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Damage evolution during freeze–thaw cycling of cement mortar, studied by electrical resistivity measurement

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Abstract

Damage evolution during freeze–thaw cycling of cement mortar was found by electrical resistivity measurement to involve damage accumulating gradually cycle by cycle until failure. The damage inflicted during cooling was more significant than that inflicted during heating. Damage infliction occurred smoothly throughout cooling from 52 to − 20 °C. Failure occurred at the coldest point of a temperature cycle. Electrical resistivity measurement allowed simultaneous monitoring of temperature and damage. An increase in temperature caused the resistivity to decrease reversibly, but with hysteresis, which grew with cycling. The effects of freezing and thawing on the resistivity were small compared to the effect of temperature on the resistivity.
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1. Introduction

Freeze–thaw cycling is one of the main causes of degradation of concrete in cold regions. The degradation stems from the freezing of the water in the concrete upon cooling, and the thawing upon subsequent heating. The phase transition is accompanied by dimensional change and internal stress change. Freeze–thaw cycling can result in failure.

Previous work on the freeze–thaw durability of cement-based materials has been focused on the mechanical property degradation (e.g., modulus and strength) [1–3], weight change [1,3–5], length change [5,6], microstructural change [7] and ultrasonic signature change [5,8] after different amounts of freeze–thaw cycling. Relatively little attention has been previously given to monitoring during freeze–thaw cycling. Techniques previously used for real-time monitoring include strain measurement [6] and electrical resistivity measurement [9]. Without real-time monitoring, the degradation could not be monitored during freeze–thaw cycling. Therefore, study of the damage evolution required testing numerous specimens at different numbers of freeze–thaw cycles. As different specimens were bound to be a little different in the degree of perfection, the testing of different specimens gave data scatter, which made it difficult to study the damage evolution. In order to study the damage evolution on a single specimen during freeze–thaw cycling, a nondestructive and sensitive real-time testing method is necessary.

This paper uses electrical resistivity measurement as a nondestructive method. Although the electrical resistivity method has been previously used for monitoring the freezing process of concrete [9], it has not been previously used for monitoring damage during freeze–thaw cycling. On the other hand, this method has been previously used to monitor in real time the damage evolution during compressive stress cycling of cement mortar [10]; defect generation (irreversible) resulted in an irreversible increase in the baseline resistivity as stress cycling progressed.

The electrical resistivity of cement paste has been reported to decrease reversibly upon heating at temperature above 0 °C (without freezing or thawing), due to the existence of an activation energy for electrical conduction [11]. This phenomenon allows cement paste to function as a thermistor for sensing temperature. A similar phenomenon was observed in this work for cement mortar both above and below 0 °C. Thus, electrical resistivity measurement allows simultaneous monitoring of both temperature and

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damage. A temperature increase causes the resistivity to decrease reversibly, whereas damage causes the resistivity to increase irreversibly.

2. Experimental methods

The cement used was portland cement (Type I) from Lafarge (Southfield, MI). The sand used was natural sand (100% passing 2.36-mm sieve, 99.9% SiO_2). The sand/cement ratio was 1.0. The water/cement ratio was 0.35. A water-reducing agent (WR) was used in the amount of 1.0% by weight of cement. The WR was TAMOL SN (Rohm and Haas, Philadelphia, PA), which contained 93–96% sodium salt of a condensed naphthalene sulfonic acid. No coarse aggregate was used. A Hobart mixer with a flat beater was used for mixing, which was conducted for 5 min. After that, the mix was poured into oiled molds. A vibrator was used to facilitate compaction and decrease the amount of air bubbles. Specimens were demolded after 1 day and then allowed to cure in a moist chamber at a relative humidity of 100% for 28 days.

Specimens were rectangular bars of size 70 × 10 × 5 mm. The four-probe method was used for DC electrical resistivity measurement. The electrical contacts were silver paint in conjunction with copper wires, which were wound around the entire perimeter of a specimen at four planes that were perpendicular to the direction of resistance measurement (along the longest dimension of the specimen). The outer two contacts, 60 mm apart, each being 5 mm from an end of the specimen bar, were for passing current. The inner two contacts, 40 mm apart, each being 10 mm from a voltage contact, were for voltage measurement. A Keithley (Cleveland, OH) 2001 multimeter was used.

The temperature was cycled between − 20 and 52 °C in Experiment 1 in order to investigate the effect of freeze–thaw cycling and between 0 and 52 °C in Experiment 2 in order to investigate the effect of thermal cycling in the absence of freezing. In both Experiments 1 and 2, the time for each cycle was 40 min (Fig. 1). Heating was achieved by using a nichrome resistance heating wire wound around a specimen in the region between the voltage contacts. Cooling was achieved by using a freezer. The specimen was in the freezer throughout heating and cooling. The temperature during temperature cycling was measured by using a T-type thermocouple located near the center point of the rectangular specimen bar. Temperature cycling was conducted for 15 h in either experiment.

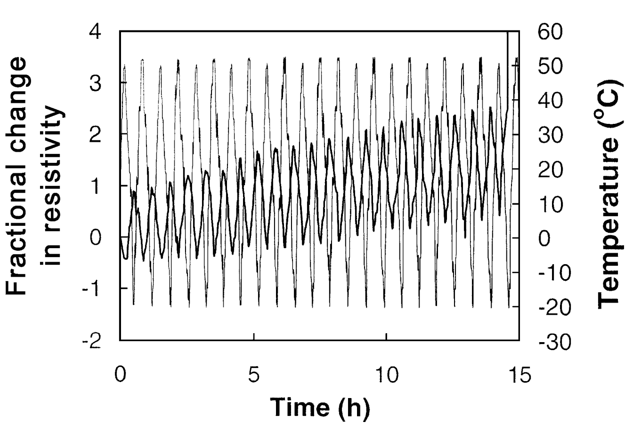

Fig. 1. The fractional change in resistivity vs. time (thick curve) and the temperature vs. time (thin curve) during fast freeze–thaw cycling.

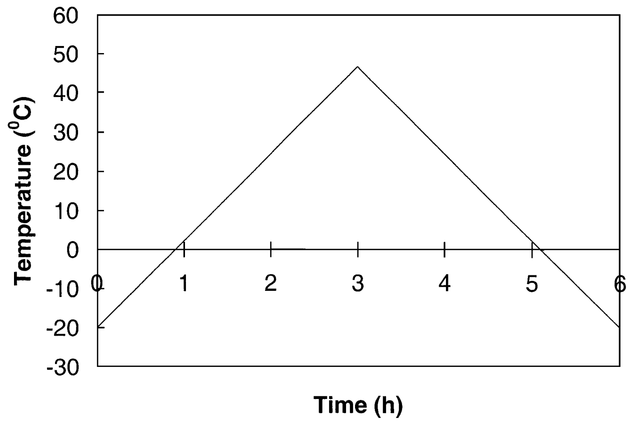

Fig. 2. The variation of temperature with time in a cycle of slow freeze–thaw cycling.

In Experiment 3, the temperature was cycled only once between − 20 and 46 °C, as shown in Fig. 2. Prior to cycling, the temperature had been equilibrated at − 20 °C. Note that the heating and cooling rates were much lower in Experiment 3 than Experiments 1 and 2. Heating and cooling in Experiment 3 were conducted by heating and cooling a stainless steel cylindrical box containing the specimen. Heating was achieved by using a resistance heater coil wound around the box in addition to a resistance hot plate under the box. Cooling was achieved by using liquid nitrogen, which flowed in a copper tubing wound around an outer stainless steel vessel containing the above-mentioned box and the hot plate below the box. The space between the inner box and the outer vessel was filled with a liquid to enhance heat transfer during cooling. The electrical leads emanating from the specimen for resistivity measurement came out of the box through the rubber lid of the box.

Due to the voltage present during electrical resistance measurement, electric polarization occurs as the resistance measurement is made continuously [12]. The polarization results in an increase in the measured resistance [3]. The polarization-induced resistance increase, as separately measured as a function of the time of resistance measurement in the absence of temperature cycling, was subtracted from the measured resistance change obtained during cyclic loading in order to correct for the effect of polarization.

The resistivity was obtained from the resistance and the dimensions. The change in dimensions during temperature variation was neglected. The fractional change in resistance was thus equal to the fractional change in resistivity.

Multiple specimens were tested in order to confirm the reproducibility of the behavior reported in this paper.

3. Results and discussion

3.1. Fast freeze–thaw cycling

Fig. 1 shows the fractional change in resistivity and the temperature during thermal cycling between − 20 and 52 °C (Experiment 1). The resistivity decreased upon heating and increased upon cooling in every cycle, due to the existence of an activation energy for electrical conduction. The resistivity changed smoothly and similarly above and below 0 °C, indicating that the phase transition did not affect the resistivity.

The resistivity at the end of a heating–cooling cycle was higher than that at the beginning of the cycle. In other words, the upper envelope of the resistivity variation (corresponding to the resistivity at − 20 °C) increased cycle by cycle. The lower envelope (corresponding to the resistivity at 52 °C) also increased cycle by cycle, but the increase was less significant than that of the upper envelope. As a consequence, the amplitude of resistivity variation increased with cycling. This behavior is attributed to damage, which caused the resistivity to increase irreversibly. That the upper envelope upshifted more than the lower envelope means that the damage occurs more significantly upon cooling than upon heating. This is expected since (i) thermal contraction occurs upon cooling and the surface of the specimen cools faster than the center of the specimen and (ii) water expands upon freezing.

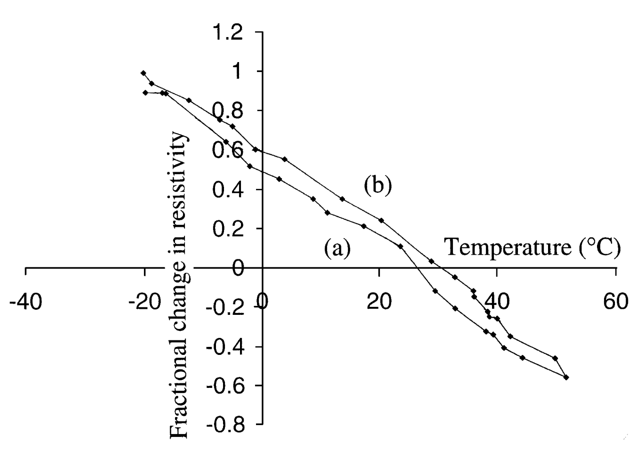

Fig. 3. The fractional change in resistivity vs. temperature during the first complete cycle in Fig. 1 of (a) heating from − 20 to 52 °C and then (b) cooling back to − 20 °C.

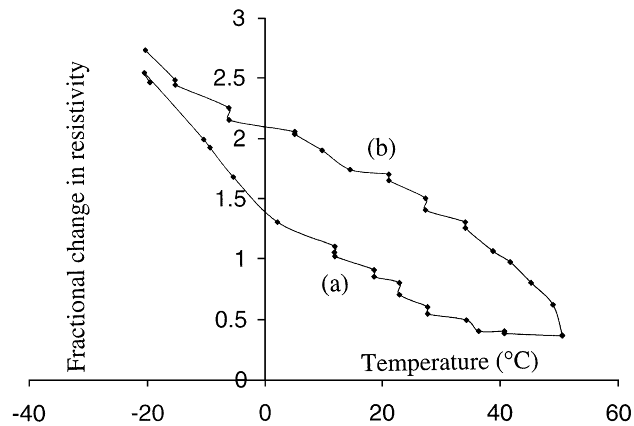

Fig. 4. The fractional change in resistivity vs. temperature during the last complete cycle in Fig. 1 of (a) heating from − 20 to 52 °C and then (b) cooling back to − 20 °C.

Upon freeze–thaw failure, the resistivity rose abruptly to essentially infinity, as observed before the completion of 15 h of cycling. This rise occurred at − 20 °C (the coldest point of a cycle), again indicating that damage during cooling was more significant than that during heating. Prior to failure, no abrupt resistivity increase was observed. This means that the damage evolution involved damage accumulating gradually cycle by cycle, until failure occurred.

The resistivity varied smoothly with temperature throughout the temperature range from 52 to − 20 °C (Fig. 1). This means that the freezing of water did not affect the resistivity, as expected from the fact that water contributed to ionic conduction and the electrical circuit used was an electronic circuit rather than an ionic circuit. At a given temperature, the resistivity during heating was slightly lower than that during subsequent cooling, as shown in Fig. 3 for the first complete cycle of heating from − 20 to 52 °C and then cooling back to − 20 °C in Fig. 1. This hysteresis had been previously reported [9]. Fig. 4 shows a plot similar to Fig. 3, but for

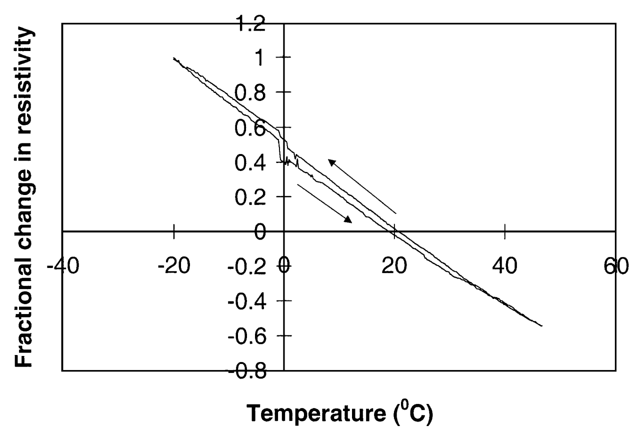

Fig. 5. The fractional change in resistivity vs. temperature in a cycle of slow freeze–thaw cycling (Fig. 2).

the last complete cycle in Fig. 1. The hysteresis became more severe as cycling progressed. The hysteresis is attributed to the damage inflicted during cooling and the association of damage with a higher resistivity. That damage infliction occurred smoothly throughout cooling from 52 to − 20 °C means that the damage was not due to freezing itself, but was due to thermal contraction and the fact that the surface cooled faster than the center of the specimen.

3.2. Slow freeze–thaw cycling

Fig. 5 shows results for one cycle of slow freeze–thaw cycling (Experiment 3). The behavior was similar to that for fast freeze–thaw cycling (Experiment 1) (Fig. 3), except that the resistivity decreased abruptly upon thawing at 0 °C and increased abruptly upon freezing at 0 °C and that the degree of hysteresis was less. Although the resistivity changed abruptly at 0 °C, the effects of freezing and thawing on the resistivity were small compared to the effect of temperature on the resistivity. It is reasonable that this small effect was only observed when the heating and cooling rates were low. That freezing caused the resistivity to increase and thawing caused the resistivity to decrease had been previously reported [9].

3.3. Thermal cycling without freezing

Fig. 6 shows the fractional change in resistivity and the temperature during thermal cycling between 0 and 52 °C (Experiment 2). The resistivity decreased reversibly upon heating, due to the existence of an activation energy for electrical conduction. In contrast to the case of freeze–thaw cycling at a similar cycling rate (Fig. 1), the lower envelope of resistivity variation did not shift upon cycling and the upper envelope upshifted only slightly. As the irreversible increase in resistivity is associated with damage, this means that the damage during thermal cycling without freezing is negligible compared to that during freeze–thaw cycling. As a result, failure was not observed after 15 h of thermal cycling without freezing (Experiment 2), but was visually observed before the end of 15 h of freeze–thaw cycling (Experiment 1).

As mentioned above, the damage in Fig. 1 was not due to freezing itself, but was due to thermal contraction and the fact that the surface cooled faster than the center of the specimen. Comparison between Fig. 1 (Experiment 1) and Fig. 6 (Experiment 2) shows that the damage caused by thermal contraction was significant in the presence of freezing, but negligible in the absence of freezing. In other words, freezing aggravated the damage that was due to thermal contraction.

The thermal damage observed in Fig. 1 is not related to damage that occurs at elevated temperatures (up to 52 °C). This is shown by a separate experiment in which the resistivity was monitored over time up to 4000 s at a constant temperature of 50 °C. The resistivity was observed

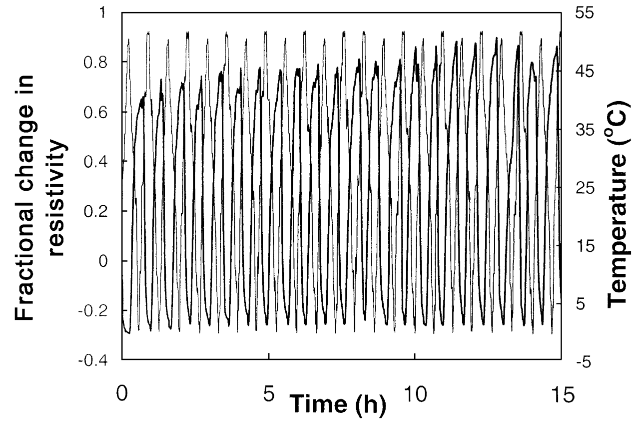

Fig. 6. The fractional change in resistivity vs. time (thick curve) and the temperature vs. time (thin curve) during temperature cycling without freezing.

to increase by less than 2%, in contrast to the much larger fractional increase in resistivity (whether the upper envelope or the lower envelope) in Fig. 1.

4. Conclusion

Damage evolution during freeze–thaw cycling of cement mortar involved damage accumulating gradually cycle by cycle, until failure occurred. The damage that occurred during cooling was more significant than that which occurred during heating. Damage infliction occurred smoothly throughout cooling from 52 to − 20 °C. Final failure occurred at the coldest point of a temperature cycle. The damage is attributed to thermal contraction during cooling rather than freezing itself, though freezing aggravated this damage. Without freezing, the damage during thermal cycling was negligible. The damage is not due to the elevated temperatures encountered during thermal cycling.

Electrical resistivity measurement allowed simultaneous monitoring of both temperature and damage in real time. An increase in temperature caused the resistivity to decrease reversibly, due to the activation energy of electrical conduction. The resistivity decrease exhibited hysteresis, which grew as cycling progressed. Damage caused the resistivity to increase irreversibly.

Freezing was accompanied by a resistivity increase, whereas thawing was accompanied by a resistivity decrease. However, the effects of freezing and thawing on the resistivity were small compared to the effect of temperature on the resistivity.

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