Feasibility of using near-field microwave reflectometry for monitoring autogenous crack healing in cementitious materials

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Iman Mehdipour [a], Reza Zoughi [b], Kamal H. Khayat [a, *]

[a] Center for Infrastructure Engineering Studies, Department of Civil, Architectural and Environmental Engineering, Missouri University of Science and Technology, Rolla, MO, USA
[b] Applied Microwave Nondestructive Testing Laboratory (amntl), Department of Electrical and Computer Engineering, Missouri University of Science and Technology, Rolla, MO, USA

A B S T R A C T

This study demonstrates the feasibility of using the near-field microwave reflectometry technique to *nondestructively* monitor the evolution of autogenous crack healing of mortar containing high volume of supplementary cementitious materials. Mortar samples were subjected to controlled compressive loading to generate cracks, and subsequently exposed to wetting/drying cycles to initiate the autogenous crack healing process. Test results indicate that cracked mortar samples exhibit higher point-to-point microwave reflection coefficient variations caused by cracking and moisture ingress (i.e., larger coefficient of variation (COV) values of magnitude of reflection coefficient, $|\Gamma|$, obtained from microwave reflectometry). When subjected to wetting/drying cycles, samples with higher crack healing capability are found to undergo less variation in microwave reflection coefficient. Based on the results for cracked samples, the COV trends obtained for microwave reflection properties can be divided into three parts as a function of wetting/drying cycles: part (I) corresponding to a significant point-to-point microwave reflection variations resulting from crack formation and moisture ingress after the first wetting/drying cycle; part (II) indicating the onset of the crack healing process identified by the reduction in the COV values; and part (III) representing slow-down of crack healing process for the regions exposed to microwave radiation as indicated by the relatively constant COV values during additional wetting/drying cycles. Such variations in microwave reflection properties can be linked to changes in moisture transport properties and subsequent crack healing process. To corroborate the microwave reflectometry results, concurrent ultrasonic measurements were conducted on the mortar samples during the self-healing process, and a good correlation was observed between the outcomes of these two test methods. The results of material characterization assessments including capillary water absorption, crack healing quantification, as well as X-ray diffraction and scanning electron microscopy of crack healing products were also used to quantify the crack healing evolution for the investigated mortar samples.

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1. Introduction

Concrete structures are inherently susceptible to microcracking. Over time, microcracking can lead to formation of larger cracks under structural and/or environmental factors. Cracking can adversely impact durability and structural integrity of a concrete structure, thus limiting service lifespan. However, given the autogenous healing capability of cementitious materials under certain conditions, cracks can heal due to additional interaction between cementitious materials, water, and the surrounding environment [1,2]. Autogenous crack healing in concrete is related to the physical and/or chemical composition of the cementitious matrix [3–5]. Autogenous crack healing process is primarily caused by: (i) the formation of calcium carbonate ($CaCO_3$) and calcium hydroxide ($Ca(OH)_2$) [2]; (ii) the sedimentation of particles and loose cement grains in the presence of water [3]; (iii) the continuation of the hydration of unhydrated cementitious materials and pozzolanic reaction associated with the use of supplementary cementitious materials (SCMs) [5–10]; and (iv) further swelling of the cement matrix [2]. Although ongoing hydration of unhydrated

cementitious materials and pozzolanic reaction due to the use of SCMs are thought to be the main contributing factors to crack healing at early-age, autogenous crack healing at later ages is mostly associated with the precipitation of calcium carbonate [11–14]. It is important to note that the presence of water is a primary component for the occurrence of autogenous crack healing, and hence, exposure conditions play important role in this process. Sisomphon et al. [15] reported that optimal recovery of mechanical properties caused by autogenous healing in cracked concrete can occur under cyclic wetting/drying exposure, while no recovery was observed for samples exposed to only air drying. Furthermore, autogenous crack healing is reported to occur mainly in narrow cracks [10,11,16–18]. Cracks with maximum widths of 200–300 μm are found to be fully closed due to the autogenous healing [11,16–20].

A number of destructive and nondestructive testing (NDT) techniques have been employed to evaluate and monitor autogenous self-healing capability in concrete. Tests involving resonant frequency [14,21–24], acoustic emission [25], electrical resistivity [26,27], X-ray computed tomography [28], ultrasonic pulse velocity (UPV) [18,29–34], and more recently diffuse ultrasound [35] and coda wave interferometry [36] have been employed as nondestructive means to evaluate the self-healing process in cementitious materials. Autogenous crack healing is inherently a slow process, in which small changes in material characteristics occur gradually. Therefore, NDT methods used for this purpose must be sufficiently sensitive to small changes in materials properties. It is important to develop a practical and robust NDT methodology for monitoring variations in materials and water transport properties (i.e., moisture ingress) associated with autogenous crack healing process.

Microwave nondestructive testing and evaluation (NDT&E) techniques have shown a good promise for evaluating and characterizing a diverse array of cement-based materials. This technique is completely nondestructive, one-sided (i.e., no need to have access to two or more sides of a sample), fast, easy-to-employ, and the obtained data can be correlated to the changes in materials [37]. As a microwave signal reflects from a dielectric material, its signal properties (magnitude and phase) change as a function of parameters, such as dielectric properties, volume contents, and level of homogeneity of material constituents [38]. The effective dielectric constant of a cement-based material is directly affected by its constituent dielectric constants and their volumetric content (i.e., paste, water-to-cement ratio, aggregate content, etc.) as well as any ongoing chemical reaction (i.e., cement hydration, variation in chemistry of pore solution, etc.) [38]. In the microwave NDT technique, the magnitude of the reflection coefficient, $|\Gamma|$, measured by an open-ended rectangular waveguide and conducted at different operating frequencies, has shown to correlate well with the aforementioned parameters. Some of the previous studies undertaken using microwave NDT technique include: (1) determination of water-to-cement ratio (w/c) [39–41] and compressive strength of cement paste and mortar [42]; (2) evaluation of sand-to-cement ratio in mortar; (3) detection of aggregate segregation [43]; (4) monitoring changes in material properties of concrete as hydration progresses (transformation of free water to chemically bound water) [39]; (5) evaluation of chloride ingress in mortar subjected to cyclic wetting/drying exposures [44]; (6) evaluation of crack formation for mortar samples subjected to cyclic compressive loading [44]; (7) detection of carbonation and alkali-silica reactions [45–47]; and (8) evaluation of steel fiber distribution in mortar [48,49].

The sensitivity of microwave signals to variations caused by water transport properties (i.e., presence of free water and transformation into bound water, capillary draw over wetting/drying cycles, etc.) and formation of cracks due to loading makes the microwave NDT technique an attractive approach for monitoring crack healing performance in cementitious materials. Therefore, the aim of this study is to demonstrate the feasibility of using near-field microwave reflectometry to nondestructively monitor autogenous crack healing in cement-based materials. Two sets of mortar samples with different binder compositions were prepared. After three days, one sample from each set was subjected to the controlled compressive loading corresponding to 90% of its 3-day compressive strength to generate cracks with varying widths. In order to initiate the autogenous crack healing process, cracked mortar samples were exposed to several wetting/drying cycles and their performance were compared with uncracked samples (reference samples). The statistical properties of microwave measurements (i.e., mean and coefficient of variation (COV)) of the magnitude of reflection coefficient ($|\Gamma|$) were determined to assess the degree of variations in microwave reflection properties. An independent set of experimental assessments, including ultrasonic transmission measurements, capillary water absorption, crack healing quantification using optical microscopy, as well as X-ray diffraction (XRD) and scanning electron microscopy (SEM) examinations were carried out to corroborate the results derived from microwave measurements.

2. Experimental approach

2.1. Materials and sample preparation

In this study, Type I/II ordinary Portland cement (OPC) conforming to ASTM C150 [50] was used. In order to examine the effect of SCMs on autogenous crack healing, Class C fly ash (subsequently labeled as FA) and blast-furnace slag (subsequently labeled as SL) were incorporated as a partial replacement for OPC. A continuously graded siliceous river sand with a maximum size of 5 mm, density of 2500 kg/m^3, and water absorption capacity of 0.6% was incorporated.

Two sets of mortar samples with dimensions of 200 × 200 × 200 mm^3 were prepared including: (i) plain OPC mortar (referred to as OPC); and (ii) mortar with 55% replacement of OPC with 20% SL and 35% FA, by mass of binder (referred to as OPC-SCM). The synergistic interactions between FA and SL have previously demonstrated good early- and later-age properties of cement-based materials due to the pozzolanic reaction [51–54]. For both mortar mixtures, the water-to-binder ratio (w/b) and sand-to-binder ratio were kept constant at 0.40 and 2.5, respectively. The slump consistency for the OPC and OPC-SCM mortar mixtures were 60 and 80 mm, respectively, in accordance with ASTM C143 [50].

2.2. Crack initiation and exposure condition

After demolding the 200 × 200 × 200 mm^3 mortar samples at 24 h, samples were moist cured for 3 days under wet burlap and plastic sheet. After 3 days, one sample from each set of mixtures was subjected to a compression load corresponding to 90% of its 3-day compressive strength (as identified by its corresponding 100 × 200 mm^2 cylindrical sample) to generate cracks. Given higher porosity of matrix and larger volume of unhydrated cementitious components at early-age, the pre-cracking at early-age can contribute to higher autogenous crack healing capability in cracked mortar samples made with high volume of SCMs compared to the similar samples that are cracked at later-age [54]. The widths of surface cracks were measured at 40 points along their lengths using an optical microscope. The measured crack widths varied from 10 to 500 μm. It is important to note that in terms of surface crack distributions and dimensions, the cracks