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Karl Joseph Bois

Aaron D. Benally

R. Zoughi
Missouri University of Science and Technology, zoughi@mst.edu

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Near-field microwave non-invasive determination of NaCl in mortar

K.J. Bois, A.D. Benally and R. Zoughi

Abstract: In recent years near-field microwave nondestructive testing and evaluation techniques have shown great promise for evaluating different properties of cement-based structures. An important issue regarding the inspection of these structures is the ability to determine the presence and evaluate the content of chloride in them. Chlorides can be introduced in these structures in many different ways including when salts are present in their mixing water. Consequently, for this investigation, two sets of 8" × 8" × 8" (203 mm) cubic mortar specimens were prepared each with a water-to-cement (w/c) ratio of 0.5 and 0.6, respectively, and both sets with sand-to-cement ratios of (s/c) of 1.5. Four specimens were produced for each set with different salt (NaCl) contents added to the mixing water, producing salt-to-cement (NaCl/c) ratios of 0.0, 1, 2 and 3%, respectively. For the purpose of compressive strength measurement, cylindrical specimens of these mortar specimens were also prepared and tested for their compressive strength. Microwave reflection properties of these specimens were measured at S-band (2.6–3.95 GHz) and X-band (8.2–12.4 GHz), employing open-ended rectangular waveguide probes. It is shown that the magnitude of reflection coefficient is a useful parameter for detecting different chloride levels in these specimens. Moreover, the influence of chloride on the curing properties (i.e. setting time) and the compressive strength of these specimens are subsequently shown to be well correlated to the measured magnitude of reflection coefficient of these specimens at S-band. It is also shown that this correlation is unambiguous as a function of w/c which possesses significant practical ramifications.

1 Introduction

The construction industry is continuously seeking novel techniques for *in situ* and nondestructive inspection of reinforced cement-based structures. Most such techniques are often limited in their use and may not always produce reliable results [1]. One important issue in the concrete industry is the nondestructive determination and evaluation of chloride in cement-based materials. Chloride is responsible for the corrosion of steel in reinforced concrete. Early detection and close monitoring of chloride penetration are required to ensure the integrity of these reinforced members.

Microwave nondestructive testing (NDT) techniques have been successfully used for interrogating nonconducting materials (i.e. dielectric materials) [2–10]. They can also be used to characterise properties of mixtures composed of several dielectric constituents (i.e. constituent dielectric properties and volume content), and to determine the cure-state and the presence of chemical reactions in these materials [9, 10]. In recent years, near-field microwave NDT techniques have been utilised for inspecting cement-based construction materials [11–22]. These techniques are divided into two main groups: near-field and far-field techniques. Although the modelling of the electric and magnetic fields is relatively more tedious in the near-field region, the practical implementation of near-field measurement techniques is quite simple. Furthermore, these techniques readily provide for material composition characterisation of cement-based materials. Thus far, several near-field microwave NDT techniques have been successfully used for inspection and characterisation of cement-based materials [12–22]. These studies have included:

- detection of rebar in reinforced concrete [12]
- determination of compressive strength and water-to-cement (w/c) ratio of hardened cement paste (cement and water) using two different approaches [13, 16, 17, 21]
- prediction of the microwave reflection properties of mortar (cement, sand, water and porosity) using a dielectric mixing model as a tool for obtaining the volume fraction of individual constituents of mortar [14]
- determination of sand-to-cement (s/c) ratio in mortar using the stochastic properties of its microwave reflection properties [15]
- determination of cure state in concrete [16]
- determination of w/c in cured and fresh concrete [16, 22]
- detection of aggregate segregation in concrete [19]
- determination of coarse aggregate volumetric distribution in concrete [17, 18]
- detection of grout in masonry bricks [20].

In this paper, the ability of these techniques to detect salt content added to the mixing water of mortar, as well as the influence of this additional salt to the compressive strength

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K.J. Bois was with Colorado State University and is now with Hewlett-Packard Company, Ft. Collins, CO 80528, USA

A.D. Benally and R. Zoughi are with the Applied Microwave Nondestructive Testing Laboratory (*amntl*), Electrical and Computer Engineering Department, Colorado State University, Ft. Collins, CO 80523, USA

of mortar and its correlation to microwave reflection property measurements are addressed.

Concrete normally provides reinforcing steel with adequate corrosion protection. When steel is encased in concrete, a protective iron oxide film forms at the steel–concrete inteiface due to the high pH level associated with concrete. This film protects the steel from corrosion. However, the intrusion of chloride ions in reinforced concrete can destroy this protective film. Moreover, if moisture and oxygen are present in the concrete, the steel will corrode through an electrochemical process. Once the steel begins to corrode, the concrete will deteriorate, since the byproducts of corrosion occupy a greater volume than the steel itself and subsequently exert a substantial stress on the surrounding concrete.

Chloride can be introduced into concrete in many ways. It may be introduced into the concrete mix by the aggregates, cement, admixtures and/or the mixing water. Moreover, chloride may enter into a concrete structure, while in use, through exposure to deicing salts, seawater or salt air environment. Therefore, it is important to be able to measure the chloride content of concrete in order to indicate the likelihood of corrosion of its embedded reinforcing steel bars.

The limit placed on the amount of chloride content in concrete is a function of the type of structure and the environment to which it is exposed during usage. Limits on chloride content in reinforced concrete are set in two ways: water-soluble chloride ion content and the total chloride ion content. The two values are not substantially different from one another because the water-soluble chlorides are only a part of the total chloride content, namely the free chlorides in the pore water [23].

Steel in concrete begins to corrode when the water soluble chloride content in the concrete is about 0.15% of the cement weight [24]. Of the total chloride ion content in concrete, only about 50 to 80% is water soluble, the rest becoming chemically bound in the chemical process involving cement [25]. Limits on the water-soluble chloride content in concrete have been set by the American Concrete Institute [26]. The water-soluble chloride ion content of hardened concrete may be determined by a procedure similar to that reported in a Federal Highway Administration (FHWA) report [27]. This procedure is destructive in nature, and is performed in a laboratory on concrete that has hardened for 28 to 42 days.

The standard approaches for determining chloride content in concrete (i.e. the total amount of chlorides) are the procedures outlined by the American Society for Testing and Materials (ASTM) and the American Association of State Highway and Transportation Officials (AASHTO) [28, 29]. All of the methods mentioned for determining chloride content in concrete require obtaining a sample of the hardened concrete. Subsequently, it is ground up and tested following the procedures outlined in [27–29]. These methods present several distinct disadvantages such as being destructive, time consuming and prohibitive to the large-scale testing of structures. Moreover, these structures cannot be tested again at the same location for determining the progression of chloride penetration. Consequently, non-destructive solutions are highly advantageous for chloride detection.

As mentioned earlier, microwave NDT techniques have already demonstrated the ability to evaluate various important properties of cement-based structures. This success is mainly due to the inherent sensitivity of microwaves to the presence of bound or free water in these materials [16]. In the case where the chloride content in a cement-based specimen is above a certain limit, the chloride will interact with any free water and also become bound with the cement which is expected to affect the dielectric properties of the specimen. It has already been shown that at relatively low microwave frequencies, an increase in the salinity of water significantly increases its microwave dielectric properties [30].

The influence of chlorides on the curing process of cement-based materials, using established civil engineering standards, has been well documented [31–33]. However, a consensus opinion as to the maximum allowable chloride content in a mixture or its specific effect on curing has yet to be reached. It has been determined that, if the chloride content in the mixing water (for concrete) does not exceed 500 ppm, the water is considered harmless to the concrete [31]. It has also been shown that water with high salt content (e.g. seawater) has been used satisfactorily in producing concrete [32]. Typical seawater has a total salinity of about 3.5%, 78% which is NaCl and 15% of $MgCl_2$ and $MgSO_4$. It has been found that using seawater in concrete produces a slightly higher early strength but a lower long-term strength, usually by less than 15% [33]. Also, some tests have indicated that seawater accelerates the setting time of cement to its respective final cure state. However, others have shown a substantial reduction in the initial setting time, but not necessarily in the curing time required to reach its final cure state [33].

In this paper the influence of adding salt to the mixing water of several mortar specimens is investigated, as it relates to their microwave reflection properties. Moreover, the influence of this salt addition to the setting time of these specimens as well on their compressive strength is studied. The results are shown to correlate well with the microwave reflection properties of these specimens.

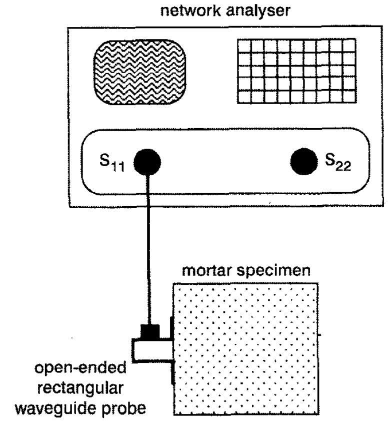

Fig. 1 *Microwave measurement apparatus*

2 Approach

To determine and correlate the influence of chloride content in the mixing water of cement-based materials to their microwave reflection properties, two sets of four 8" × 8" × 8" (203 mm) mortar cubic specimens, were produced. The side dimension of 8" guarantees that the specimen is an infinite half-space and there is no reflection from the opposite face of the specimen. Each set was produced with w/c ratios of 0.50 and 0.60, respectively, and both with an s/c of 1.5. Varying levels of table salt were dissolved in the mixing water of these specimens, resulting in salt-to-cement (NaCl/c) ratios of 0.0, 1, 2 and 3%, respectively. The specimens were moist cured in a hydration room for one day and air cured (in ambient temperature and low humidity) for the remaining 28-day curing period. Subsequently, the reflection properties of these specimens were measured using open-ended rectangular waveguide probes at S-band (2.6–3.95 GHz) and X-band (8.2–12.4 GHz), utilising an HP8510 vector network analyser, as shown in Fig. 1. Each measurement reported here is the calculated average of 20 measurements performed on all sides of the specimens (omitting their top and bottoms). To correlate the microwave reflection properties of these specimens to their compressive strength, for each mortar specimen four identical cylindrical samples of 4" in diameter and 8" long were also produced and cured in the same manner as the cubic specimens. Once cured, the compressive strength of each mortar specimen was determined by crushing these cylinders using a 300-kip testing machine. The average measured compressive strength (i.e. obtained from the four samples of each mortar) results were then correlated to the cured reflection properties of the mortar specimens as a function of w/c and NaCl/c.

3 Results

Fig. 2 shows the daily mean and standard deviation of the measured magnitude of reflection coefficient, $|\Gamma|$, at a frequency of 3 GHz (S-band) over the curing period for the four specimens with 0.5 w/c. and varying levels of NaCl/c. The error bars indicate one standard deviation (above and below) of the measurements. As expected from previous studies [13, 15, 16], the behaviour of $|\Gamma|$ for all specimens decays rapidly during the first few days and then slows down considerably past day 12. This is due to the fact that during the first few days each specimen contains a significant level of free water. Since the dielectric properties (relative permittivity and loss factor) of free water are relatively high compared to that of cement powder, the resulting $|\Gamma|$ is also expected to be relatively large. As the curing process progresses water molecules bind with those of cement, transforming from free water to bound. In addition, some of the free water is lost to evaporation as time progresses. This evaporation is more significant as a function of increasing w/c. Consequently, the measured $|\Gamma|$ for each specimen decreases as a function of curing time, as shown in Fig. 2.

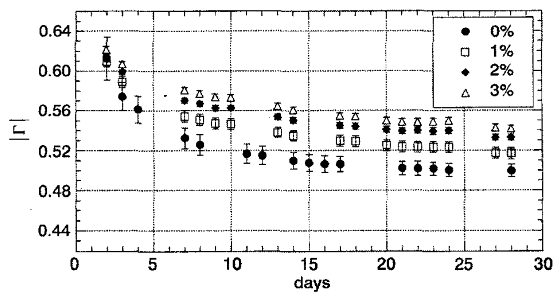

Fig. 2 *Change in measured $|\Gamma|$ as a function of curing time for mortar specimens with w/c of 0.50 and varying NaCl/c at 3 GHz*

First and foremost, comparing the results for different NaCl/c specimens indicates that the presence of chloride in the mixing water can be unambiguously detected in particular after the sixth day when a substantial amount of free water has disappeared. The results also indicate that the difference between the measured $|\Gamma|$ for the first and the final days of measurements (i.e. the 28th day of curing) is progressively smaller as a function of increasing NaCl/c. Thus, based on the results of previous studies, it may be expected that the specimens with increasing salt content may not have undergone a complete curing process. Since all specimens contained identical proportions of water at the time of mixing, one can hypothesise that the presence of salt in these specimens changes the curing process and possibly affects their long-term compressive strength. This is based on the fact that in previous investigations specimens with higher final $|\Gamma|$ have been shown to have higher compressive strengths [13, 18]. The relationship between NaCl/c and $|\Gamma|$ for all specimens will be shown later.

Fig. 3 shows the daily mean and standard deviation of the measured $|\Gamma|$ as a function of time for the same four specimens but at a frequency of 10 GHz (X-band). Although the same overall behaviour is observed as that at 3 GHz, there is not much distinction among the different specimens as a function of NaCl/c. The difference in the dielectric properties of water containing increasing levels of salt is more substantial at 3 GHz than at 10 GHz [30]. Hence, the measured $|\Gamma|$ which is directly related to the dielectric properties of water as a function of salinity is not expected to produce substantial variation in the measured $|\Gamma|$ either [18].

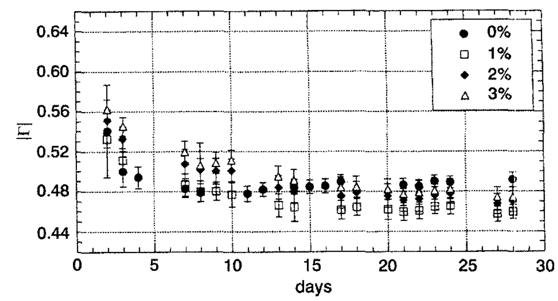

Fig. 3 *Change in measured $|\Gamma|$ as a function of curing time for mortar specimens with w/c of 0.50 and varying NaCl/c at 10 GHz*

Fig. 4 shows similar results for the specimens with 0.6 w/c at 3 GHz (S-band). The results regarding the relative differences in the measured mean $|\Gamma|$ as a function of time for these specimens are quite similar to those obtained for the 0.5 w/c specimens. However, the dynamic range of the measured $|\Gamma|$ is larger for these specimens. This is expected since these specimens contain a larger fraction of free water at the beginning [13, 16, 18]. In addition, as they cure there is less cement for the water to combine with. Therefore, more of the free water is lost to evaporation during the curing process, resulting in more porosity. Thus, in the final days the measured $|\Gamma|$ is lower than those for the 0.5 w/c specimens. Similar to the results shown in Fig. 3, but not presented for brevity, at 10 GHz these specimens were not readily distinguishable from one another.

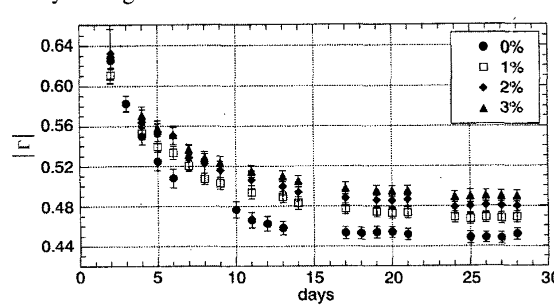

Fig. 4 *Change in measured $|\Gamma|$ as a function of curing time for mortar specimens with w/c of 0.60 and varying NaCl/c at 3 GHz*

To demonstrate the capability of this microwave nondestructive testing technique for detecting chloride content in the mixing water of these specimens, the 28th day measured |Γ| at 3GHz as a function of NaCl/c for both set of specimens is shown in Fig. 5. The results clearly show, as stated earlier, that the measured |Γ| (discrete points in Fig. 5) can be used to detect and estimate the level of chloride content in these specimens. The solid lines are linear fits through the measurement points indicating a linear correlation between the measured |Γ| and NaCl/c for chloride content evaluation purposes. In addition, as expected the specimen with 0.5 w/c produced higher |Γ| for all NaCl/c [13, 16]. It is also expected that these specimens have higher compressive strength than those with 0.6 w/c [31].

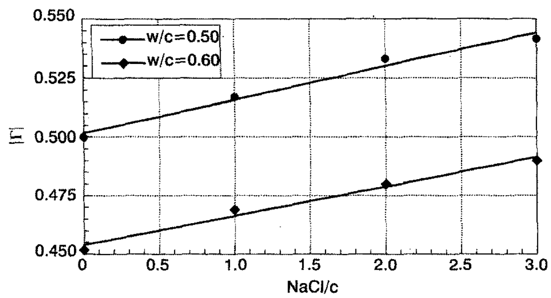

Fig. 5 *Measured |Γ| as a function of NaCl/c for all specimens at 3GHz*

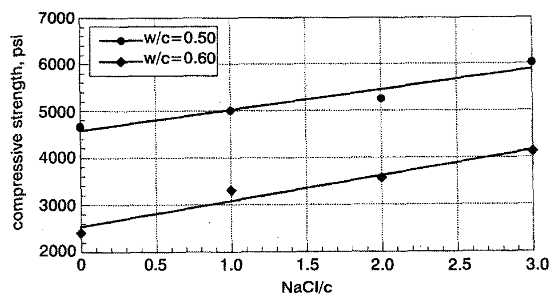

Fig. 6 *Measured compressive strength as a function NaCl/c for all specimens*

To determine the influence of chloride content on the compressive strength of these specimens and show a correlation to the measured |Γ|, the cylindrical specimens were tested for their compressive strength using a 300-kip cylinder-testing machine. Fig. 6 shows the average measured compressive strength for all specimens (discrete points in Fig. 6). The results show that the specimens with lower w/c consistently have higher compressive strength, as expected [13, 16, 18, 31]. Moreover, compressive strength is shown to increase as a function of increasing NaCl/c, as hypothesised earlier. Finally, the solid lines in Fig. 6, which show a linear fit through the measured points, follow a similar increasing linear trend in the compressive strength as a function of NaCl/c as did the measured |Γ| results shown in Fig. 5. This fact indicates that a simple and linear correlation exists between the measured |Γ| and the compressive strength of these 28 day cured specimens. This correlation, for each of the 0.5 and 0.6 w/c specimens, is shown in Figs. 7 and 8, respectively. The discrete points are the measured values, while the solid lines indicate a linear fit through these points. Clearly, there exists a linear correlation between the measured |Γ| and the compressive strength of these specimens as a function of w/c and NaCl/c. Fig. 9 shows the combined results of Figs. 7 and 8. The results clearly show the increase in the compressive strength and the measured |Γ| as a function of w/c and NaCl/c. More importantly, this Figure shows that there is no ambiguity in evaluating both the w/c and NaCl/c for these eight mortar specimens at 3GHz (S-band). This frequency has also been shown to be optimal for determining w/c in hardened cement paste, mortar and concrete [13, 15, 16, 18].

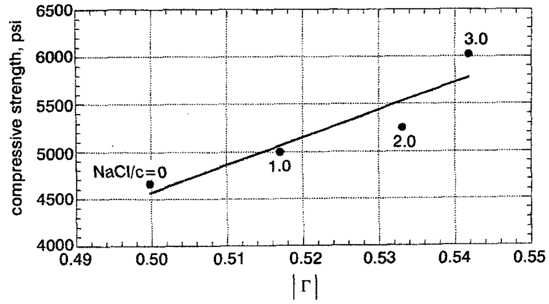

Fig. 7 *Relationship between measured compressive strength and |Γ| for specimens with w/c of 0.50 and varying NaCl/c at 3GHz*

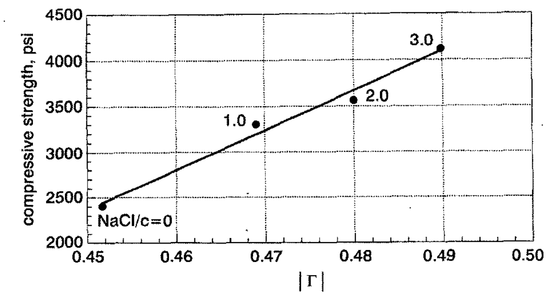

Fig. 8 *Relationship between measured compressive strength and |Γ| for specimens with w/c of 0.60 and varying NaCl/c at 3GHz*

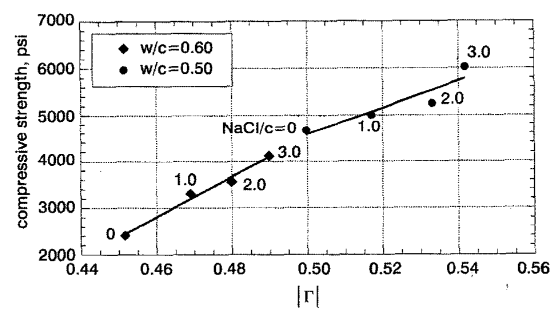

Fig. 9 *Relationship between measured compressive strength and |Γ| for all specimens as a function of w/c and NaCl/c at 3GHz*

4 Conclusion

In this paper, microwave detection and content evaluation of chloride, introduced as the addition of salt to the mixing water of several mortar specimens with w/c of 0.50 and 0.60 and all with s/c of 1.5 and varying NaCl/c, was shown to be possible. The microwave technique used here employed open-ended rectangular waveguide probes at S-band (2.6–3.95GHz) and X-band (8.2–12.4GHz) in conjunction with a vector network analyser suitable for calibrated reflection coefficient measurement. This detection and evaluation was made possible through the influence of chloride in the measured magnitude of reflection coefficient at relatively low microwave frequencies (e.g. 3GHz). At 10GHz, it was not possible to distinguish among these specimens. This is also supported by the fact that the dielectric properties of free or bound water present in a specimen, as a function of varying salinity, are more significantly influenced at lower frequency bands. The results also showed the reduction in setting time of these specimens as a function of increasing NaCl/c. The results of earlier investigations had already established that when conducting these measurements, the magnitude of reflection coefficient increases as a function of decreasing w/c. This was shown to be true once again for these specimens in addition to the fact that this parameter was also shown to increase as a function of increasing NaCl/c, indicating an increase in their compressive strength as a function of this parameter. To verify this, cylindrical samples of these mortar specimens were also prepared and tested for their compressive strength. It was subsequently shown that the measured compressive strength of these specimens also increased as a function of NaCl/c. Subsequently, a simple and linear correlation between the measured magnitude of reflection coefficient of these specimens at 3 GHz and their compressive strength was shown to exist as a function of w/c and NaCl/c. The results of this investigation are very encouraging for detecting and evaluating chloride content, as a result of salt added to the mixing water of mortar. The next step in this study should involve detecting and evaluating chloride penetration in cement-based materials through successive introduction of these materials to salt water. This investigation is currently ongoing. It must be noted that, if and when such a technique becomes available for on-site testing, one must be cognisant of the fact that any excess free water that exists on the surface or has permeated through the concrete will influence the measurement results. In such cases one must either make the measurements a few days after an episode of rain or use a complementary technique to measure the free water level and calibrate the system accordingly.

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