

SUM NOTES

Preparation of Cement Clinkers

Introduction

Cement is an important component of concrete. Cement clinkers are produced by mixing limestone or other lime-bearing materials with silica-bearing materials such as clays. The mixture is heated to a temperature of approximately 1500 °C in a kiln. When the material comes out of the kiln, it is in the form of nodules. These nodules may vary in size and are commonly referred to as "clinkers". The clinker is ground with gypsum to a fine powder and becomes cement. This fine powder then reacts with water to bond together other components such as sand and aggregates.

A properly prepared cross-section of a clinker can reveal a variety of information when examined under the microscope.

- Shape and size of phases
- Homogeneity of the mix
- Alteration products
- Porosity level

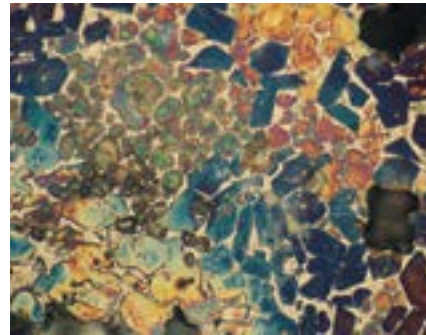
Evaluating these criteria will enable proper control of the product quality and the production costs. For example, grinding the clinkers to form a fine powder consumes a significant amount of energy. Controlling the porosity is key to optimizing the grinding process and therefore the manufacturing costs.

Preparation Procedure

Reflected Light Preparation

1. Place several clinkers in a mounting cup Vacuum impregnate the clinkers with EpoThin® Low Viscosity Epoxy to fill the pores. The procedure is:
 - Measure the resin and hardener according to the directions.
 - Blend the ingredients thoroughly, but gently to avoid excessive formation of air bubbles.
 - Allow the mixture to sit for a few minutes before using. This will allow any remaining entrapped air to rise to the top.
 - Pour enough epoxy over the specimen to fill the mold to the desired height and impregnate.
2. To achieve a flat surface grind the surface of the mount with 400 (P800) grit CarbiMet® SiC Paper or coarser depending upon the amount of material that has to be removed. To save grinding time large clinkers should be sectioned first with one of the IsoMet® Saws. If hydration is a concern, use a 50:50 mixture of propylene glycol and alcohol or an oil based lubricant.

Although clinkers can be ground with loose abrasives such as silicon carbide or aluminum oxide, loose abrasives have a



Polished bulk specimen of a cement clinker showing alite and belite, 100x.

tendency to seep into pores and are difficult to remove. Fixed abrasives will minimize cross contamination.

3. The specimen must be thoroughly cleaned at the end of each preparation step. Use alcohol to prevent hydration. If hydration is not a concern water and liquid soap can be used.
4. Continue grinding with 600 (P1200) grit CarbiMet® SiC Paper.
5. Observe the specimen surface under a microscope at the end of each step to determine if the time is sufficient. Pits and scratches proportional to the current abrasive size will be present. A uniform scratch pattern that doesn't improve regardless of the time spent indicates that the current step is complete. Clean the specimen thoroughly and move to the next step.
6. Use TexMet® 1500 with 6µm MetaDi® Diamond Paste for the first polishing step. An oil based lubricant or a 50:50 mixture of propylene glycol and alcohol is recommended. The polishing time will vary depending on the method of preparation, manual or automated.
7. Final polish with MicroCloth® and MicroPolish® B 0.05µm alumina polishing powder combined with a 50:50 mixture of propylene glycol and alcohol.
8. Wash the specimen thoroughly. Rinse with alcohol and dry.
9. Etchants are commonly used to distinguish the various phases. See Table 1 for an overview of etching reagents and stains.

Table 1. Etching Reagents and Stains for Cement Clickers

Structure to be Viewed	Etchant Composition	Comments
Aluminates and Free Lime	Distilled Water	Immerse the polished surface for 3 to 5 seconds
Silicates	1% Solutions of NH ₄ Cl in water or 1% HNO ₃ in Ethyl alcohol	Immerse the polished surface for 5 to 10 seconds
Interstitial Glass	10% KOH in water	For best results, the temperature of the etchant should be 30°C (86°F). Immerse for 10 to 15 seconds.
Stain to resolve Alite and Belite	Step 1: 1 gram NH ₄ NO ₃ , 150mL Isopropyl Alcohol, 20mL Water, 20mL Ethyl Alcohol, 10mL Acetone Step 2: Salicylic Acid Etchant 0.2grams Salicylic Acid, 25mL Isopropyl Alcohol, 25mL Water	Follow with Step 2 to resolve Alite and Belite. Apply for 30 seconds. Coloration may vary. When Alite is yellow-green, Belite will probably be brown.

Transmitted Light Preparation

Thin sections are observed under transmitted polarized light and permit a more comprehensive analysis of the specimen. In addition to revealing the features described thus far, mineral contents can also be determined.

Grinding and polishing thin sections by hand requires a great deal of expertise and time. Grinding by hand also tends to favor one side or another of the thin section, eventually making one side thinner.

Because uniformity of the specimen thickness is a primary concern, the process is often automated. The PetroThin® Thin Sectioning System is a self contained unit consisting of a diamond cutting blade, a diamond grinding wheel, and a vacuum chuck that accepts five sizes of glass slides. Two precision micrometers are used for controlling cutting and grinding of the thin section.

1. Section the clinker, preferably using an IsoMet® Precision Saw with a 15LC diamond wafering blade. A coolant such as straight propylene glycol can be used to eliminate hydration.
2. Embed in EpoThin® Low Viscosity Epoxy under vacuum. When cured, grind one side with 600 (P1200) and 800 (P1500) grit CarbiMet® Silicon Carbide Abrasive Paper until the entire surface is exposed and is flat.
3. Clean the specimen and slide thoroughly and dry. Attach the ground side of the specimen to the slide using epoxy. Use of PetroBond® Thin Section Bonding Fixture will ensure a strong uniform bond.
4. After the epoxy has cured, secure the slide in the PetroThin®.
5. Move the mounted specimen into the path of the PetroThin® Grinding Wheel as the micrometer accurately advances the specimen. Initially, remove 20 to 30µm of the specimen surface in each pass. However, as the specimen thickness approaches 50µm, only 5 to 10µm should be removed at one time to prevent shattering of the grains.
6. Remove the thin section when it is approximately 40µm thick.
7. Lap the thin section on a glass wheel with 9.5µm aluminum oxide, or with abrasive papers until the desired thickness is obtained.

8. After grinding, the thin section can be observed under the microscope or it can be polished.
9. Use TexMet® 1500 with 6µm MetaDi® Diamond Paste for the first polishing step. An oil based lubricant or a 50:50 mixture of propylene glycol and alcohol is recommended. The polishing time will vary depending on the method of preparation.
10. Final polish with MicroCloth® and MicroPolish® B 0.05µm alumina polishing powder combined with a 50:50 mixture of propylene glycol and alcohol.
11. Wash the specimen thoroughly. Rinse with alcohol and dry.

The polished thin section can be observed with either transmitted or reflected light.

Equipment*

Cast N' Vac 1000 Castable Vacuum System

IsoMet® Family of Linear Precision Saws

PetroThin® Thin Sectioning System

Consumables*

IsoMet® Diamond Wafering Blade

EpoThin® Low Viscosity Epoxy

CarbiMet® Abrasive Discs

TexMet® 1500

MetaDi® Diamond Paste

MetaDi® Fluid

Aluminum Oxide Abrasive Powder

MicroCloth® Polishing Cloth

MicroPolish® B 0.05µm Alumina

*For a complete listing of Buehler Equipment and Consumables, please refer to Buehler's Equipment Buyer's Guide and Buehler's Consumables Buyer's Guide

Sectioning AbrasiMet • AbrasiMatic • IsoMet	Mounting SimpliMet	Grinding & Polishing EcoMet • AutoMet • MetaServ	Imaging & Analysis OmniMet	Hardness Testing Wilson® Hardness
--	-----------------------	---	-------------------------------	--------------------------------------



BUEHLER Worldwide Headquarters
 41 Waukegan Road
 Lake Bluff, Illinois 60044-1699 USA
 P: (847) 295-6500
 www.buehler.com | info@buehler.com

BUEHLER Germany
 info.eu@buehler.com

BUEHLER France
 info.fr@buehler.com

BUEHLER United Kingdom
 info.uk@buehler.com

BUEHLER Canada
 info@buehler.ca

BUEHLER Japan
 info.japan@buehler.com

BUEHLER China
 info.cn@buehler.com

BUEHLER Asia-Pacific
 info.asia@buehler.com