

ETCHING & STAINING

Etching

Etching a polished section of a clinker, cement, slag, or concrete with a chemical reagent (etchant) highlights various components (e.g., individual clinker phases, residual clinker particles in concrete, slag, etc.) by selective absorption of the etchant with removal of surface layers of the components of interest in solution. The etched surface is observed in a high-power reflected-light (metallurgical) microscope. Etching of carbonate aggregates by dilute hydrochloric acid produces CO₂-effervescence. Limestone aggregates show higher effervescence than dolomitic aggregates. Etching can be performed on a smooth, dry, highly polished section, which is free of any surface irregularities and lubricants from the previous grinding or polishing operations. Usually the polished surface is immersed into a thin layer of etchant in a shallow petri dish (or held above in case of diluted HF acid vapor etchant; HF acid is placed in a platinum crucible and the inverted polished surface is held above it) and is then washed with alcohol to stop the reaction and quickly dried in an air current. Nital, HF vapor, potassium hydroxide in alcohol, and salicylic acid in alcohol are the most common etchants used in clinker microscopy. Borax and sodium hydroxide solutions are the etchants for examinations of high alumina cement. Table 3 provides twenty different staining and etching procedures for examination of polished sections of clinker, cement, raw feeds, slag, concrete, and aggregates.

Crack Identification

Various authors suggested a dye impregnation method for highlighting cracks in concrete by solvent replacement procedure, which involves immersing a wet, cleaned, finely ground, and polished section of concrete (polished with 6- μ m diamond paste) in a red or fluorescent dye-mixed alcoholic solution, followed by careful re-polishing in water with 1 to 3- μ m diamond to remove any excess dye from the surface. Dye-impregnated micro and macro cracks are easily highlighted in reflected-light examination. A fluorescent dye mixed alcohol treated ground or polished surface can highlight many fine cracks when examined in ultraviolet light. Dye-mixed epoxy impregnated thin sections are also excellent for highlighting cracks.

Staining

Selective staining of finely ground, polished or thin sections of rock, clinker, cement, or concrete with a chemical reagent highlights various components with a reaction product remaining on the surface which is either colored or can develop

characteristic colors by further treatment. Hutchinson and Campbell described various staining techniques applied on uncovered or polished thin sections to highlight various silicates (feldspar, quartz, mica) and carbonates (calcite versus dolomite) in rocks, aggregates, and raw feeds.