

# Metallographic Application Note

## Cement Clinkers

In the production of cement, clinkers are the sintered lumps exiting the cement kiln. Clinkers are typically 1 to 25 mm in diameter.

### Cement Clinker Analysis

Clinker materialography is commonly performed and serves three basic purposes: quality control, problem solving, and the monitoring of process improvements.



### Quality Control

When clinkers are examined regularly, the microstructure of a plant's "typical" clinker is well known. If a change in the microstructure occurs (e.g. if the size or shape of the crystals changes, or a key component is not properly distributed), plant operators can react to modify processing parameters to return the clinkers to "normal". Mechanical tests (e.g. strength) and chemical tests (e.g. % free lime) provide valuable quality control information but do not tell the whole story. The relatively quick preparation of clinker samples and the information their examination can reveal make clinker materialography a valuable diagnostic tool.

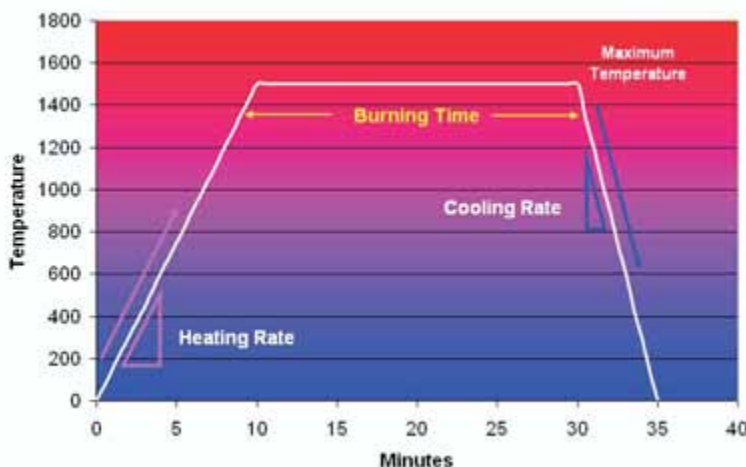
### Problem Solving

Examination of clinker microstructure can provide clues to solving cement production or performance issues. As stated previously, routine mechanical and chemical tests often provide insight, but microscopy can provide the missing pieces to the puzzle.

### Monitor Process Improvements

If a potential process improvement is implemented (e.g. temperature set point change, cooling rate increase, new raw material source, etc.), an analysis of the clinker microstructure is recommended before and after ANY change. Plant personnel can determine the positive and negative effects of the change and perhaps predict changes in end product performance.

Cement Clinker Thermal History



Typical Heating, Burning, and Cooling Cycle

## **Sample Storage**

Prior to beginning the preparation process, clinkers should be stored in a humidity controlled room or cabinet. This will help prevent hydration and carbonation of the clinker samples.

After preparation, polished samples can be protected during storage with an acrylic or lacquer spray, which is removed by rubbing gently with an acetone-soaked cotton ball.

## **Sample Preparation and Analysis**

### **Sectioning/Cutting**

**Equipment:** MSX205M2 sectioning machine equipped with a small vise (811-651-144) and a 7 inch (175 mm) diamond blade (809-150).



### **Mounting**

**Equipment:** Cold mounted with Long Cure Epoxy (812-522-HAZ) using 1.5 inch diameter plastic mold cups (810-992-012). Filled molds were placed in a vacuum chamber (at 28 in. Hg) for two 30 second cycles.



### **Grinding and Polishing**

**Equipment:** GPX200 with an Automatic Grinding/Polishing Head.



### Grinding (Fixed Holder)

GPX200 10" Wheel	Time (Minutes:Seconds)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (FPM)
Platinum #1 812-337, Microid Extender	2:00	CW	35	75	CCW	200
Platinum #2 812-338, Microid Extender	2:00	CW	35	75	CCW	200

### Polishing

	Time (Minutes:Seconds)	Head Direction	Head Pressure (lb.)	Head Speed (RPM)	Wheel Direction	Wheel Speed (FPM)
3 $\mu$ Premium Suspension, Ultra Silk, Microid Extender, 810-997-016, 812-438, 811-003	3:00	CW	35	100	CCW	200
0.05 $\mu$ Gamma B Alumina Powder, Microid Extender, Lecloth, 810-778, 811-003, 812-224	1:00	CW	30	75	CCW	150

Note: Rinse only with ethyl alcohol between preparation steps to prevent staining of clinkers.

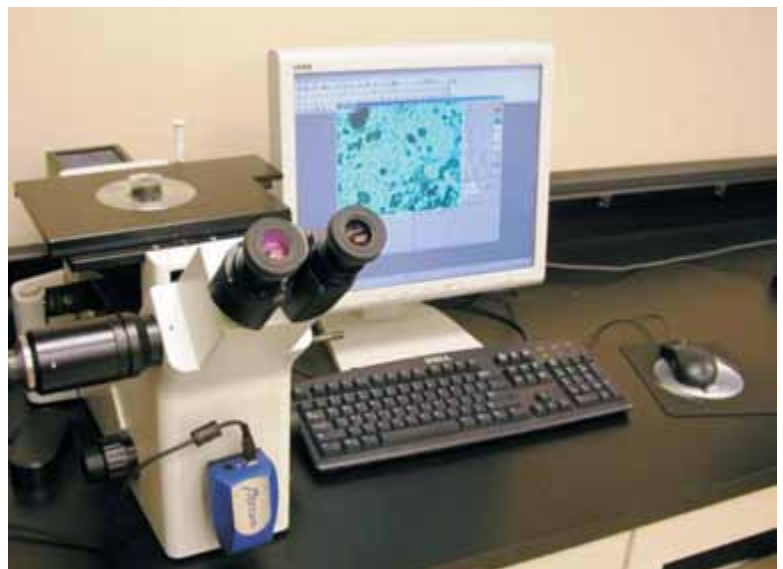
### Etching

A common clinker etchant is 2% Nital (a mixture of nitric acid and alcohol). The sample is typically immersed for 5 to 10 seconds. This etch, or stain, is used to differentiate the Silicates, Alite, and Belite in the Clinker sample.

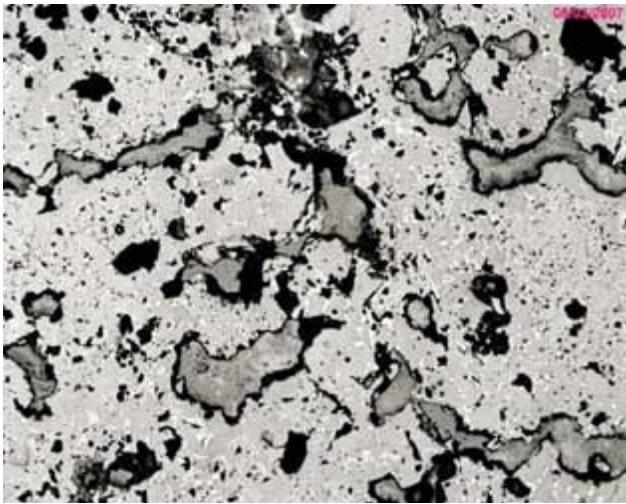
Another method of etching is the immersion in distilled water, at a temperature of 40 to 50°C (104 to 122°F), for 10 seconds. The etched sample is rinsed with alcohol and dried with forced warm air. With a water etch, free lime crystals show up as vivid, multicolored balloons.

### Analysis

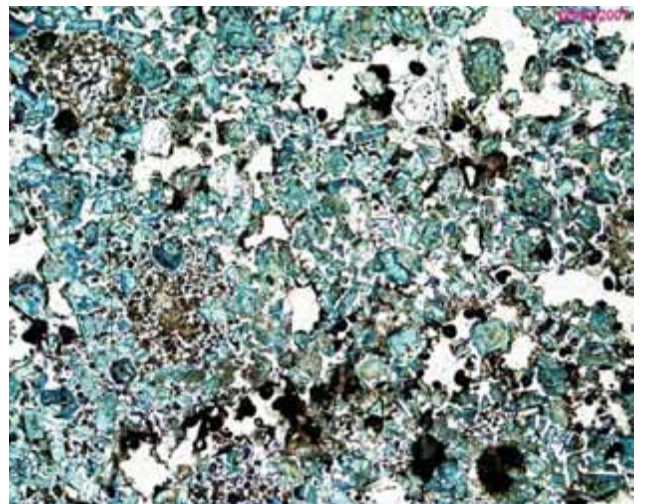
**Equipment:** Olympus GX51 BrightField Metallograph, PAXcam3 (3 Mp Color Digital Camera), and PAX-it™ Image Analysis and Management Software.



**Olympus GX-51 Metallograph with  
PAX-it Image Analysis software  
and PAXcam Camera**



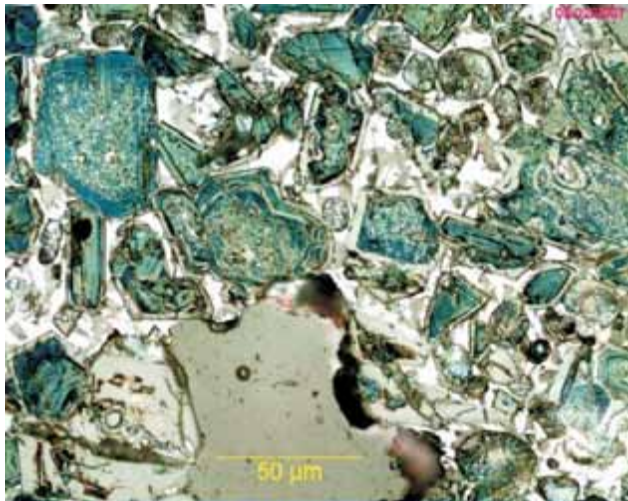
100X Water Etch



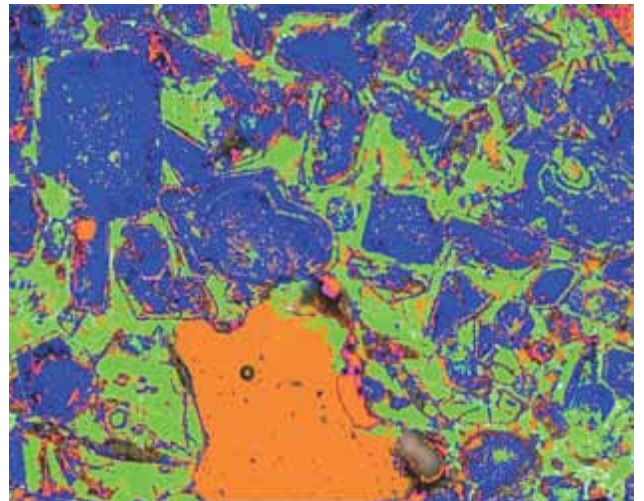
100X Nital Etch

### **PAX-it Analysis**

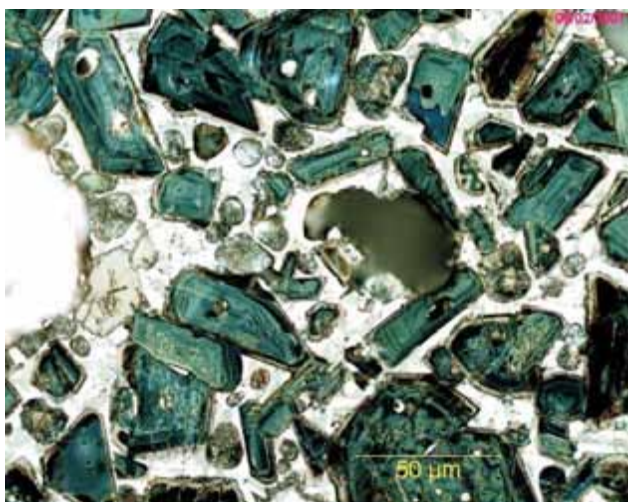
A PAX-it "Detect Areas" routine can be utilized to analyze the various phases in a prepared clinker sample. Because most of the phases appear as different colors under brightfield illumination, the analysis is best performed using thresholding by color, rather than utilizing grayscale. The results can be outputted via an Excel template. Example analysis results can be seen on the next page.



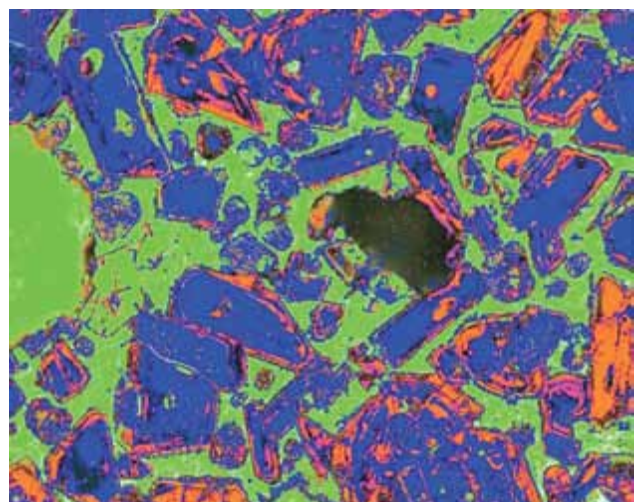
500X Nital Etch



500X Pax-it Color Thresholding



500X Nital Etch



500X Pax-it Color Thresholding



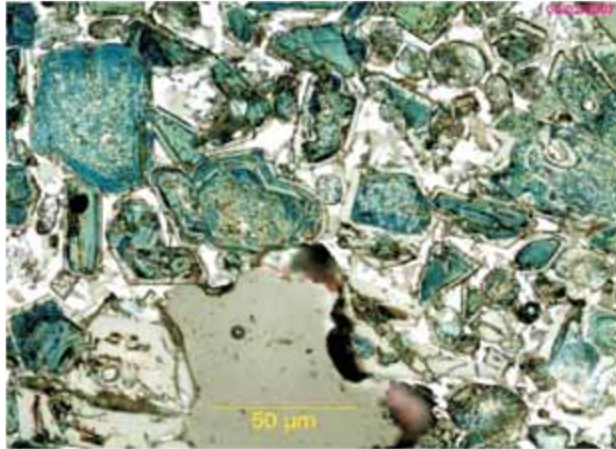
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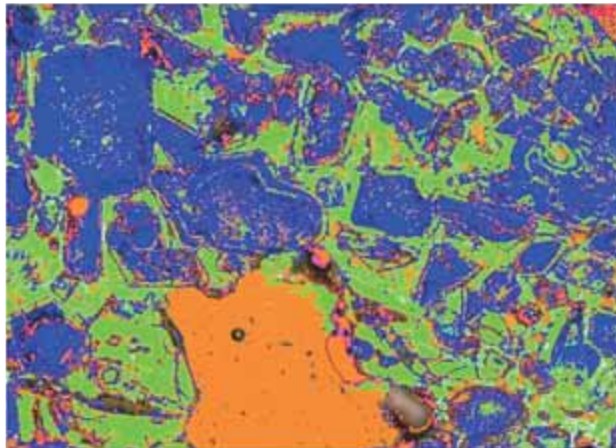
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Title **Mount 1 - 500X - Nital**  
Customer: **Anorex Cement**  
Magnification: **50X Front Coupler**  
Lab Tech: **43309**  
Comment: **500X**  
Condition: **Nital Etch**



Belite	0.0%	
Alite	29.7%	
Porosity	2.4%	
Liquid Phase	15.7%	
C3A	15.1%	
Total	62.8%	

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