OKLAHOMA TRANSPORTATION CENTER

ECONOMIC ENHANCEMENT THROUGH INFRASTRUCTURE STEWARDSHIP

PROCUREMENT OF NOVEL MICROANALYSIS EQUIPMENT FOR CONSTRUCTION MATERIALS

TYLER LEY, PH.D., P.E.
BRYAN SUDBRINK
MOHAMMED ABOUSTAIT

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Oklahoma Transportation Center
2601 Liberty Parkway, Suite 110
Midwest City, Oklahoma 73110

Phone: 405.732.6580
Fax: 405.732.6586
www.oktc.org

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16. Abstract

The equipment procured (i.e. an Orbis micro X-ray Fluorescence (MXRF) and an APSEX "personal" Scanning Electron Microscope (PSEM)) is part of the next generation of micro analytical equipment. These tools have the ability to make large volumes of observations with very little human interaction. This ability to make these educated observations will make huge impacts in the future of materials research. These instruments combine recent improvements in computing power, image analysis, automation, optics, and signal intensities to investigate samples autonomously while using higher signal intensities that minimize sample preparation.

Within the first year of owning this equipment it has been highlighted in grants that have totaled almost $600K in external grants. These major projects were funded by the Oklahoma DOT and the National Science Foundation. In addition several seed grants have been obtained that will hopefully turn into future funding.

Several technology transfer efforts from this project have been completed. An award winning poster was presented at the ODOT research day. In addition a website (http://oktcml.okstate.edu/) has been launched that describes these microscopes and shows their abilities. Results and findings from two different research efforts are presented and summarized.

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SI (METRIC) CONVERSION FACTORS

Approximate Conversions to SI Units

Symbol	When you know	Multiply by	To Find	Symbol
		LENGTH		
in	inches	25.40	millimeters	mm
ft	feet	0.3048	meters	m
yd	yards	0.9144	meters	m
mi	miles	1.609	kilometers	km
		AREA		
in^2	square inches	645.2	square millimeters	mm
ft^2	square feet	0.0929	square meters	m^2
yd^2	square yards	0.8361	square meters	m^2
ac	acres	0.4047	hectares	ha
mi^2	square miles	2.590	square kilometers	km^2
		VOLUME		
fl oz	fluid ounces	29.57	milliliters	mL
gal	gallons	3.785	liters	L
ft^3	cubic feet	0.0283	cubic meters	m^3
yd^3	cubic yards	0.7645	cubic meters	m^3
		MASS		
oz	ounces	28.35	grams	g
lb	pounds	0.4536	kilograms	kg
T	short tons (2000 lb)	0.907	megagrams	Mg
		TEMPERATURE (exact)		
°F	degrees Fahrenheit	(°F-32)/1.8	degrees Celsius	°C
		FORCE and PRESSURE or STRESS		
lbf	poundforce	4.448	Newtons	N
lbf/in^2	poundforce per square inch	6.895	kilopascals	kPa

Approximate Conversions from SI Units

Symbol	When you know	Multiply by	To Find	Symbol
		LENGTH		
mm	millimeters	0.0394	inches	in
m	meters	3.281	feet	ft
m	meters	1.094	yards	yd
km	kilometers	0.6214	miles	mi
		AREA		
mm^2	square millimeters	0.00155	square inches	in^2
m^2	square meters	10.764	square feet	ft^2
m^2	square meters	1.196	square yards	yd^2
ha	hectares	2.471	acres	ac
km^2	square kilometers	0.3861	square miles	mi^2
		VOLUME		
mL	milliliters	0.0338	fluid ounces	fl oz
L	liters	0.2642	gallons	gal
m^3	cubic meters	35.315	cubic feet	ft^3
m^3	cubic meters	1.308	cubic yards	yd^3
		MASS		
g	grams	0.0353	ounces	oz
kg	kilograms	2.205	pounds	lb
Mg	megagrams	1.1023	short tons (2000 lb)	T
		TEMPERATURE (exact)		
°C	degrees Celsius	9/5+32	degrees Fahrenheit	°F
		FORCE and PRESSURE or STRESS		
N	Newtons	0.2248	poundforce	lbf
kPa	kilopascals	0.1450	poundforce per square inch	lbf/in^2

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Final Report: February, 2012

Tyler Ley, Ph.D., P.E.
Bryan Sudbrink
Mohammad Aboustait

Oklahoma State University
Civil and Environmental Engineering Department
207 Engineering South
Stillwater, OK 74078

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EXECUTIVE SUMMARY

This report outlines the procurement of three novel pieces of analytical equipment and their use to investigate construction materials. This equipment includes an Orbis micro X-ray Fluorescence (MXRF), an APSEX "personal" Scanning Electron Microscope (PSEM), and an Automated Scanning Electron Microscope (ASEM). The equipment has been purchased, installed, inspected by the OSU radiation safety officers and are currently being used on two major grants.

The equipment procured is part of the next generation of micro analytical equipment. These tools have the ability to make large volumes of observations with very little human interaction. This ability to make these educated observations will make huge impacts in the future of materials research. These instruments combine recent improvements in computing power, image analysis, automation, optics, and signal intensities to investigate samples autonomously while using higher signal intensities that minimize sample preparation.

Within the first year of owning this equipment it has been highlighted in grants that have totaled almost $600K in external grants. These major projects were funded by the Oklahoma DOT and the National Science Foundation. In addition several seed grants have been obtained that will hopefully turn into future funding. These grants have come from Virginia Department of Transportation, Nelson Architectural Engineering, The Light Weight Aggregate Association, and Kleinfelder Engineering.

Several technology transfer efforts from this project have been completed. An award winning poster was presented at the ODOT research day. In addition a website (http://oktcml.okstate.edu/) has been launched that describes these microscopes and shows their abilities.

Results and findings from two different research efforts are presented and summarized.

INTRODUCTION

In the coming decades, expenditures to restore and renew transportation infrastructure in the United States will be considerable. Improvements in infrastructure durability, sustainability, economy, safety, and construction speed are needed. One technical area linked to these needs is the material science of construction materials. Despite using materials such as metals, concrete, asphalt and soils in transportation infrastructure for centuries, much is still unknown as they are very complex materials. The National Academy of Engineers reiterated this point in their report "Grand Challenges in Engineering for the 21st Century"[1] as it specifically mentions the need to improve existing construction materials and possibly develop new ones.

Because of this need, the OkTC researchers have devoted considerable efforts to improving the understanding and performance of construction materials. While previous research has focused on the bulk properties of these materials, significant work is needed to correlate these bulk properties to properties at the micro to nano scales. Work at smaller scales has posed considerable challenges due to the great amount of labor required to prepare specimens and manually operate the instruments to generate the massive number of observations needed to draw useful conclusions.

BACKGROUND

Through collaborations with the National Institute of Standards and Technology (NIST), the investigators have become familiar with the next generation of micro analytical equipment to help overcome these challenges. These instruments combine recent improvements in computing power, image analysis, automation, optics, and signal intensities to investigate samples autonomously while using higher signal intensities that minimize sample preparation. The addition of this recently available equipment to the central core facilities of the Oklahoma Transportation Center (OkTC) has allowed new findings and improvements in construction materials, promoted collaboration between researchers from different disciplines and universities, and elevated the facilities of the OkTC above other research institutions.

[1] NAE, Grand Challenges in Engineering for the 21st Century. 2009 [cited 2012 February 1]; Available from: http://www.engineeringchallenges.org/.

OBJECTIVES

The goal of this project is to purchase three major pieces of analytical equipment for all OTC researchers to use in their projects.

1. X-ray Florescent Microscope (MXRF) – A microscope that allows automated surface and chemical mapping of large areas (100 mm x 200 mm) with very little sample preparation
2. Automated Scanning Electron Microscope (ASEM) –An SEM that has been specifically designed to measure the shape, size, and chemistry of a large number of micro and nano particles.
3. X-ray tube for the Micro Computed Tomography Scanner (MCT) – The X-ray tube for a piece of equipment that provides 3D mapping of materials at the micron scale

SCOPE

This effort allowed the OkTC and OSU to be at the forefront of construction materials research. This equipment has and will continue to allow the OkTC peer institutions to complete highly innovative research and be strong competitors for national funding for decades to come. We look forward to building regional and national collaborations.

This report shows that within the first year this equipment has led to funding for two major multi-discipline research projects with funding of almost $600K. One of these includes the prestigious National Science Foundation CAREER Award. In addition four pilot projects with total funding over $20K have been granted that will hopefully lead to larger projects.

In addition a website has been established to highlight the capabilities of the equipment. (http://oktcml.okstate.edu/)

TECHNOLOGY TRANSFER

To help facilitate the use of this equipment and promote collaborations between the OTC partners and outside industry, a website has been created that is designed to help educate about the capabilities and use of these pieces of equipment for construction and other materials. This website provides a brief explanation of how the equipment works, training videos on how to use the equipment.

The investigators are also fortunate to have the capabilities of the film and editing crews of the OSU Institute of Teaching and Learning Excellence (ITLE) available for free assistance with the creation of these videos. This service is free from the OSU Provost if the videos are to be used for educational purposes. In addition an award winning poster was created for the ODOT research day of 2011 that strongly utilized the equipment in this proposal.

RESULTS

The Orbis micro X-ray Fluorescence (MXRF), APSEX "personal" Scanning Electron Microscope (PSEM), and Automated Scanning Electron Microscope (ASEM) have been received, installed, and started to be used for this project. The training for these instruments have also been completed and the University radiation safety office has accepted the safety plans proposed by the PI. The instruments are now opperational and are being used on several different research projects.

A short summary about the installation and applications of the instruments are described below. Some description about future work is also given. An overview of the users of the instrument is given in Table 1.

Table 1: A Summary of the Experiments Completed On the Purchased Equipment

User	Department (OSU)	Item	Summary of Work
John Veenstra	Civil and Environmental Engineering	ASEM	Investigate the heavy fowling on a polymer filter for water treatment for carbon content and metals.
Tyler Ley	Civil and Environmental Engineering	ASEM	Rapid measurement of fly ash diameter and chemistry
Tyler Ley	Civil and Environmental Engineering	MXRF	Determine a profile of chlorides in service concrete
Tyler Ley	Civil and Environmental Engineering	MXRF	Observing the presence of organosilanes in concrete
Tyler Ley and Jay Hanan	Civil/Mechanical Engineering	MXRF	Image the chemical dissolution of fly ash particles over time.
Tyler Ley	Civil and Environmental Engineering	MCT	Evaluate the air-void system within air-entrained paste and determine the response of bubbles to pressure.
Tyler Ley and Jay Hanan	Civil/Mechanical Engineering	MCT	Complete 3D chemical maps of fly ash particles and investigate subsequent dissolution in different soak solutions.
Jay Hanan	Mechanical and Aerospace Engineering	MCT	Deformation mechanics of amorphous metallic foam.
Jay Hanan	Mechanical and Aerospace Engineering	MCT	Mechanics if polymer (PMI) foam in compression.
Hongbing Lu	Mechanical and Aerospace Engineering	MCT	First visualization of the human cochlea paired with the structure of the ear drum.
Jay Hanan	Mechanical and Aerospace Engineering	MCT	Investigate the interface and 3-D structure of ceramic composites.
Jay Hanan	Mechanical and Aerospace Engineering	MCT	Compression mechanics of sand.
Hongbing Lu	Mechanical and Aerospace Engineering	MCT	Structure of a sugar composite
Jay Hanan	Mechanical and Aerospace Engineering	MCT	First observation of cross-linked aerogel 3-D nanostructure.
Jay Hanan	Mechanical and Aerospace Engineering	MCT	FEM of real dental crowns using MCT structure to predict residual stress.
Jay Hanan	Mechanical and Aerospace Engineering	MCT	Analysis of injection molded polymer components for design optimization.
Jay Hanan	Mechanical and Aerospace Engineering	MCT	Defect distribution in ceramic dental crowns for sample selection and life prediction
Jay Hanan	Mechanical and Aerospace Engineering	MCT	Defect distribution in ceramic armor materials for dynamic modeling predictions (a collaboration with John Hopkins University).
Ranga Komanduri	Mechanical and Aerospace Engineering	MCT	Arrangement and compaction of glass beads.

USE OF THE MXRF FOR IMAGING OF ORGANOSILANE IN CONCRETE

Organosilanes are used in many countries and states including Oklahoma to prolong the service life of bridge decks by minimizing the damaging effects of chloride ingress. Currently, no literature exists that outlines the useful lifespan of an organosilane coating. This project is funded by the Oklahoma Department of Transportation to investigate organosilane that has been in service in bridges from 5 to 25 years to determine the presence and effectiveness of the organosilane layer.

X-Ray Fluorescence Microscope

Micro X-ray Fluorescence is a microanalysis technique that uses an X-ray optic to focus a stationary beam of X-rays onto a sample to perform a chemical composition analysis. The sample will absorb the X-rays and emit a secondary fluoresced X-ray. The fluoresced X-ray intensities are then assigned as pixels into an image; examples are in Figures 1 and 2.

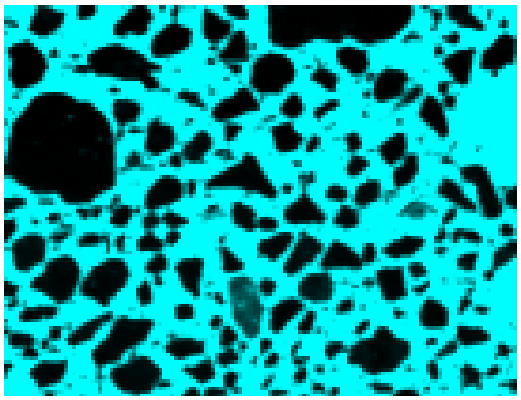

Figure 1: Calcium Image

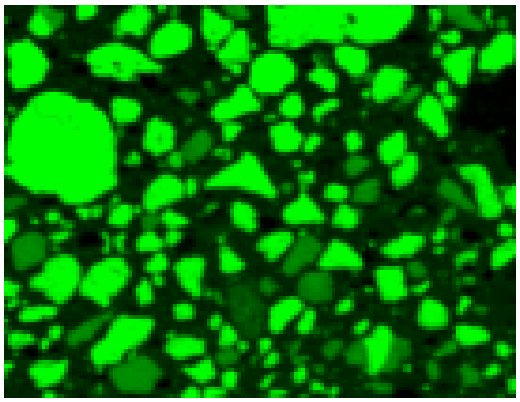

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Figure 2: Silicon Image

Cluster Analysis

The data from the MXRF is analyzed with cluster analysis. Cluster analysis is a means to identify the surface of the scanned sample by grouping areas that have a similar chemical composition into a cluster (which is called a 'phase' by the image processing software). This analysis provides valuable information such as what the sample consists of (i.e. chemical composition), where these components are located on the sample surface, and the quantity of those components at that location.

Clusters, or phases, are developed by selecting areas of high concentration because this is what is most prevalent in any given area. Since an area will most likely consist of multiple elements, the phase will consist of the area of overlap of all elements in consideration for the given phase, as shown in Figure 3.

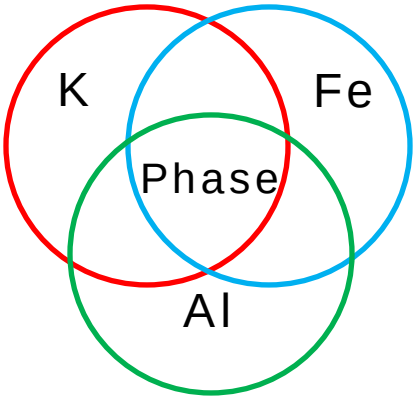

Figure 3: A Phase Defined by Aluminum, Iron, and Potassium

This process is repeated until the entire surface has been defined (i.e. multiple phases). Then a phase map, like that shown in Figure 4, is produced which combines all individual phases into a single image where each color in the phase map represents a separate phase.

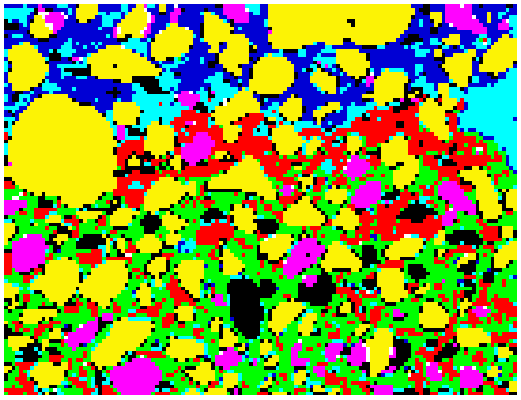

Figure 4: Example of a Phase Map

Using MXRF to Identify Organosilane in Concrete

One of the main goals of this project was to be able to identify the organosilane coating within a concrete sample using MXRF. To accomplish this, a series of scans of organosilane coated concrete were performed using the machine.

The first scan was performed on a coated sample made in the laboratory which was not modified in any way in order to see what kind of data it would produce. One very distinct observation, as shown in Figure 5, was a noticeable high band of sulfur (i.e. bright blue pixels) which was not found in samples without an organosilane coating. This high band of sulfur was thought to be just below the organosilane boundary.

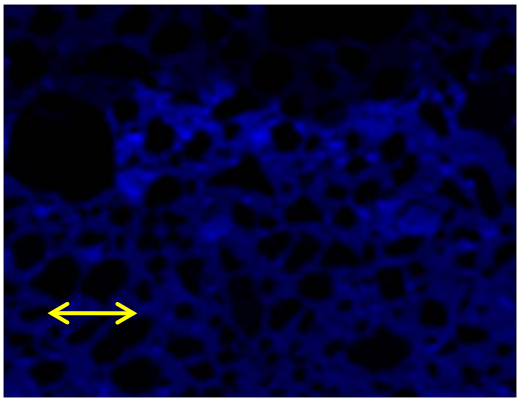

Figure 5: Sulfur Image from MXRF Machine (double arrow represents 5 mm)

To confirm this observation, the sample was ponded in a colored dye for 5 minutes and rescanned. The dye permeated the concrete only where organosilane was not present, thus establishing a visual boundary that could be observed. A comparison of an optical image of the sample (Figure 6) after it was ponded in colored dye and the sulfur image is shown in Figure 7. The organosilane boundary has been highlighted with a yellow line.

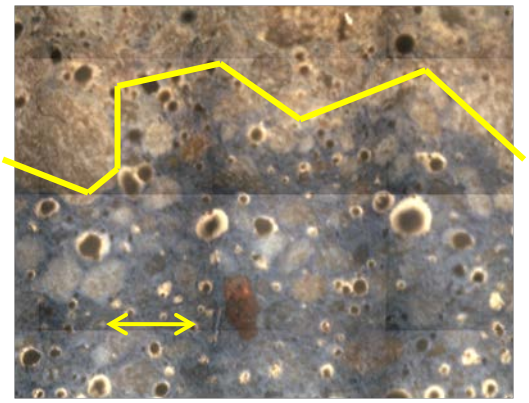

Figure 6: Optical Image with Colored Dye (double arrow represents 5 mm)

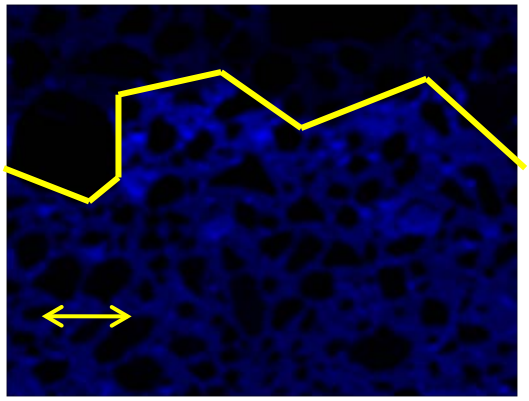

Figure 7: MXRF Sulfur Image (double arrow represents 5 mm)

The corresponding phase map is displayed in Figure 8 where the high sulfur band in the cement paste is characterized by the area in red. Likewise, low and medium concentrations of sulfur containing cement paste are characterized by blue and green, respectively. The same boundary that is highlighted by the yellow line in Figure 7 has been highlighted by the black line in Figure 8.

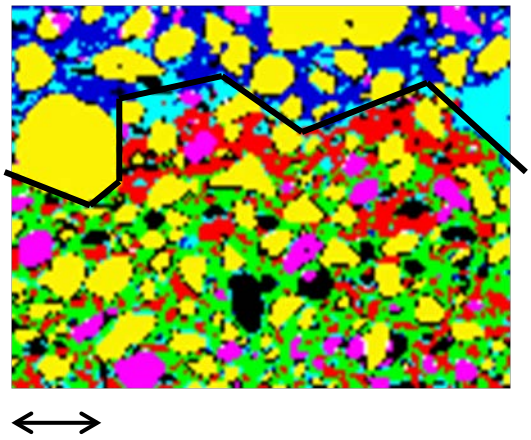

Figure 8: Phase Map Corresponding to Figures 6 and 7 (double arrow represents 5 mm)

In addition to using dye, MATLAB ver. R2009a, image processing software, was used to establish a mathematically based contour line. A comparison of the optical image of the sample after it was ponded in colored dye and the image processed in MATLAB is shown in Figures 9 and 10. The organosilane boundary in all subsequently scanned samples was then established by means of this observation.

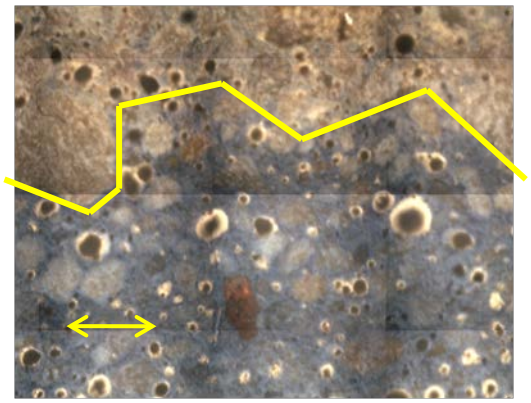

Figure 9: Optical Image with Colored Dye (double arrow represents 5 mm)

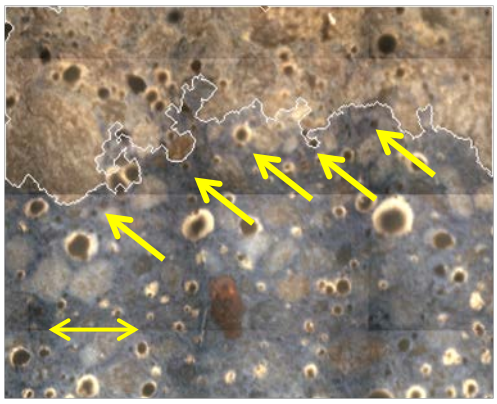

Figure 10: Image Processed in MATLAB where the Arrows Point to the Contour Line (double arrow represents 5 mm)

Field Cores

Using the techniques described above, a total of 12 field samples were investigated using MXRF to determine whether or not each sample contained organosilane. A total of 10 samples showed no definitive evidence of the presence of organosilane. As shown by the example of sample SH-51 #2 in Figure 11, those that showed no signs of organosilane were deemed so by an absence of the high sulfur band and displayed no particular sulfur pattern.

Two samples showed definitive evidence of the presence of organosilane, as shown by the example of sample I-40 #1 in Figure 12.

Conclusion

Micro X-ray Fluorescence is a powerful tool capable of identifying the presence of organosilane in concrete. With the aid of this tool, routine maintenance can performed on

structures where organosilane has been applied. This routine maintenance will help maximize the service life of the structure by minimizing the damaging effects of chloride ingress.

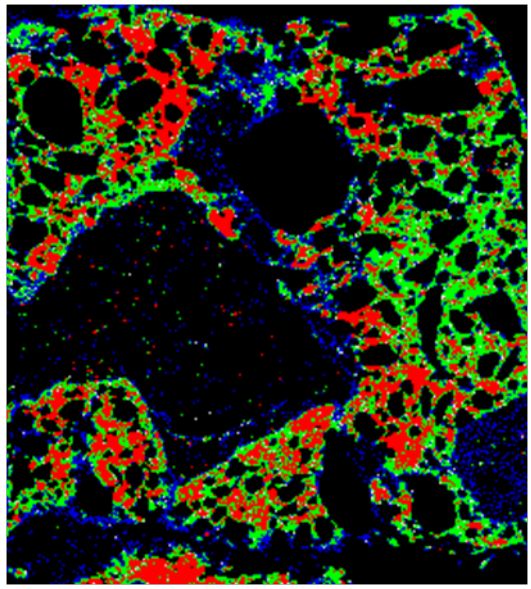

Figure 11: Cement Paste Phase Map Showing No Definitive Organosilane Evidence (double arrow represents 5 mm)

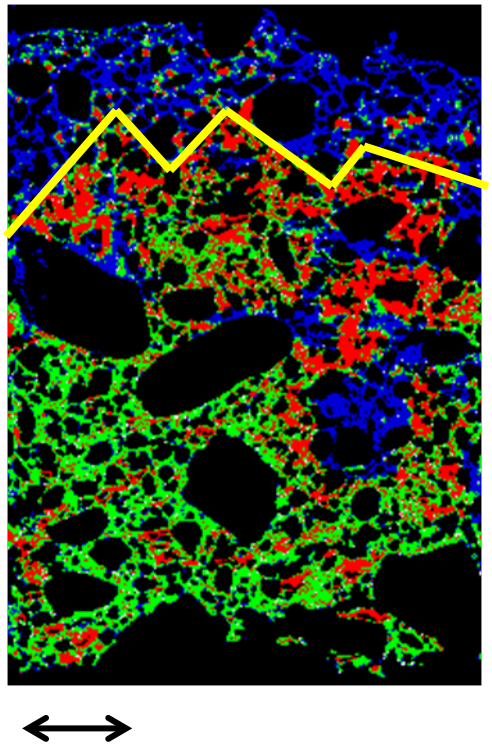

Figure 12: Cement Paste Phase Map Showing Definitive Organosilane Evidence (double arrow represents 5 mm)

USE OF THE ASEM TO INVESTIGATE FLY ASH

The ASPEX "personal" Scanning Electron Microscope (PSEM) Explorer has been installed at the Microscopy Center at OSU. This system provides an SEM with built-in energy dispersive x-ray spectrometry (EDS) to provide a fully integrated platform with superior capabilities such as outputting fully detailed chemical elemental mapping for the scanned surfaces.

In general, SEM uses a focused electron beam to scan the surface of a sample. During such a scan, a variety of signals that contains diverse information is being generated from the sample surface and collected by different detectors. There are three modes of operation installed within this SEM which are the backscattered electron detector (BSD), secondary electron detector (SED), and EDS. Where BSD and SED are used to obtain images for tomography purposes at high and low accelerating voltages respectively, while the EDS offers the chemical composition and characterization X-ray spectrum of the scanned sample subjectively under vacuum conditions.

This SEM contains an Automated Feature Analysis (AFA) option which can produce fully detailed information about the scanned samples. The EDS AFA data are usually obtained from high energy beam scan (20 KeV) in a preferably raster chord scan pattern. The output contains high magnification images, compositional analysis and robust data for size, shape and elemental composition. A sample of the data produced for each fly ash particle is shown in Figure 13.

This analysis can be completed in an automated fashion where 5,000 particles can be investigated in a night. The SEM outputs its result, where the upper half of the picture is divided into two halves the left half shows a perfectly circular 20 micron particle, while the right half shows the crosser which indicates where the electron beam is shooting the same particle. The graph at the bottom shows the cross bonding x-ray spectrum where the excited elements from the fly ash particle are being presented depending on their quantity within it.

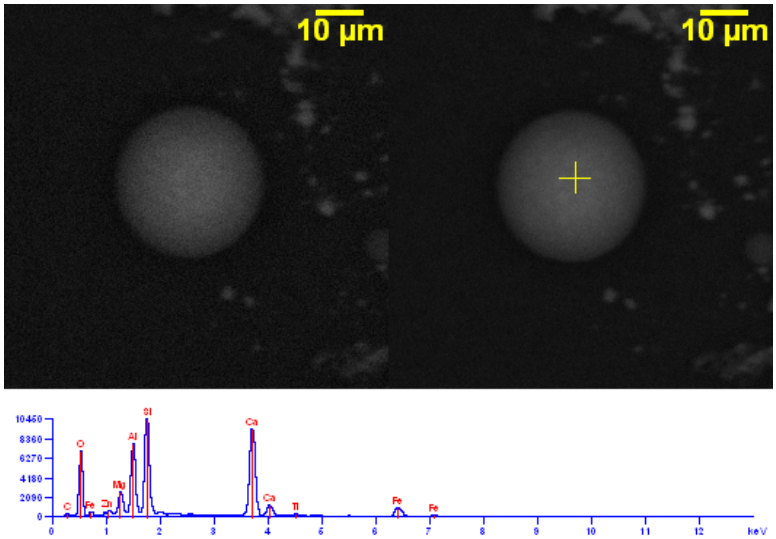

Figure 13: Fly Ash Particle Imaged Using BSD and Analyzed with EDS

Currently, there are two experiments that are being completed by the ASEM that are very important to the characterization and investigation of the reactivity of fly ash in concrete.

a) Fly ash soak dissolution
b) Fly ash surface chemistry and morphology on directly exposed stubs

Fly Ash Soak Dissolution

In this experiment, the particles have been attached to the end of tungsten pens in order to be investigated with the μCT to examine the solubility of the different constituents in the fly ash particles. In each cycle, the fly ash particles have been immersed in 6.5 mL of a 1N NaOH solution for 24 hours. While in this solution it is common for the portions of the fly ash particle to dissolve. By investigating the samples before and after they have been immersed in solution then one can learn about the reactivity of the different phases. Volumetric changes in the particle have been observed using multiple imaging techniques μCT, SEM (both SED and BSD) and a light microscope. At each cycle, a good positioning for the sample was maintained through marking one side of the tungsten pen and sample holder, such procedure helped in preserving the consistency of the obtained images. This information obtained from each cycle is required for better understanding the fly ash effects on the properties of concrete.

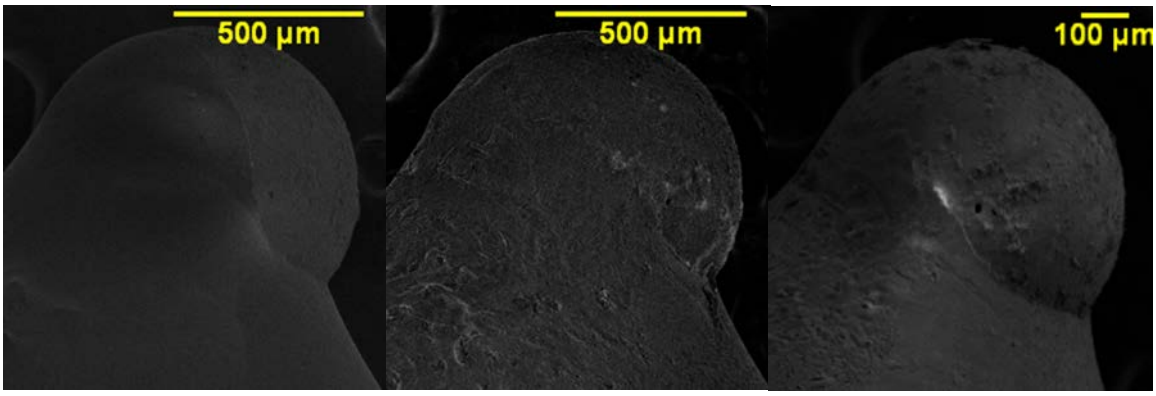

Figure 14: Fly Ash Particle at 0, 24, And 48 Hours Exposure to 1N NaOH Solution

Fly Ash Surface Chemistry and Morphology on Directly Exposed Stubs

In this experiment small fly ash particles are placed on carbon tape and investigated in the SEM. The automated capabilities of the SEM allow it to find the particles and then image them and then measure their chemistry. One challenge with this technique is that the SEM can only interrogate the near surface of the fly ash particles. While this is easy and rapid, this technique is not able to determine the chemistry throughout the sample. New techniques are being developed that are able to investigate fly ash particles that have been sectioned through polishing. Again the automated features of the microscope make it easy to get high quality images of the samples in a very rapid manner.

These techniques are able to provide large data sets about the chemistry of different fly ash particles in a very rapid fashion. This is a very useful tool to chemically characterize the different fly ash particles in a much larger powder.

Conclusions

This report has shown how several novel pieces of analytical equipment have been obtained and already used to obtain almost $600K in external grants within one year. These projects were funded by the Oklahoma DOT and the National Science Foundation. In addition several seed grants have been obtained that will hopefully turn into future funding. These grants have come from Virginia Department of Transportation, Nelson Architectural Engineering, The Light Weight Aggregate Association, and Kleinfelder Engineering.

Several technology transfer efforts from this project have been completed. An award winning poster was presented at the ODOT research day. In addition a website has been launched that describes these microscopes and shows their abilities.