Cement and Concrete Composites 79 (2017) 71–75

Contents lists available at ScienceDirect

Cement and Concrete Composites

journal homepage: www.elsevier.com/locate/cemconcomp

Fracture properties of the alkali silicate gel using microscopic scratch testing

Caroline V. Johnson [a], Jiaxin Chen [a], Nicole P. Hasparyk [d], Paulo J.M. Monteiro [c], Ange Therese Akono [a, b, *]

[a] Department of Civil and Environmental Engineering, University of Illinois at Urbana-Champaign, 61801, USA
[b] Department of Mechanical Science and Engineering, University of Illinois at Urbana-Champaign, 61801, USA
[c] Department of Civil and Environmental Engineering, University of California, Berkeley, 94720, USA
[d] Department of Technical Support and Control, Aparecida de Goiânia/GO 740001-970, Brazil

ARTICLE INFO

Article history:
Received 3 June 2016
Received in revised form
18 October 2016
Accepted 26 January 2017
Available online 9 February 2017

Keywords:
Scratch test
Alkali-silica gel
Fracture toughness

ABSTRACT

We carry out a multi-scale investigation of the fracture properties of the gel produced by the alkali-silica reaction in concrete. Milimeter-sized alkali gels are tested via scratch tests at the micrometer level. Scanning electron microscopy reveals curved fracture surfaces produced during the scratch test, warranting a fracture mechanics analysis. By application of a nonlinear fracture mechanics model to the recorded load and depth data, we estimate the fracture toughness of ASR gel to be $K_c = 0.62$ MPa$\sqrt{m}$. In turn, by combining micro-indentation and scratch tests, we can estimate the fracture energy $G_f = 11.2$ J/m^2. These experimental results are important for the multi-scale modeling of the alkali-aggregate reaction.

1. Introduction

The alkali-silica reaction, ASR, can cause significant distress in concrete structures. The reaction occurs in concretes between amorphous or poorly crystalline phases in the aggregate with alkali and hydroxide ions existing in the pore solution of the cement matrix. The reaction produces an amorphous silicate gel which swells by adsorbing water and causes micro-fractures. The gel expands as it absorbs water from the surrounding concrete, causing micro-fractures. Over time, the presence of ASR can lead to the decrease of stiffness and strength of concrete structures [9,15,21,23].

Several methods have been proposed to measure the mechanical properties of ASR gel. The challenge is that the gel is often intermixed with hydration products in the complex cement matrix. Sometimes, it is possible to obtain centimeter-size gels from the inside of the galleries in a dam; but even so, the ASR gel crystals are typically too small (less than 10 mm) to perform traditional macroscopic tests such as uniaxial compression or 3-point bending tests. Recently, Brillouin spectroscopy [17] was employed to characterize the Young's modulus of ASR gel. Microscopic indentation [12] has also been proposed to assess the elastic properties. However to our knowledge, no method has attempted to measure the fracture toughness of ASR gel. In turn, understanding the fracture behavior of ASR gel is important to fully describe concrete deterioration and cracking due to the alkali silica reaction.

We present a novel study that employs instrumented scratch testing in order to assess the fracture toughness of ASR gel at the microscopic level. This paper is organized as follows: first the materials are presented. Then, a brief overview of scratch testing for fracture assessment is presented. Finally, tests are carried out on ASR gel specimens and the results are discussed.

2. Materials and methods

2.1. Materials

Samples were collected from the Furnas dam in Brazil and their physico-chemical properties were described elsewhere [4,10,22]. As shown in Fig. 1a), the glassy specimens measured less than one cubic centimeter each. Because of the small size of the samples, macroscopic testing would not have been effective, hence we focused on microscopic methods. Due to the hydrophilic nature of

* Corresponding author. 3108 Newmark Civil Engineering Laboratory, 205 N. Mathews Ave.Urbana, IL, 61801, USA.
E-mail address: aakono@illinois.edu (A.T. Akono).

http://dx.doi.org/10.1016/j.cemconcomp.2017.01.012
0958-9465/

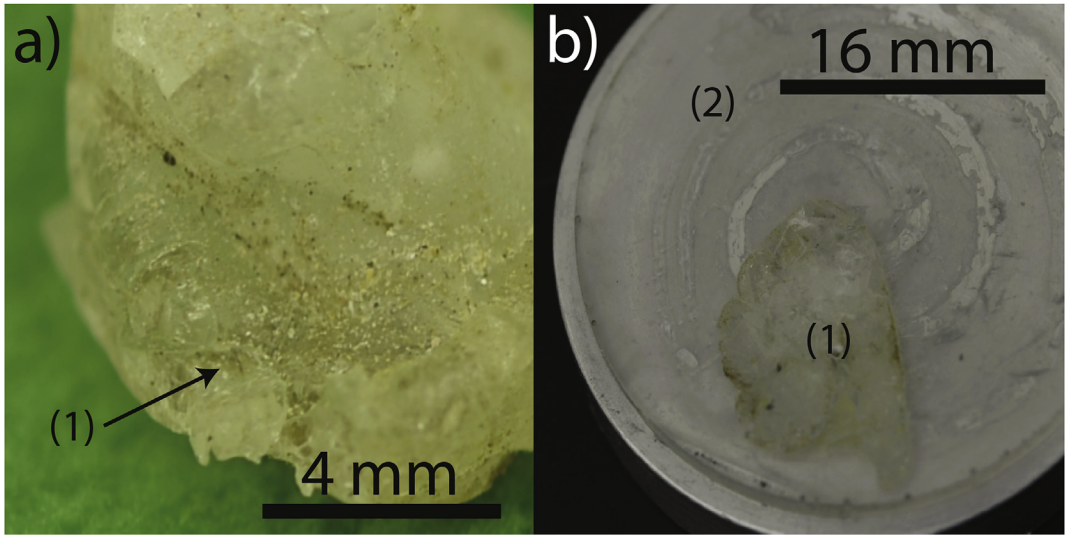

Fig. 1. a) Digital photography of an ASR gel crystal collected. b) ASR gel specimen embedded in epoxy resin. (1) indicates the ASR gel and (2) indicates the epoxy resin. Both images were captured using a digital single-lens reflex (DSLR) camera Nikon D800E along with a Nikon AF-S Micro-Nikkor lens. Credit: Caroline Johnson, Jiaxin Chen, Ange-Therese Akono, UIUC, 2016.

the samples, it was important to store them away from moisture. Prior to preparation, specimens were wrapped in plastic and stored in a dry location at ambient temperature. After preparation, the specimens were kept in a desiccator under vacuum at room temperature.

2.2. Methods

2.2.1. Specimen preparation procedure

Each ASR gel specimen was embedded in a 32-mm cylindrical mold filled with EpoxiCure™resin (Buehler, LakeBluff, IL). Epoxy embedding is used to provide structural support and facilitate the metallographic polishing phase. Specimen 1 was simply embedded whereas specimen 2 was embedded in epoxy under vacuum. In particular, during vacuum embedding, the epoxy resin is pushed into microscopic pores resulting in a smoother surface. Prior to testing, the embedded specimens were cut to micron-precision using an Isomet 5000™(Buehler, LakeBluff, IL) diamond saw. Each resulting 5-mm thick and 32-mm round cylinder was then glued to a circular aluminum disk using cyano-acrylate glue for further metallographic polishing.

The objective of the metallographic polishing step is to yield a uniformly flat and smooth surface with a low roughness so as to increase the accuracy of small-scale mechanical testing. The rule of thumb is to have a surface roughness less than one fifth of the maximum penetration depth [16]. Surface polishing was done using Carbimet™(Buehler, LakeBluff, IL) abrasive paper. Specimens 1 and 2 were prepared using separate procedures. For specimen 1, several grit sizes were used: 400, 600, 800 and 1200 consecutively for 2 min each. In between each grit size, the specimen was rinsed in *N*-decane using ultrasonic waves. Fig. 2 displays the optical microscopy images of the surface after grinding and polishing. Specimen 2 used grit sizes of 240, 400, 600, 800, and 1200 consecutively for 1, 5, 10, 20, and 30 min respectively. Specimen 2 was then polished with diamond paste of 3-μm and 1-μm particle size, for 60 and 90 min respectively. The same cleaning protocols as Specimen 1 were used between steps. Vacuum embedding leads to a finer surface with the absence of surface scratches. Nevertheless both specimen 1 (air-embedded) and specimen 2 (vacuum-embedded) exhibit an amorphous microstructure, which is characteristic of alkali silicate gels.

2.3. Scratch testing

A scratch test consists in pushing a sphero-conical diamond stylus across the surface of a softer material while linearly increasing the vertical force as shown in Fig. 3a) and b). The basics of the test date back to the hellenic era where scratching was utilized to rank minerals according to their relative hardness. The advent of instrumented scratch testing in the late 1980s led to a broader range of applications for scratch testing including, but not limited to, strength of ceramics [13], damage of metals and polymers [6], cohesion and adhesion of coatings [8] and quality control of thin films [7].

All tests were conducted using an Anton Paar (previously CSM-Instruments) Micro-Scratch Tester. The multi-scale experimental platform consists of a scratch testing head in series with a Nikon optical video microscope. In particular, the microscope and the scratch testing unit are synchronized so as to select the location of the test with high accuracy and image the surface after testing. Our tests were carried out using a Rockwell C probe that consists of a cone of half-apex angle 60° ending in a sphere of tip radius 200-μm with a sphere-to-cone transition depth of 27 μm. During the tests, the vertical force is prescribed using piezo-actuation whereas the force and displacements are measured using high accuracy transducers.

3. Results and discussion

3.1. Scratch testing of ASR

Scratch tests were carried out on the epoxy-embedded ASR gel. The specimen embedded in air was tested with a maximum vertical force of 30 N. Fig. 3c) displays the horizontal force-penetration depth recorded. The maximum penetration depth is 180 μm. As a result, the scratch probe can be approximated by a cone. In contrast, we used a maximum vertical force of 10 N for the vacuum-embedded specimen. During epoxy-embedding under vacuum, the resin is pushed into the microscopic pores due to the vacuum. Unfortunately, this results in a composite structure at the mesoscopic level consisting of the resin and the ASR gel. Therefore, we purposely selected a lower force to obtain a depth of testing in the microscopic range. The maximum penetration depth in these tests