Florida Department of Transportation Research

Use of Scanning Electron Microscopy and Microanalysis to Determine Chloride Content of Concrete and Raw Materials
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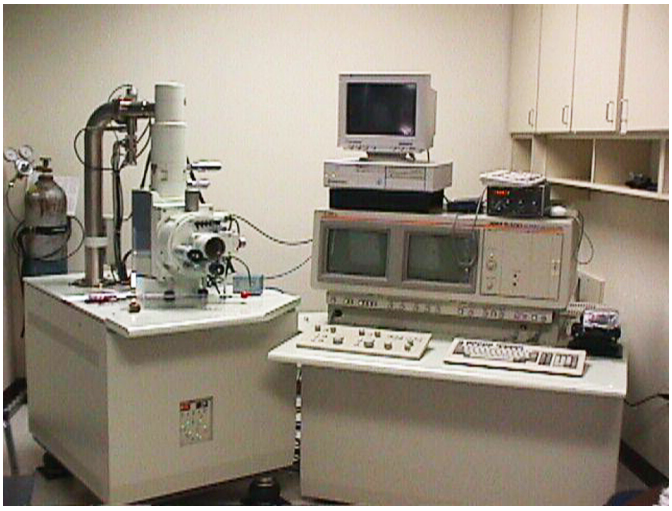
The Florida Department of Transportation (FDOT) is responsible for the maintenance of thousands of concrete structures that are exposed to or situated in salt water. Considering the significant cost of each of these structures, FDOT would like a 75-year span before any major repair is needed. These structures are invariably reinforced with steel, which is subject to corrosion if chloride ions from the marine environment can penetrate the concrete, so the diffusion rate of chloride ions through concrete is a critical durability issue.

FDOT had established an in-house chloride monitoring system at the State Materials Office (SMO) in Gainesville, Florida. Samples cored from structures like bridge piles and foundations in chloride-rich aqueous environments are routinely analyzed and profiled by FDOT-SMO to assess structure durability and develop corrosion-remediation and repair plans.

Basic analysis of a cored sample involves cutting the sample into sections, each of which is then pulverized, digested in nitric acid, and titrated using silver nitrate to determine the total chloride content. While this method provides an extremely accurate and sensitive measurement of *total* chloride in the section, it is time consuming and arduous, destroys the sample, yields only one data point per section, has poor spatial resolution, and provides no information about the in situ spatial distribution of chloride in the sample.

To investigate the possibility of a new chloride-diffusion test, FDOT-SMO collaborated with the Major Analytical Instrumentation Center (MAIC), a research service center in the University of Florida's College of Engineering; the MAIC has a vast array of analytical instruments. In this project, they sought to develop a non-destructive analytic method using the various electron beam instruments and corresponding x-ray spectroscopy techniques available at MAIC.

The scanning electron microscopes (SEM) at



A suite of scanning electron microscopes with enhanced capabilities makes possible a new kind of chloride diffusion analysis.

MAIC are equipped with Energy Dispersive Spectroscopy (EDS) systems. One SEM is an Electron Probe Micro-Analysis instrument (EPMA) equipped with a wavelength-dispersive x-ray spectroscopy (WDS) system. The capabilities of these instruments were the basis of the developed method. However, MAIC researchers also had to define and develop specific techniques for sample preparation and calibration. The resulting x-ray maps of chloride ion concentration produced diffusion measurements within a few percent of the same values from the conventional methods — a remarkable correspondence between two very different approaches.

Use of the latest technologies to create a more rapid and accurate chloride ion diffusion test will give FDOT a more precise understanding of chloride penetration through concrete structures. This in turn can lead to development of better concrete mixtures and better construction methods for new structures and more accurate service plans for existing structures, all of which results in savings while improving the durability of Florida's transportation infrastructure.

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For more information, visit http://www.dot.state.fl.us/research-center