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.

3. Although clinkers can be ground with loose abrasives such as silicon carbide or aluminum oxide, loose abrasives have a tendency to seep into pores and are difficult to remove. Fixed abrasives will minimize cross contamination.

4. 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.

5. Continue grinding with 600 (P1200) grit CarbiMet® SiC Paper.

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

<table>
<thead>
<tr>
<th>Structure to be Viewed</th>
<th>Etchant Composition</th>
<th>Comments</th>
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</thead>
<tbody>
<tr>
<td>Aluminates and Free Lime</td>
<td>Distilled Water</td>
<td>Immerse the polished surface for 3 to 5 seconds.</td>
</tr>
<tr>
<td>Silicates</td>
<td>1% Solutions of NH₄Cl in water or 1% HNO₃ in Ethyl alcohol</td>
<td>Immerse the polished surface for 5 to 10 seconds.</td>
</tr>
<tr>
<td>Interstitial Glass</td>
<td>10% KOH in water</td>
<td>For best results, the temperature of the etchant should be 30°C (86°F). Immerse for 10 to 15 seconds.</td>
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</table>

Stain to resolve Alite and Belite

1. Step 1: 1 gram NH₄NO₃, 150mL Isopropyl Alcohol, 20mL Water, 20mL Ethyl Alcohol, 10mL Acetone
2. 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.
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