1-1-2004

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Microwave Reflection Properties of Concrete Periodically Exposed to Chloride Solution of 3% Salinity and Compression Force

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Abstract—Corrosion of steel rebar in a concrete structure compromises its structural integrity and hence its performance. Chloride intrusion into concrete can lead to depassivation of the steel and initiation of corrosion. Methods exist to detect chlorides in concrete, but the practical use of many of these may be problematic because they are destructive and time consuming, and cannot be used to analyze large structures. Microwave nondestructive evaluation techniques applied to mortar have proven successful for detecting mixture constituents, chloride ingress, and cure-state monitoring. In this paper several concrete samples are cyclically soaked in distilled water and saltwater while also experiencing compression force. Compression force, simulating in-service loading, results in increased microcracking and permeability, which promotes chloride ingress. The daily microwave reflection properties of these samples were measured at 3 GHz. The results show the capability of these microwave measurements for detecting the increased level of chloride permeation as a function of increasing number of soaking cycles. In addition, comparisons between the reflection properties of mortar and concrete cubes soaked in distilled water exhibit similarity in trends, indicating that the various phenomena that occur within them are systematically similar.

Index Terms—Chloride, concrete, microwaves, nondestructive testing, reinforcing steel.

I. INTRODUCTION

CORROSION of steel compromises the strength of reinforced concrete structures, and the presence of chlorides is known to be the leading cause of such damage [1]. In practice, many cement-based structures are usually subjected to both loading and exposure to chloride solution in a cyclical fashion. Under sufficient loading, there is then the possibility that the preexisting microcracks can grow, thus forming new and longer cracks. This results in higher permeability and hence increased chloride penetration in the structure. Thus, the development of a reliable and nondestructive as well as a less time-consuming test method, for in-situ measurement of chloride penetration in in-service concrete, is of great interest. From an economic standpoint, accurate measurement of chloride concentration and penetration depth in concrete structures would allow agencies to more effectively allocate funding for repair of those structures with the most critical needs.

In recent years, microwave nondestructive testing (NDT) techniques have been successfully utilized for evaluating various properties of cement-based materials [2]–[7]. Although in-service structures are usually made of concrete, previous investigations primarily focused on the influence of chloride ingress (including compression force) on the microwave reflection properties of mortar cubes [6], [7]. This was done since mortar is the basic building block of concrete and is a relatively simpler medium in comparison to concrete. The complexity of concrete primarily arises from the presence of coarse aggregates. Therefore, the information obtained from studying mortar serves as a prelude to understanding the behavior of concrete. The objective of this investigation is then to determine whether similar microwave NDT techniques are capable of detecting moisture and chloride ingress in several specially prepared concrete cubes.

II. APPROACH

Four 8 × 8 × 8-in concrete cubes were prepared with water-to-cement ratio (w/c) of 0.71, sand-to-cement ratio (s/c) of 2.96, and coarse aggregate-to-cement ratio (ca/c) of 3.65 using portland cement Type I/II. These mixture parameters were determined for a sample of relatively low strength, 3000 psi, and high permeability [8]. All of the cubes were allowed to cure for one day in a hydration room and subsequently were left in ambient conditions for another three months. Microwave reflection properties of these cubes were then measured at S-band (2.6–3.95 GHz) using an open-ended rectangular waveguide probe in conjunction with a calibrated HP8510C vector network analyzer for four soaking cycles. Each cycle included soaking two cubes in a sodium chloride solution (using ACS-certified NaCl) having a salinity of 3% and one in distilled water for 72 h in three individually covered containers. Each sample was placed on small spacers, at its corners, to allow unimpeded moisture ingress into all sides of the cube, excluding the top. The top 1 cm of the cube was not submerged.

The fourth cube was kept in ambient conditions and was never exposed to any solution or loading to serve as a reference and provide information about the influence of ambient conditions (i.e., variations of relative humidity and temperature), if any. After soaking, the microwave reflection properties of all of the cubes were measured immediately and for the subsequent 63 days, as they dried in the ambient conditions. This process
constituted one cycle. During the cycle, the mass of the cubes and the ambient conditions were monitored daily as well. Remaining cycles differed from the first in that one of the saltwater-soaked cubes was loaded to 90 000 lb (1400 psi) of compressive force. This was done by utilizing a 120 000-lb capacity Tinius and Olson testing machine to load the cube at a rate of 300 lb/s (136 kg/s) and unload at the same rate. To obtain an average value of the reflection properties, 16 measurements were performed on the cube (four per side). The results reported in the following sections are those at a frequency of 3 GHz, representing typical results at S-band.

III. Results

Fig. 1 shows the magnitude of reflection coefficient, hereon referred to as \( |\Gamma| \), for the four concrete cubes and for the four cycles. From the measurements at day zero (the day prior to soaking), it is evident that all of the cubes possessed similar reflection properties, as expected, since they were all made from the same batch of concrete mix. However, once the cubes are soaked, their reflection properties are expected to change as a result of absorbing water or saltwater. This aspect is also confirmed by the fact that from day zero to day one, there is a significant increase in \( |\Gamma| \) of the soaked cubes in comparison to that of the reference cube. In the context of this investigation, day one corresponds to the measurement conducted immediately after removing the cubes from their respective baths. Therefore, the measurements for day one are influenced both by the presence of free water/saltwater on the surface as well as that which permeated into the respective cubes. By the time day two measurements were conducted it was observed that there were no visible traces of free water/saltwater on the surface of the cubes. Thus, any further variation in \( |\Gamma| \) from day two onwards is primarily attributed to the variations in the temporal water/saltwater content within the respective cubes.

Once the cubes are taken out of the distilled water or saltwater solutions, their daily measured reflection properties progressively change, indicating the effect of evaporation of free liquid water from, and the capillary draw of water or saltwater toward, the core of the cube [6], [7]. This fact is also evident from the progressive decrease of \( |\Gamma| \) as a function of days. Toward the final days of the cycle, it is observed that the \( |\Gamma| \) for all the cubes converge toward the reference cube. Based on this information, it can then be inferred that much of the moisture has evaporated from the cubes, to the extent that they can now be considered relatively dry. However, the inspection of phase of reflection properties suggests that this might not be the case, as will be shown later. One may also see that the rate of decline of \( |\Gamma| \) increases as a function of cycles. To fully understand its significance, it is important to consider the issue of effective penetration depth of the microwave signal. This aspect will be revisited during the comparison of mortar and concrete.

Fig. 2 shows the phase of reflection properties, hereon referred to as \( \varphi \), for the four cubes and for the four cycles. The initial presence of water/saltwater within the respective cubes influences the reflection properties in such a way that the magnitude of reflection properties increases while the phase of reflection properties decreases [7]. The results indicate that from day zero to day one there is a significant decrease in \( \varphi \) for all three cubes indicating the presence of water/saltwater within the cubes. For the remaining days of each cycle there is a gradual increase in \( \varphi \) primarily signifying the process of evaporation. The important point to be noted is that at the end of a cycle there is still a difference in \( \varphi \) between the reference cube and the cubes that were soaked. This indicates that the cubes did not completely dry out and that there was still some amount of residual water/saltwater left behind in the cubes, which is contrary to what is suggested by the magnitude of reflection coefficient. To gain an insight into the reasons causing this phenomenon, it becomes necessary to conceptualize the drying cycle as it pertains to the reflection property measurements at 3 GHz. Previous investigations have shown that such contrasting behavior in \( |\Gamma| \) and \( \varphi \) could be as a result of: a) the distribution gradient of water/saltwater that develops within the cubes as the drying cycle progresses and b) the quantity of water/saltwater that exists up to the depth to which the microwave signal penetrates into the concrete cube [7].

Although the magnitude of reflection coefficient for all of the cubes presents similar characteristics in both trend and value, the phase of reflection coefficient indicates that there is a noticeable difference among the cubes. It is observed that the saltwater-soaked cubes have a lower \( \varphi \) than the distilled water-soaked cube. This is primarily due to the fact that the dielectric properties of saltwater having a salinity of 3% possess much greater loss-factor than distilled water [9]. Hence, the saltwater-soaked cubes have different \( \varphi \) characteristics than the distilled water-soaked cube. This difference in \( \varphi \) is also partially attributed to the hygroscopic properties of salt, which reduces the rate at which moisture evaporates from the saltwater-soaked cubes. Hence, saltwater-soaked cubes will have a lower \( \varphi \) as compared to the distilled water-soaked cube as the drying cycle progresses. During the first few days of a cycle, this difference is more a function of the difference between the ionic concentration of the pore solutions (i.e., water versus saltwater) in the respective samples [7]. As the cycle nears its end, the remaining difference may be attributed to a) the salt crystals that precipitate out from the salt solution for the saltwater-soaked cubes and b) the increase in salinity level in the residual solutions left behind in the pores of the respective cubes (i.e., as moisture evaporates from the saltwater-soaked cube, the salinity of the remaining solution within the cube increases).

As mentioned earlier, at the end of each drying cycle, one of the saltwater-soaked cubes was loaded to 90 000 lb, which
corresponds to approximately 40% of the final compressive strength of these cubes. Compressive loading produces microcracks, and it is expected that these microcracks allow more solution to permeate into the cube during the soaking period. As more saltwater is contained within the cube, it is also expected that its microwave reflection properties will be different than the cube that was not loaded. This aspect has been verified in previous investigations carried out on mortar cubes, where it has been shown that the effect of loading is clearly evident from the magnitude of reflection properties [6]. However, the results shown here for the concrete cubes do not indicate any clear difference between the reflection properties of the cube that was loaded and that which was not loaded, as a function of cycles. This could be due to the fact that the compressive stress that was applied to the cube was insufficient to generate significant microcracking.

To verify this aspect one may also need to monitor the daily mass of the cubes. Fig. 3 shows the temporal additional mass of solution present within the cubes per unit volume for all the three cubes. The results indicate that there is no distinct mass difference between the saltwater cubes that were loaded and not loaded, verifying the fact that the effect of loading did not cause significant microcracking leading to increase in permeation of saltwater into the cube during the soaking period. The results also indicate that the rate of decrease of mass for the saltwater-soaked cubes is slower than that for the distilled water-soaked cube. This is attributed to the fact that the hygroscopic properties of salt prevent the water from evaporating as quickly as the water-soaked cube, as mentioned earlier. It is also observed that the mass of the cubes for the first and last days of the cycle progressively increases as a function of the number of cycles. This is the result of the fact that there is some amount of residual water/saltwater left behind in the respective cubes at the end of a cycle (as was suggested by the phase of reflection coefficient), and this mass contributes to the total mass of the next soaking cycle. It is also expected that this residual solution is mostly toward the core of the cube (due to capillary draw). Additionally, the results also show that there is a gradual increase in separation in the additional mass curves between the water and saltwater soaked cubes as a function of cycles. This could be as a result of the increasing amount of residual salt that is left behind in the cube as water evaporates from the saltwater-soaked cubes. This aspect is consistent with the phase of reflection coefficient results. The information provided by the additional mass could then potentially serve as a complimentary piece of information to the microwave measurements in that a one-to-one correspondence may be established between the reflection properties and the amount of solution present in the cubes. This relationship can then be used as a guiding factor for the development of a semiempirical model that could simulate the reflection properties of the cubes [7]. The outcome of such a model would then describe the temporal distribution of water/saltwater content within the respective cubes as a function of distance into the cubes for each day of the cycle [7], [10].

IV. COMPARISON WITH MORTAR

Previous investigations similar to the ones discussed here were carried out on mortar cubes. The results of these investigations showed the capability of microwave measurements for detecting the effect of chloride permeation and loading as a function of increasing number of soaking cycles [6]. A comparison between the reflection properties of mortar and concrete cubes may then enable us to evaluate variations in the various phenomena such as absorption, diffusion, dispersion, permeation, etc., that occur within the two cubes. The mortar cube used here for comparison was made using portland cement Type I/II with a w/c of 0.5 and s/c of 2.5. The compressive strength of the cube was found to be approximately 2900 psi. Both the mortar and the concrete cubes were soaked in distilled water solution for 72 h, after which microwave measurements were carried out on a daily basis.
had lower moisture content for the first day of the cycle. As mentioned earlier, the first day measurements corresponded to immediately after taking the cubes out of their respective soaking solutions and are therefore influenced (to a greater extent) by the excessive free water on the surface, and therefore do not provide an accurate indication of the moisture content present within the cubes. Thus, the day one reflection property measurements should be considered with this fact in mind. From day two onwards, it is observed that $|\Gamma|$ for the concrete cube is lower than that of the mortar cube while $\varphi$ for the concrete cube is higher than that of the mortar cube. Both these trends are expected and indicate lower moisture content within the concrete cube, which is in accordance with the additional mass results. Additionally, the rate of decrease of $|\Gamma|$ and the rate of increase of $\varphi$ as a function of days is higher for the concrete cube indicating that the moisture evaporates from the concrete cube at a faster rate. An important point to note is that during the first few days of the cycle, the mortar cube exhibits a flattened shape for the phase of reflection coefficient. It has been shown that this flattened shape is due to the sensitivity of the microwave signal to the movement of water/saltwater within the cube [7], [10]. However, the fact that this particular behavior does not manifest itself for the concrete cube suggests that the soaking time (72 h) did not allow for sufficient permeation of water/saltwater into the concrete cubes. Additionally, the higher permeability of mortar in concrete allows for the easy permeation of water/saltwater toward the inner regions of the cube. Consequently, there will be lesser water/saltwater content in regions near the surface for the concrete cube. As a result of all these factors, the microwave signal is influenced to a lesser extent by the water/saltwater content as well as its movement (as a function of days) within the concrete cube as compared to mortar. This aspect becomes more pronounced as a function of cycles and also explains why the magnitude of reflection properties (Fig. 1) for the soaked concrete cubes decrease at a faster rate, thereby catching up with the reference cube within a shorter period of time for increasing number of cycles.

V. Conclusion

Nondestructive detection and evaluation of sodium chloride ingress in cement-based materials and structures are important issues in the construction industry. This investigation explored the possibility of employing microwave nondestructive techniques to evaluate the effects of cyclical chloride ingress and compressive stress on concrete cubes. For this purpose several concrete cubes were cyclically soaked in distilled water and saltwater (with 3% salinity) solutions. Compression stress equal to 90 000 lb was also applied to one of the cubes soaked in saltwater solution at the end of each drying cycle to simulate in-service loading that such materials may experience. Microwave reflection property measurement of these cubes at S-band (3 GHz) showed a notable difference in $\varphi$ between the water and saltwater-soaked cubes. Although the influence of compression stress, which is expected to increase permeability, was detected in previous investigations conducted on mortar cubes, the results presented here do not show any clear evidence of this aspect. It is believed that the compressive
strength of 90,000 lb, which corresponds to approximately 40% of the compressive strength of these cubes, may have been insufficient to generate any significant micro cracking. Finally, a comparison of the reflection properties of concrete and mortar cubes subjected to similar conditions of soaking and drying was carried out to evaluate variations in the various phenomena that occur within the two cubes. In the case of concrete, it was observed that the microwave signal was not sensitive to the movement of water/saltwater within the cube, which primarily manifests itself in the phase of reflection properties, although evidence of this aspect was observed in the case of mortar. This suggests that the soaking time of 72 h, which was sufficient to cause significant permeation of water/saltwater in the mortar cube, was insufficient in the case of the concrete cube. This issue notwithstanding, the similarity in trend of the reflection properties for the two cubes over an entire cycle allows us to infer that the various phenomena occurring within the two cubes during the drying cycle are also similar. However, further study is required to fully understand these phenomena and to relate these microwave measurements to physical as well as any potential chemical changes within the cubes.

REFERENCES


J. T. Case (M’99) received dual B.S. degrees in electrical engineering and physics in 2003 from the University of Missouri-Rolla (UMR), where he is currently pursuing the M.S. degree in electrical engineering with an emphasis on microwave nondestructive evaluation. Currently, he is an Undergraduate Research Assistant and Graduate Research Assistant with the Applied Microwave Nondestructive Testing Laboratory, University of Missouri-Rolla.

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