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Communication

Defect dynamics of cement paste under repeated compression studied by electrical resistivity measurement

Sihai Wen, D.D.L. Chung*

Composite Materials Research Laboratory, Department of Mechanical and Aerospace Engineering, University at Buffalo, State University of New York, Buffalo, NY 14260-4400, USA

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Abstract

Defect dynamics, as studied by electrical resistivity measurement during repeated compression of cement paste in the elastic regime, are characterized by defect generation that dominates during the first loading, defect diminution that dominates during subsequent loading, and defect extension that dominates during subsequent unloading.

Keywords: Microcracking; Microstructure; Electrical properties; Strain effect; Cement paste

1. Introduction

Defects greatly affect the properties of a material, so their control is of practical importance. Stress, heat, and the environment all affect the defects, but this paper is focused on the effect of stress. Previous work on the effect of stress is mainly concerned with stress in the plastic deformation regime [1–6]. In the case of metals and polymer–matrix composites (such as asphalt), plastic deformation, particularly involving compression, can give rise to healing [1–5]. However, the effect of deformation is very different in the case of cement due to the relatively brittle nature of cement. Plastic deformation of cement can lead to damage [6]. This paper is not centered on damage, but on defect dynamics under repeated loading. Therefore, cement is studied in this work within the elastic deformation regime.

Stress application can generate defects (such as microcracks), which may be a form of damage in a material. Stress application can also diminish defects (such as diminution in the form of reduction of the crack separation distance), particularly in the case of the stress being compressive. This diminution is induced by stress and is to be distinguished from healing that is induced by liquids, chemicals, or particles [7]. On the other hand, stress removal can extend defects (such as extension in the form of relaxation and enlargement of crack dimensions at the microscale), particularly in the case of the stress being compressive and the material being brittle. The generation, diminution, and extension of defects during dynamic loading are referred to as defect dynamics, which constitute the subject of this paper. The little prior attention on defect dynamics is mainly due to the dynamic nature of defect diminution and extension. For example, stress application can cause defect diminution, and subsequent unloading can cancel the diminution. This reversible nature of the diminution makes the diminution observable only in real time during loading. On the other hand, defect generation tends to be irreversible upon unloading, so it does not require observation in real time.

Observation in real time during loading is difficult for microscopy, particularly transmission electron microscopy, which is the type of microscopy that is most suitable for the observation of microscopic defects. However, observation in real time during loading can be conveniently performed by electrical measurement. As defects increase the electrical resistivity of a material, defect generation increases the resistivity whereas defect diminution decreases the resistivity.

This paper uses electrical resistivity measurement during repeated compressive loading and unloading to monitor the dynamics of defects in cement paste. As stress affects the strain (i.e., the dimensions), which in turn, affects the elec-

* Corresponding author. Tel.: +1-716-645-2593x2243; fax: +1-716-645-3875.

E-mail address: ddlchung@acsu.buffalo.edu (D.D.L. Chung).

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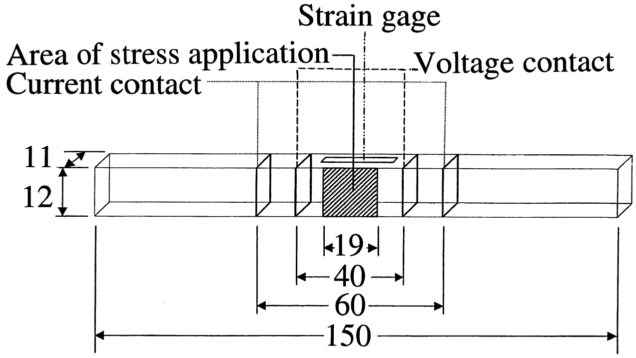

Fig. 1. Sample configuration for measuring the transverse electrical resistivity during uniaxial compression. Dimensions in millimeters (mm).

trical resistance, this work involves simultaneous measurement of resistance and strain. In order to confirm the interpretation of the results in terms of defect dynamics, this work involves measurement of the resistivity in the stress direction (longitudinal resistivity) and that perpendicular to the stress direction (transverse resistivity).

2. Experimental methods

2.1. Materials

The cement used was Portland cement (Type I) from Lafarge (Southfield, MI). A rotary mixer with a flat beater was used for mixing. Water and cement in the ratio 0.35 were mixed in the mixer for 5 min. After pouring the mix into oiled molds, an external electric vibrator was used to facilitate compaction and decrease the amount of air bubbles. The specimens were demolded after 1 day and then allowed to cure at room temperature in air (relative humidity = 100%) for 28 days.

2.2. Testing

For compressive testing according to ASTM C109-80, specimens were prepared using a 2 × 2 × 2 in. (51 × 51 × 51 mm) mold. The strain was measured by using a strain gage attached to the middle of one of four side surfaces of a specimen. The strain gage was centered on the side surface and was parallel to the stress axis. Compressive testing under load control was performed using a hydraulic mechanical testing system (MTS Model 810). Testing was conducted under repeated loading at various stress amplitudes within the elastic regime.

During compressive testing of the cubes mentioned above, the longitudinal DC resistivity was measured using the four-probe method, in which silver paint in conjunction with copper wires served as electrical contacts. Four contacts were perimetrically placed around the specimen on four planes that were all perpendicular to the stress axis and that were symmetric with respect to the midpoint along the height of the specimen. The outer two contacts (typically 40 mm apart) were for passing current. The inner two contacts (typically 30 mm apart) were for measuring the voltage. A Keithley 2001 multimeter was used.

Samples for transverse DC resistivity measurement were in the form of rectangular bars of size 150 × 12 × 11 mm. Each electrical contact was applied around the entire 12 × 11-mm perimeter of the bar. The voltage contacts were on two parallel cross-sectional planes that were 40 mm apart. Thus, the resistivity was measured along the length of the rectangular bar. During the resistivity measurement, compressive stress was applied to the middle portion (19 × 12 mm) of the rectangular sample (Fig. 1), such that the electrical contacts were away from the stressed portion and the stress was in a direction perpendicular to the direction of resistivity measurement. The stress (repeated loading at increasing stress amplitudes) was provided by a hydraulic mechanical testing system (MTS Model 810). The transverse strain was measured using a strain gage attached to a side of the specimen, as shown in Fig. 1.

The resistivity was obtained from the resistance and the dimensions, which changed with the measured longitudinal strain and with the transverse strain resulting from the Poisson effect. Neglecting the transverse strain affected the longitudinal resistivity value negligibly. The fractional change in resistance was essentially equal to the fractional change in resistivity.

3. Results and discussion

Fig. 2 shows the fractional change in longitudinal resistivity as well as the longitudinal strain during repeated compressive loading at an increasing stress amplitude within the elastic regime. The strain varies linearly with the stress up to the highest stress amplitude. The strain returns to zero at the end of each cycle of loading. During the first loading,

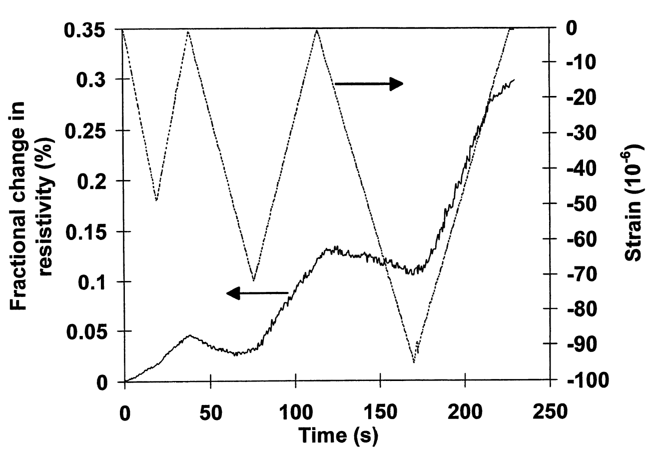

Fig. 2. Variation of the fractional change in longitudinal resistivity with time and of the longitudinal strain (negative for compressive strain) with time during dynamic compressive loading at increasing stress amplitudes within the elastic regime.

the resistivity increases due to defect generation. During the subsequent unloading, the resistivity continues to increase, due to defect extension (such as the opening of the microcracks generated during prior loading). During the second loading, the resistivity decreases slightly as the stress increases up to the maximum stress of the first cycle (due to defect diminution) and then increases as the stress increases beyond this value (due to additional defect generation). During unloading in the second cycle, the resistivity increases significantly (due to defect extension, probably the opening of the microcracks). During the third loading, the resistivity essentially does not change (or decreases very slightly) as the stress increases to the maximum stress of the third cycle (probably due to the balance between defect generation and defect diminution, such as the situation of crack formation and crack closing occurring simultaneously). Subsequent unloading causes the resistivity to increase very significantly due to defect extension (probably the opening of the microcracks).

Fig. 3 shows the fractional change in transverse resistivity as well as the transverse strain (positive due to the Poisson effect) during repeated compressive loading at an increasing stress amplitude. The strain varies linearly with the stress and returns to zero at the end of each cycle of loading. During the first loading and the first unloading, the resistivity increases due to defect generation and defect extension, respectively, as also shown by the longitudinal resistivity variation (Fig. 2). During the second loading, the resistivity first increases (due to defect generation) and then decreases (due to defect diminution). During the second unloading, the resistivity increases due to defect extension. During the third loading, the resistivity decreases due to defect diminution. During the third unloading, the resistivity increases due to defect extension.

The variations of the resistivity in the longitudinal and transverse directions upon repeated loading are consistent in showing defect generation (which dominates during the first loading), defect diminution (which dominates during subsequent loading), and defect extension (which dominates during subsequent unloading). The defect extension during unloading follows the defect diminution during loading, indicating the reversible (not permanent) nature of the diminution, which is induced by compressive stress. The defect extension during unloading also follows the defect generation during loading.

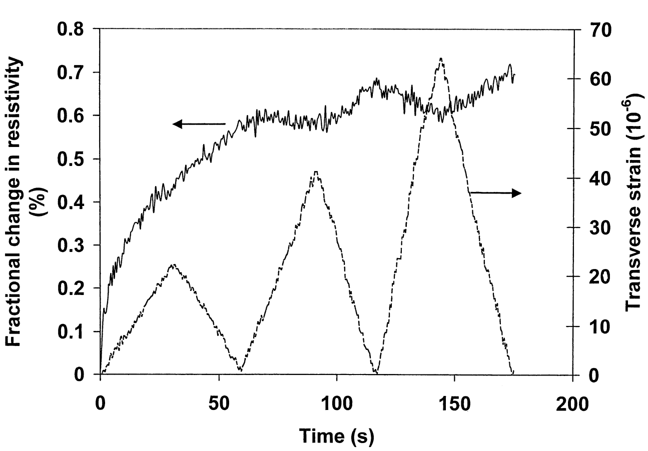

Fig. 3. Variation of the fractional change in transverse resistivity with time and of the transverse strain with time during dynamic compressive loading at increasing stress amplitudes within the elastic regime.

In spite of the Poisson effect, similar behavior was observed in the longitudinal and transverse resistivities. This means that the defects mentioned above are essentially nondirectional and that the resistivity variations are real.

Comparison of Figs. 2 and 3 shows that defect extension is more significantly revealed by the longitudinal resistivity than the transverse resistivity. Hence, the defects are not completely nondirectional.

Identification of the defect type is beyond the scope of this paper. Microcracks were mentioned above just for the sake of illustration. The defects may be associated with certain heterogeneities in the cement paste.

Defects affect the mechanical properties. Therefore, mechanical testing (such as modulus measurement, which is nondestructive) can be used for studying defect dynamics. However, the modulus is not as sensitive to defect dynamics as the electrical resistivity; the relationship between stress and strain is not affected while the resistivity is affected (Fig. 2). The low sensitivity of the modulus to defect dynamics is consistent with the fact that the deformation is elastic.

4. Conclusion

Defect dynamics, as studied by electrical resistivity measurement during repeated compressive loading of cement paste in the elastic regime, are characterized by (i) defect generation that dominates during the first loading, (ii) defect diminution that dominates during subsequent loading, and (iii) defect extension that dominates during still subsequent unloading. Defect extension during unloading follows defect generation or defect diminution during loading. Defect generation and extension cause the resistivity in both longitudinal and transverse directions to increase. Defect diminution causes the resistivity in both directions to decrease.

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