature (outside) resulted in the highest K_{1c} values. The K_{1c} values obtained from the specimens cured in the refrigerator at -20° and in the oven at 100° temperature were lower than those values obtained for specimens cured under other environmental conditions. Evaluation of Figure 2 shows that the specimens kept in the moist room were affected by the changing W-C ratio significantly more than the others. The specimens that were cured in -20° and 100° of temperature did not show appreciable change in stress intensity factor values, however, they exhibited a steady decrease with increasing W-C ratio. The largest difference is observed with the values obtained for moist room specimens for the K_{1c} value decreased from around 0.04 to around 0.01 MPa.m$^{1/2}$ at the high and the low W-C ratios of 1.00 and 0.625, respectively. The K_{1c} values of specimens cured outside decreased from 0.04 to around 0.03 MPa.m$^{1/2}$ at the these same W-C ratios.

The increasing W content appears to reduce the fracture resistance in general. This may be due to higher proportion of voids attained at higher W contents. The significant decrease in K_{1c} for the moist room cured specimens may have been caused by the lubricating and softening effect of excess water that could have entered through existing surface cracks near the notch. The same phenomena may be true for the low W-C ratio for which the initial cracks may be due to non-reacted components at low W content.

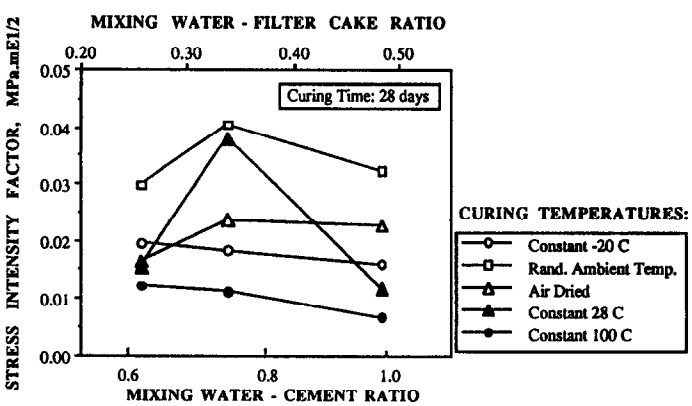

FIG. 2.
The variation of 28 days SIF values with W-C and W-FC ratios.

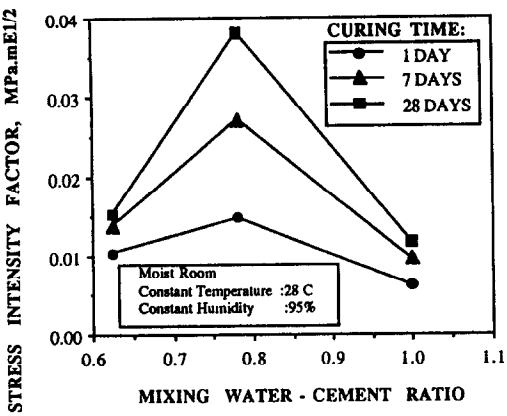

FIG. 3.
The variation of SIF values with W-C ratios for moist room curing.

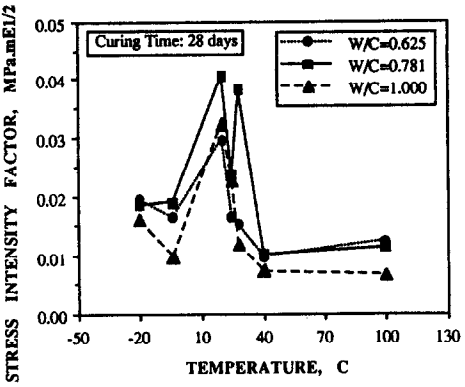

FIG. 4.
The variation of SIF values with temperatures.

One of the objectives of this particular project has been to maximize the amount of FC, a by-product, used in the solidified material for beneficial recycling. The effect of increased FC on the fracture behavior was generally observed to be negliable if the C content was kept at a constant value. However, this also meant change W content as S and C proportion was kept constant. The FC is a fine particulate material with considerable affinity to W. The relatively lower fracture toughness measured at low mixing W-FC ratios may be due to the insufficient W for hydration and pozzolonic reactions due to the adsorption capacity of the FC. At higher W values the behavior is probably more sensitive to the W-C ratio rather than the mixing W-FC ratio.

Reagent-Water Ratios: It appeares that the increased proportion of the air entrainment reagent did not have significant effect on the K_{1c} values except those of specimens cured in the moist room. At high AEA content, the increased void space in the matrix probably contributed to the low value of K_{1c}. The results with respect to the reagents show: 1) the K_{1c} values are probably more sensitive to W-C ratio than the reagent proportions used in this work, 2) the relative stacking of the K_{1c} values with curing conditions is dublicated with constant W-C and W-FC ratio.

Variation of SIF with Curing Duration and Temperature. Curing Duration: The variation of K_{1c} values with W-C ratio for the 1, 7 and 28 days cured specimens in moist room is shown in Figure 3, respectively. The maximum K_{1c} values are achieved at 28 days at the W-C ratio of 0.781. At that W-C ratio, the humidity cured specimens gain 50 % of the maximum K_{1c} values in 1 day and 75 % of it in 7 days. The K_{1c} values are approximately the same for 0.625 and 1.00 W-C ratios for the 1, 7 and 28 days of curing durations. Similar results were obtained for the ambient condition cured specimens. There is a consistent increase of K_{1c} with time under both curing conditions. It appears that there is an optimum W-C ratio which corres ponds to the highest K_{1c} value. In general, if the K_{1c} value is low to start with, a high humidity curing

environment (95%) does not improve this value whereas a moderate humidity (65%) environment appears to improve it steadily with curing time. This is observed readily in Figure as the entire curve shifts upward steadily with curing time.

Curing Temperature: The variation of K_{1c} values with temperature for three different W-C ratios is shown in Figure 4. The high K_{1c} values coincide with moderate temperature range curing at around 20°C. At curing temperatures higher than 40°C, the material exhibits low fracture toughness, probably due to the fast rate of evaporation of water. Low humidity environments, such as air conditioned indoors and also refrigeration affect to reduce K_{1c}. High humidity environment is beneficial for the development of resistance against cracking, if the solidified material does not produce initial cracks prior to curing. In such cases, the excess moisture probably produce adverse effect by entering into the matrix through the cracks and act as a softening or lubricating agent, eroding the material further. Moderate amount of humidity is probably beneficial to activate any "self-healing" capacity the material might possess.

Summary and Conclusions

The general conclusion of this study is that fracture toughness measurements of solidified composite materials such as the solidified steel industry residue material can be utilized to access their short term cracking behavior. Correlations developed between fracture toughness and strength of the material may be viable to establish better under standing of the cracking behavior of such composite materials. The following specific conclusions were derived from this study:

1. The maximum fracture toughness value of the solidified steel industry residual material used in this study was determined 0.0405 MPa.$m^{1/2}$ at 0.781 W-C ratio of the the mixture. The unconfined compressive strength was 0.7334 MPa, and the unconfined tensile strength was 0.0802 MPa at the same W-C ratio.

2. The specimens that were kept outside at moderate ambient temperature and humidity (20°C, 65%) resulted in the highest values of fracture toughness and strength consistently.

3. The fracture toughness of the composite material appeared to be influenced adversely by the water-cement ratio at high water contents, and by the water-filter cake ratio at low water contents. Fracture toughness measurement may also be adversely affected by high humidity environment prior to final setting.

4. The reagents such as air-entrainment and plasticizer did not appear to influence fracture toughness at the proportions added to the mixture in this study.

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