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New challenges for clinker microscopy

Using careful sample preparation conditions, clinker microscopy assessment of subsequent process or materials changes by an experienced operator can reveal important quality-modifying microstructure aspects. Here CTL Group's senior microscopist A Derek R Brown discusses the misconceptions, interpretations and challenges involved in clinker microscopy.

For many decades, clinker microscopy has been a valuable technique for assessing many aspects of clinker production and resulting cement quality. Clinker 'quality' can be defined in several ways, the most obvious one being the strength growth potential resulting from a well combined 'reactive' clinker that is free of detrimental defects. However, the ability to be used with an optimum level of gypsum, or to be free of a tendency to 'silo set,' or to not promote uncontrolled setting (due to excessive alkali modified aluminate), are the other 'quality' indicators. Also, the ability to activate slag and fly ash to optimise early strengths is an increasingly important quality parameter for portland cement.

Clinker microscopy can be used to assess many of the factors influencing 'quality' in its various definitions. However, it is obvious that correct interpretation of observations are vital for the technique to be useful. This calls for the need for experienced microscopists to avoid misinterpretations.

For example, a common misconception is to attribute all occurrences of large belite clusters to troublesome siliceous raw materials and/or inadequate raw milling of such materials. However, it is often the case – following correct observation of other clinker features – to see that the belite clusters are just one of several features indicating light burning conditions and incomplete combination of the raw feed compo-

nents. It can easily be shown that with optimised kiln conditions, such belite clusters will be removed due to better combination—without any change to the raw materials or to raw feed residue percentages. This illustrates the challenges facing the microscopist to make correct and meaningful assessments. This challenge is amplified by recent production practices that can introduce changes to clinker microstructure beyond those commonly encountered.

These relatively new production practices include, but are not limited to, the use of alternative raw materials derived from industrial by-products, the introduction of multi-channel burner pipes, and clinker mineralisation.

The use of by-products either into the raw feed or, more importantly, by mid-kiln introduction has been shown to sometimes introduce gross heterogeneity into the microstructure of the resulting clinker. Due to either problems in grinding such materials fine enough or the incomplete combination of coarse material introduced into the kiln later in the process, the resulting heterogeneity can cause significant loss in strength in cements prepared from such substandard clinker. Materials such as foundry sands, spent alumina catalysts, and coarse slag therefore need assessment of their effect on the clinker.

Multi-channel burner pipes have at times produced heterogeneity of a different kind in clinker microstruc-

ture. Open textured, partially combined feed materials can survive through the burning zone. The reasons for this require further study, but appear to involve localised changes to the gaseous environment in the burning zone and the greater dispersion of the flame's heat when the burner setup is not optimised.

Currently, mineralisation of clinker is primarily employed as a fuel-saving tool without compromising the properties of the clinker. It is achieved by various approaches, but they generally introduce some additional complexity into the process. Monitoring by clinker microscopy can check for aspects such as excessive belite stabilisation due to incorporation of sulfate into the lattice, or a tendency to encounter reducing conditions where fuels such as petcoke are used as a sulfate input route. Microscopy will also reveal the presence of build-up material that has dislodged and become incorporated within the clinker.

As with all comprehensive studies using clinker microscopy, two important prerequisites are:

- 1) the establishing of 'baseline clinker parameters' that are typical for one kiln system under normal operation, and
- 2) the study of sufficiently large and representative clinker samples using all nodule sizes plus fines. Clinker nodules should not be deliberately crushed for analysis since important features and microstructural variations from the nodule core to periphery may be lost.

Using careful sample preparation conditions, clinker microscopy assessment of subsequent process or materials changes, by an experienced operator, can reveal important quality modifying microstructure aspects. Observations at very low magnifications should always be the first step since some heterogeneities can be on a millimeter not micron scale. Close communications with the process engineers and control room staff is a crucial part of the procedure.

About the author



A Derek R Brown, BSc MICT, is Senior Microscopist at CTLGroup. With 28 years of forensic testing and R&D support, he continues his R&D work at CTLGroup by examining clinker quality and raising the bar for producing insightful technical reports.

An experienced concrete petrographer and clinker microscopist, he is also proficient in the use of scanning elec-

tron microscopy and X-ray diffraction. With his wide range of expertise, Mr Brown is well known for his ability to interpret results accurately and effectively to help solve a client's testing needs.

He obtained his BSc Geology and Mineralogy from the University of Aberdeen in 1975 and his MICT (Advanced Concrete Technology Course) from Imperial College in 1991. He is an active member of the Institute of Concrete Technology, Concrete Society, Society of Concrete Petrographers, Cement Chemists Society, and serves on the ASTM C09 committee.

Mr Brown will be making a presentation about current clinker microscopy opportunities at the October 2008 Cement Chemists Society's annual meeting in San Diego, California.

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Despite the usefulness of clinker microscopy, no one technique will be all-encompassing. The parallel use of X-ray diffraction employing Rietveld analysis is now providing information on clinker reactivity—not just quantification of clinker phases. This is primarily achieved via developing procedures that would probe the phase of a given crystallographic feature, such as the unit cell parameters in greater detail. Also micro-diffraction apparatus allows additional information to be obtained from small areas of clinker to supplement microscopy observations. Microscopy and other information can then be used to optimise treatment of raw materials and process conditions to adopt the desired changes at the plant, without unwanted compromise of clinker quality.

CTLGroup, a team of engineers and scientists, performs investigation and analysis of new and existing structures and materials. The firm's expertise enables it to develop the most comprehensive and effective solutions for each client's specific needs.

Microscopical examination of cement, clinker, and cement-based materials has encompassed specialised testing; condition assessment; documenting causes of deterioration and materials distress; and constituent analysis.

CTLGroup Laboratory Accreditations include: ISO/IEC 17025:2005 Accredited (International Accreditation Service) ; Cement and Concrete Reference Laboratory (CCRL) Inspection Program; AASHTO Certificate 202CR; U. S. Army Corp of Engineers Approved Test Lab; Miami-Dade County Approved Test Lab. Nuclear Quality Assurance includes 10CRF50 Appendix B approved and 10CRF830.120 approved. CTLGroup also has LEED accredited professionals on staff.

Left: A Derek R Brown in the CTLGroup laboratories in Skokie, Illinois, US.

