ICMA 2004 CONFERENCE PRESENTATION

Continuous XRD for Real Time Plant and Product Control

Summary

Control of the cement manufacturing process takes place on three levels. Process Control, for controlling temperatures, pressures, flows etc, Quality Control for controlling the material and product behaviour, and Cost Control for economic performance.

Process control is very much on-line and real time control, with automated control loops and sophisticated expert systems while traditional Quality Control is still largely off line and laboratory based, aimed at monitoring quality rather than controlling it.

Quality Control is rapidly becoming integrated with process control with the advent of new on-line analyzers such as belt analyzers allowing control of raw material proportioning by automated control loop and on-line particle size analysis for example.

The missing link in on-line quality control has been a direct measure of cement and clinker quality, and this is now possible with XRD techniques. All cement chemists know that the performance of the plant and the product is determined by the mineralogy of materials and not the chemical composition. However, the industry has had to compromise with chemical analyses because no viable means for continuous mineralogical analysis has previously been available.

FCT has developed completely new technology that allows the mineralogy of a moving material stream to be monitored on-line in real time. This breakthrough now provides the basis for monitoring and controlling clinker and cement quality on-line and as an integral part of the process control system. This technology will benefit the customer with consistent quality (setting times and strength performance), the plant manager with reduced costs through better efficiency and reduced variability and the plant operator by providing continuous on-line monitoring and trended information for automatic quality control of cement product.

Continuous XRD for Real Time Plant and Product Control

Introduction

Cement manufacture is a capital and energy intensive process. Any additional production that can be squeezed out from an existing capital investment, or any reduction in specific energy consumption has a profound positive effect on the plant's bottom line profit. This Plant Optimization is a continuous struggle for plant management.

Measurement, analysis and control form the basis of plant optimization. In this, there are three control/optimization systems run concurrently by plant management – Process Equipment Control, Product Quality Control and Cost Control.

Process Equipment Control Systems have become quite sophisticated now, with artificial intelligence and fuzzy logic systems operating the plant automatically. Such automated systems are capable of running the plant closer to an optimized state on average over time. The investment for these has been based on just a few percentage points improvements in efficiency that already justifies significant capital investment.

Quality control has been largely an off-line control system based on taking plant samples back to a laboratory for analysis followed by some manual decision to make a plant change toward optimization. It is clear how deficient this traditional approach is for the large continuous plants of today. There is always a question mark over samples taken, the analysis itself can take hours or even a month in the case of strength tests, and the plant correction is based on this old test information which may no longer be relevant.

In recent years, there has been an effort to address this. One direction has been through automated laboratories – these tend to provide faster analysis in some things, but the inherent problems of sample integrity and untimely information persist.

Another direction has been on-line analysis systems, such as the on-belt raw material analyzers and the in line particle size analyzers that have been available in recent years. This is clearly the technically better solution in principle, but so far, considerable laboratory testing has still been required to supplement the on-line analyses available.

A new Continuous On-Line XRD analyser has now been developed that bridges many of the previous gaps, such that complete on-line automated analysis and control of product quality is now a reality, delivering considerable dollar benefits arising from better control. This paper is a status report on this exciting new development.

The third control regime – cost control – is still a very much off-line system, but with the ability now to monitor and control quality, this opens the door to integrate process control, quality control and cost control into a single control/optimization system.

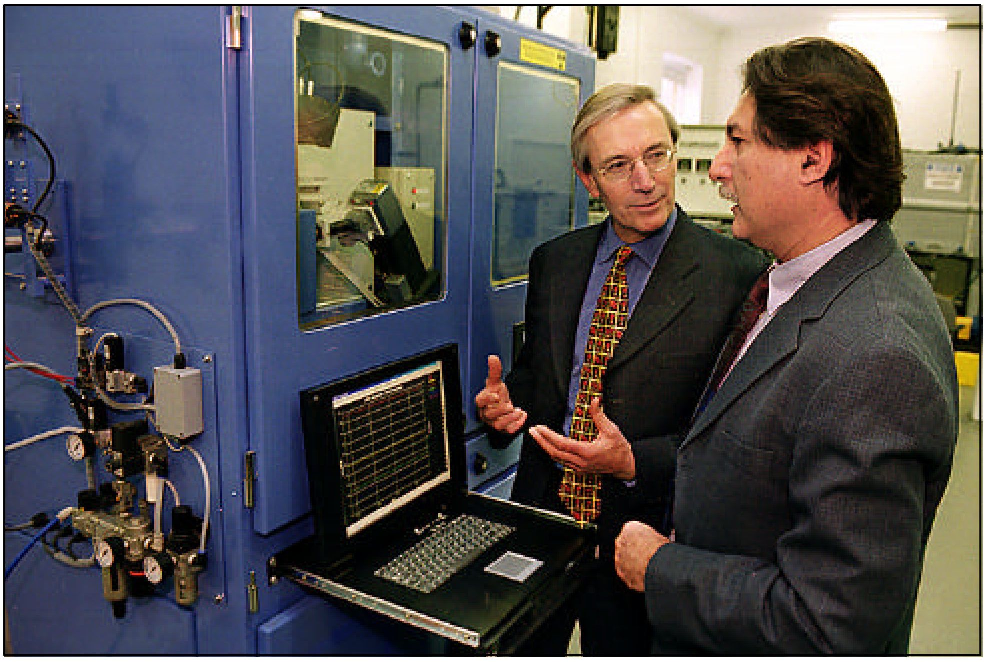

Figure 1 Continuous Real Time XRD machine

Why X-Ray Diffraction?

As experienced cement producers know, the performance of the plant and the product is governed mainly by mineralogy of the materials, and not their chemical composition. Svinning et al (2), (8), (9), have sought to correlate process operation, clinker and cement microstructure and product performance with good success. The use of microscopic techniques by renowned practitioners such as Dr Donald Campbell and Dr Ono has done much to understand the effects of cement plant process parameters on the mineralogy of cement and the impact on the products performance. It is clear that mineralogy is a key parameter for monitoring and control of cement production.

However, mineralogy has been rarely measured, except in research environments. It has hitherto been a relatively tedious analysis, and too slow to be useful for real time plant control. Defacto techniques, such as the Bogue equations, have been developed to try to estimate some aspects of mineral composition. This has been helpful, but it is well known that these theoretical calculations are often inaccurate and misleading (13).

X-Ray Diffraction is an analytical technique developed early last century, but not used widely in cement until very recently. Recent developments with Rietveld quantitative analysis software and faster computing power have seen a dramatic rise in just the last 5 years of publications referring to XRD application in cement manufacture. There is a growing awareness of the potential of XRD to give better and more relevant information on which to control cement manufacture.

It is now relatively easy to get accurate direct measurement of mineralogical composition of powder materials using XRD equipped with Quantitative Rietveld analysis software (10).

This Mineral Composition is the information of most value to the cement plant operator. It will characterize grindability and burnability of raw materials, clinker quality (strength potential and grindability) and cement strength development and setting times for example (7), (9), (13).

This paper introduces a new technology developed for continuous mineralogical analysis of a moving stream of material, opening the door for the first time to automated control action that includes mineralogy as one of the key control parameters.

Why Continuous XRD?

Cement manufacture is a continuous process, with the material flowing continuously through. It surely makes sense to analyze moving streams continuously, providing real time information to use for corrective action as required on a continuous basis.

The traditional batch sampling and batch testing techniques are a compromise, but have established in the industry because there has been no other options.

Likewise, chemical analysis of the material streams is not the most appropriate information on which to base plant control decisions in most cases, but again the industry has had to accept this because there have been no other feasible options.
This has now all changed, and continuous on-line real time mineralogical analysis of material streams is available. This technological breakthrough will have a profound effect on future monitoring and control of the cement manufacturing process.

In comparison with any system relying on taking a sample back to a laboratory based analytical point, the advantages of continuous real time analyses are:

- The Continuous XRD analyser will analyze as much material in 10 minutes as a laboratory based analyser will in 24 hours or more.

- Sampling issues are greatly reduced
- The analytical information is displayed as trends, allowing easy identification of any outlying analyses
- Direct control action can be initiated from the smoothed analysis trend

This opens the door now to control loops based on mineral analyses, with the inherent cost savings through reduced product variability.

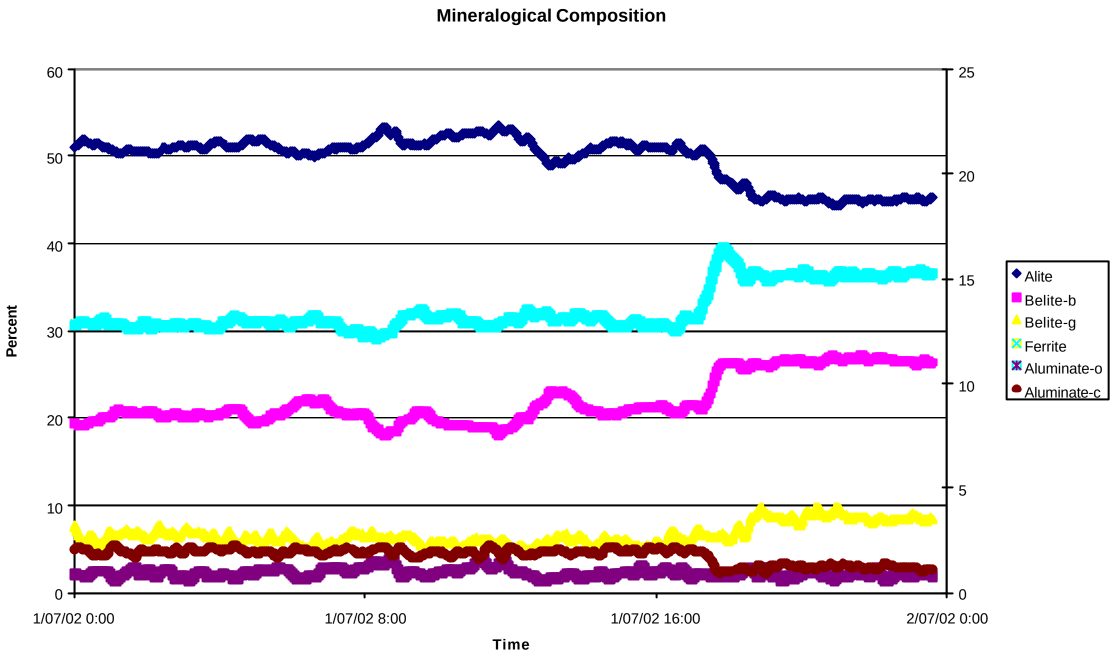

Figure 2 Continuous XRD Trend of clinker minerals in real time

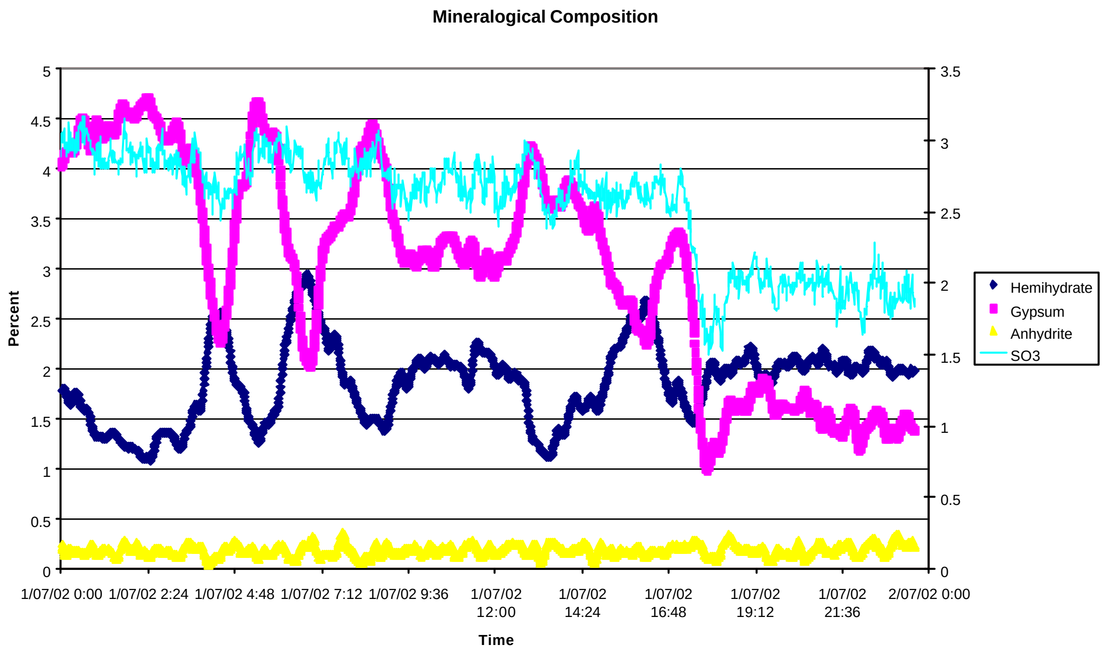

Figure 3 Continuous XRD trend of Gypsum hydration states ex cement mill and calculated SO3

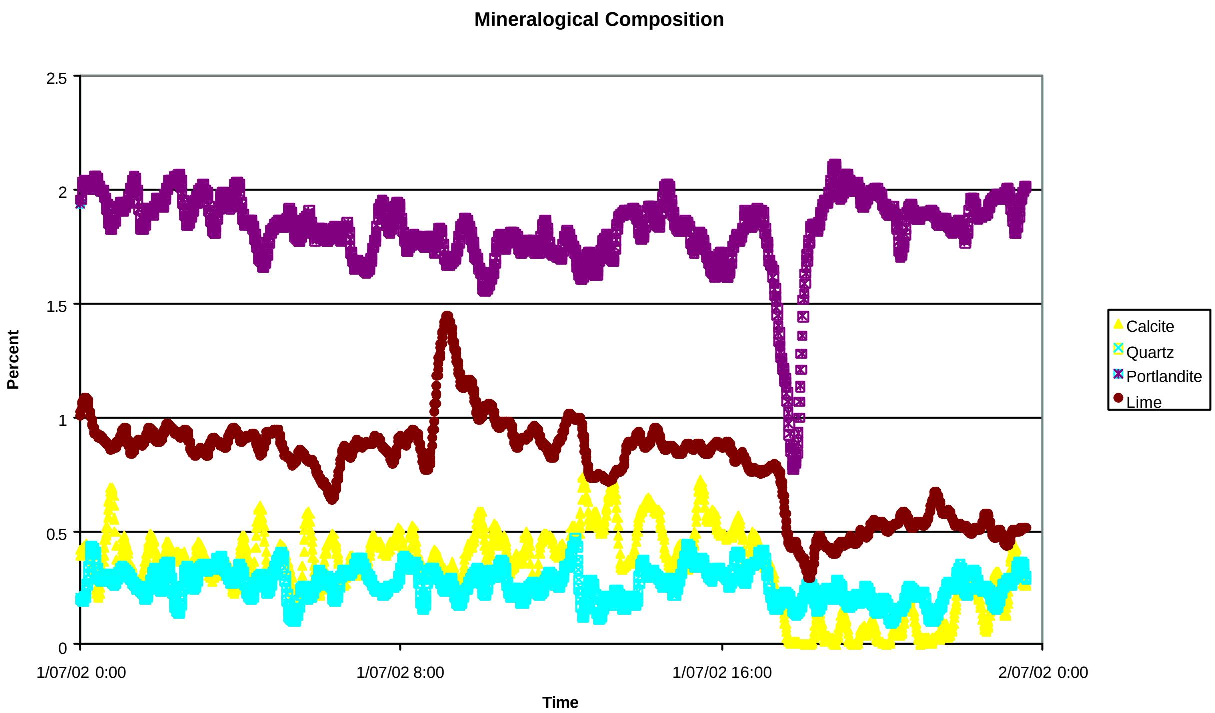

Figure 4 Continuous XRD trends of minor cement minerals

Control Applications based On Mineralogy

Continuous XRD machines can be located on clinker or cement streams.

Clinker

The clinker minerals are formed in the heat treatment through the kiln system. Given the same feed chemical composition, the range of mineral compositions that may eventuate in the clinker produced is almost limitless.

There are various forms of the clinker minerals, such as alpha, beta or gamma forms of calcium silicates and ortho-rhombic and cubic forms of calcium aluminates. The development of these depends on the kiln heat treatment of the material, and the proportions will govern the clinker quality. (4)

Continuous XRD can provide vital information about what is happening in the kiln. The clinker free lime, forms and proportions of calcium silicates, aluminates and ferro –aluminates, sulfates etc can be used to adjust burner (fuel rate, primary air, position, set up etc), cooler operation (airflows, grate speed) and kiln operation (calcination degree, kiln speed). The tuning process will aim to produce the best clinker at minimum fuel usage requiring minimum grinding energy to produce the target cement quality.

Savings in kiln fuel usage and/or cement grinding power would be the anticipated benefits from optimization of the clinker quality. The free lime can easily be monitored and controlled (as is normally done by batch techniques now), but there are potentially larger benefits in controlling the clinker quality (mineralogical composition) produced.

Cement

Cement is the final product going to the customer, and the objective is to produce the most consistent product possible on target at the minimum cost.

The cement mineralogy determines its performance, such as strength development and setting times. Correlations between cement properties, mineralogy and particle sizing can be developed using statistical means, such that cement strengths and setting times can be predicted with high confidence (13, (8), (9), (7).

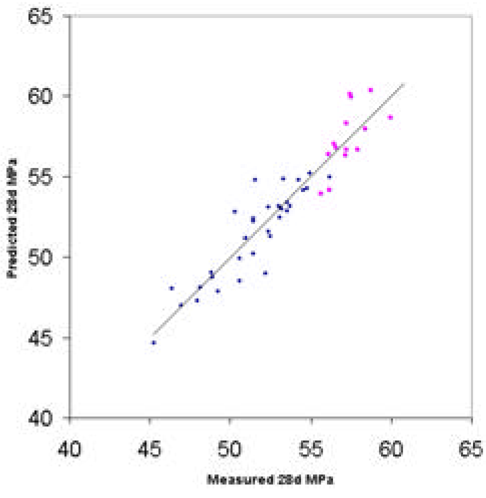

Figure 5 Predicted v Actual 28 day strength using XRD and PSD information.

Strengths can then be controlled by adjusting mill feed rate (fineness) or feed proportions and setting times by controlling gypsum dehydration for example. A tighter control on strengths (less overgrinding) will result in less kwh/t for cement milling and higher production tonnage.

The marketing value of better cement quality can be variable, as can be the insurance cost for product liability. In most countries though, this would be a significant factor.

Case Study Installation

A continuous in plant real time XRD analyzer was installed and commissioned at the Leamington plant of Ash Grove Cement Company in Utah at the end of 2001 monitoring the cement product from a 3000 HP cement mill (12).

Figure 6 In plant Continuous XRD installed below cement transport conveyor in Utah

The Continuous XRD analyzer was installed in order to gain the following benefits:

- Learn more about the product the plant produces
- Increase plant efficiency by reducing energy usage and increasing production
- Reduce Testing costs
- Develop Predictive models for strength and setting times and instigate better control of these

The steps of the process that is underway are:
1. Install and commission Continuous analyzer to achieve reliability
2. Confirm analytical integrity of information
3. Control Cement mill weighfeeders with automated loops
4. Develop strength and setting time predictive models
5. Optimize kiln operation based on mineralogical information learned
6. Develop control strategies for strength and setting times and implement manually
7. Implement automated control of product quality (strength/setting times etc)

The status of this program is as follows.

1 Installation and Commissioning

The analyzer was commissioned and producing complete mineral analysis of the cement product within 5 days of delivery to site. The information is relayed to plant control room, management and laboratory screens via the plant PLC system. The full mineral analysis of cement being produced is available to all in real time.

The machine has operated continuously since commissioning with only minor early commissioning problems. Following a mill outage in Feb/March 2002, and a short re-commissioning, the machine availability has been over 98% of mill run time since mid-April 2002.

The information produced includes:

- Trends of all clinker minerals updated every minute.
- Trends of Gypsum hydration states updated every minute
- Trends of chemical composition of cement calculated from the mineralogical composition
- Mineralogical composition in tabular form
- Chemical composition in tabular form

2 Analytical Performance

The integrity of the analytical information has been cross-checked in several ways, and found to be sound. For example, the calculated chemical composition has been checked against XRF analyses with reasonable agreement, while known percentages of known composition have also been added to clinker and the analysis has again matched the known values closely.

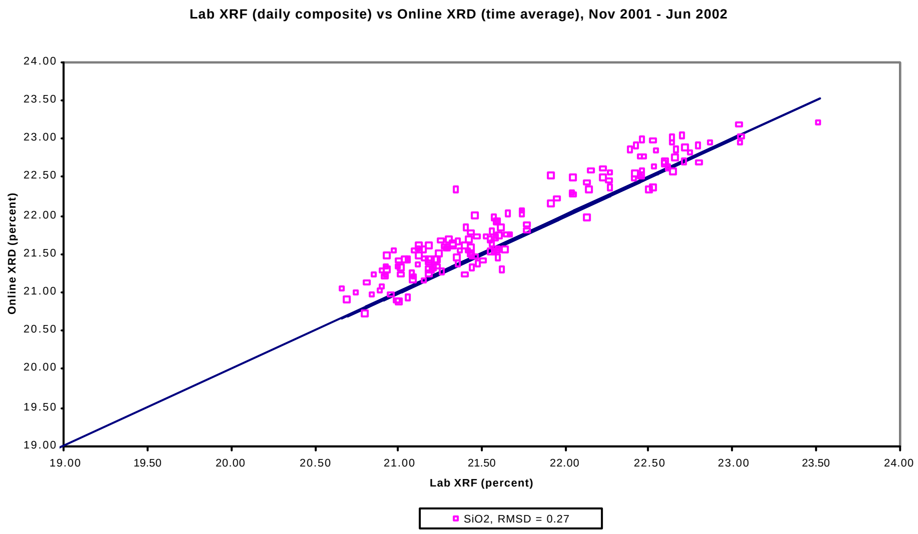

Figure 7 Calculated XRD v XRF Analysis for SiO2

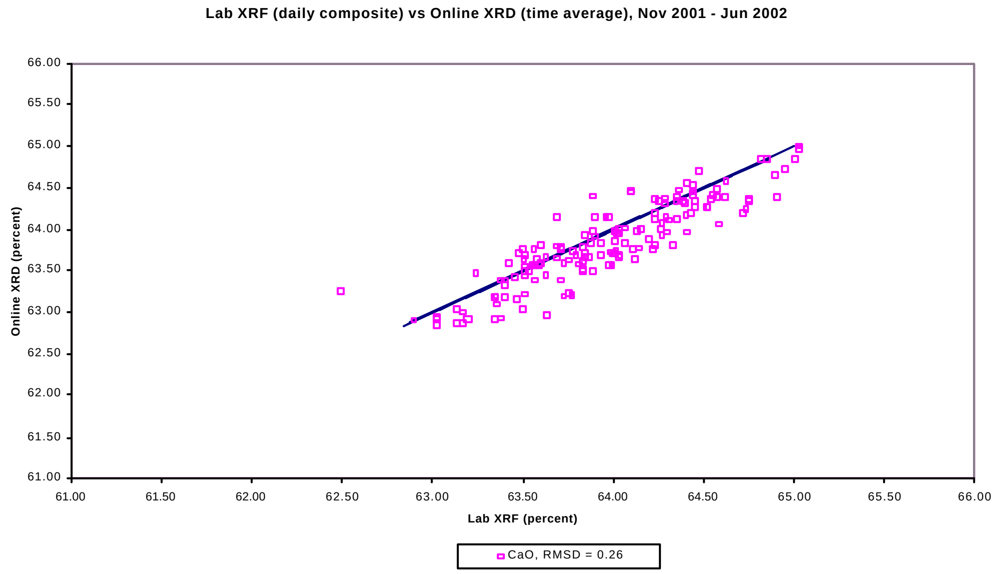

Figure 8 Calculated XRD v XRF Analysis for CaO

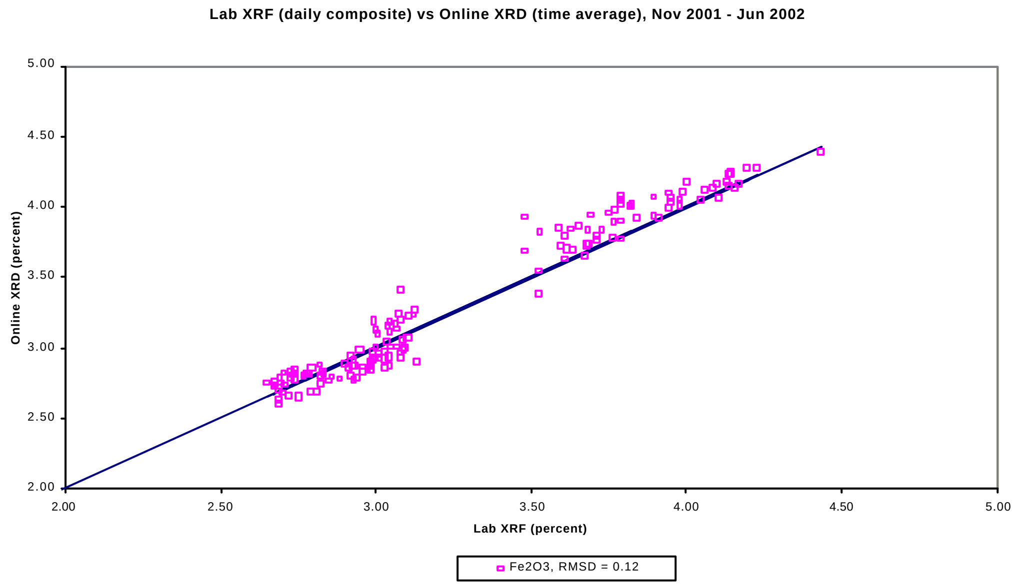

Figure 9 Calculated XRD v XRF Analysis for Fe2O3

	SiO_2	Al_2O_3	Fe_2O_3	CaO	SO_3
Mean	21.90	4.16	3.57	64.24	2.45
RMSD	0.31	0.18	0.22	0.53	0.15
% Relative Difference	1.43	4.31	6.16	0.82	6
Guarantee	5	10	10	5	N A

Table 1 RMSD between averaged continuous XRD analyzer results and the routine four hourly laboratory XRF analyses.

It should be noted that no bias correction or re-calibration has been implemented on the XRD analyses in the 6 months of operation to date. The machine produces an extremely stable result with excellent repeatability. Better agreement still could be achieved with periodic checks and bias corrections against XRF analyses, as is common with PGNAA technologies for example.

3 Cement Mill Weigh Feeder Control

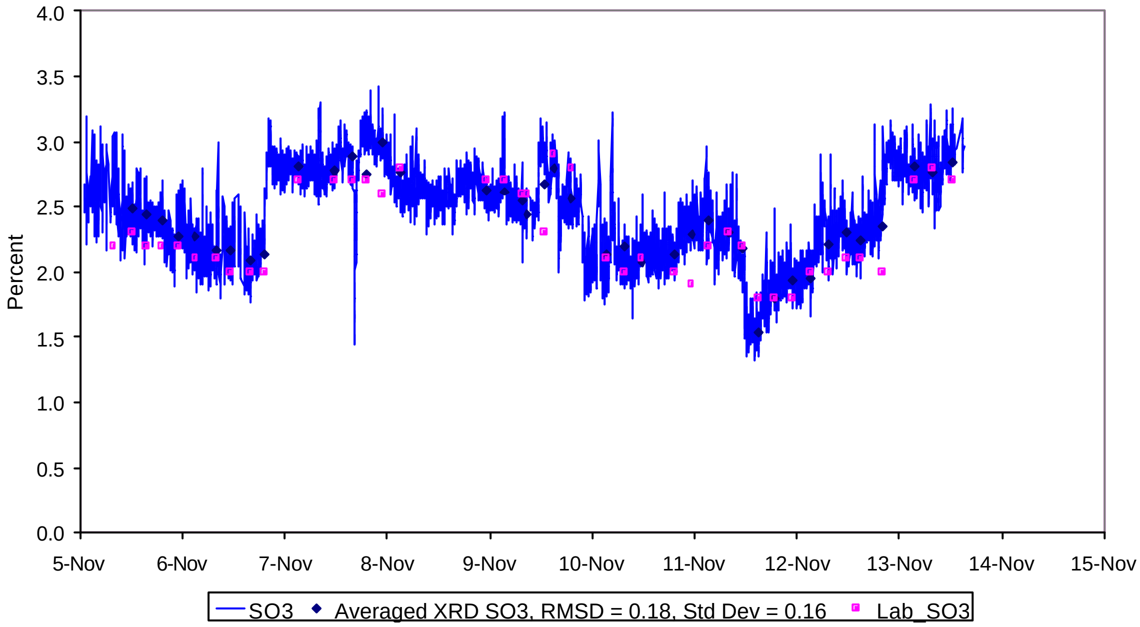

Figure 10 Calculated XRD v XRF analysis of SO3

After proving the analytical performance and equipment reliability, the instrument data is now being used to automatically control the gypsum weigh-feeder. No further SO3 testing is being done on shift.

Although the Leamington plant does not use limestone as is common in other countries, earlier work in Australia has shown that accurate analysis of limestone percentages in cement product are easily provided in real time.

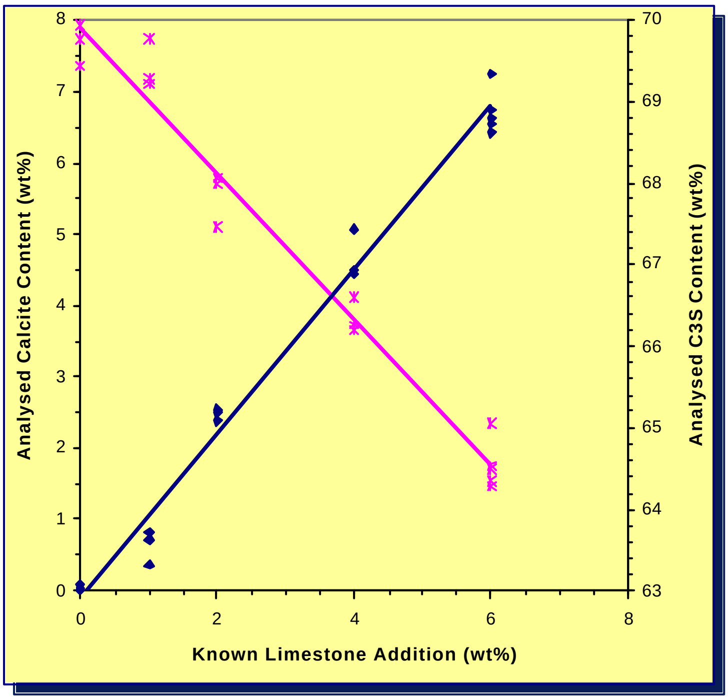

Figure 11 XRD Analysis of known limestone additions to cement (with corresponding C3S reduction)

4 Development of Strength and Setting time predictive models

To date, there has been some correlation attempted between laboratory run of the mill physical test data, mineralogical composition and particle sizing. The Leamington plant also has an on-line particle size analyser installed on the same cement stream.

With limited data, the correlations established already look promising, despite the inaccuracies associated with the basic strength test procedure. Included graphs indicate that the movement in strength results is being tracked very well.

Figure 5 is a correlation established in earlier work using a proto-type continuous XRD analyser on a cement mill in Australia. In this case, the physical tests were carried out in quadruplicate in order to narrow the test error. The agreement reached here between predicted and actual strength was exceptional, showing this is a feasible approach.

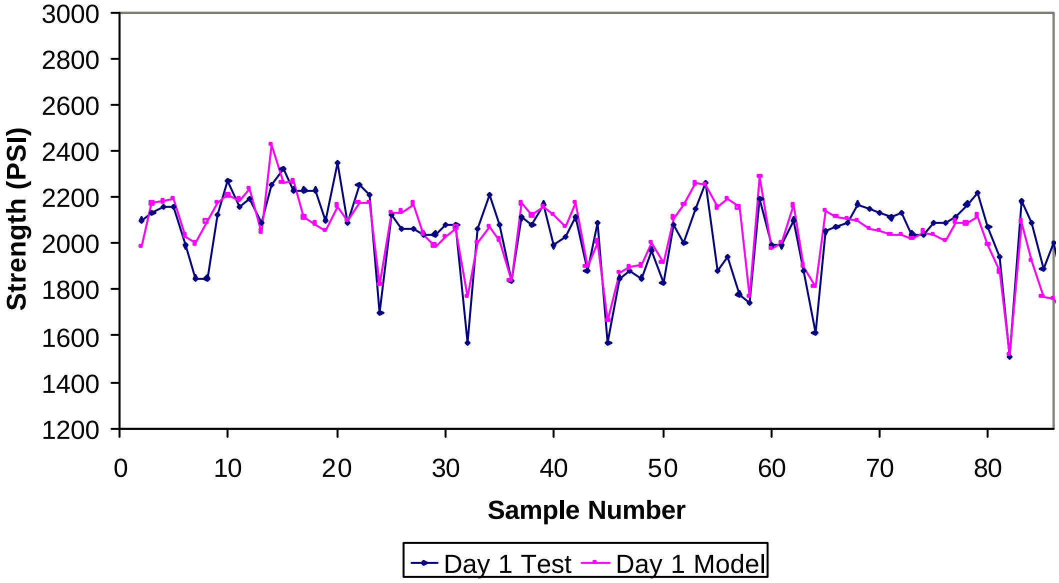

Figure 12 Predicted v Actual 1 day cement strength correlation using XRD and PSD

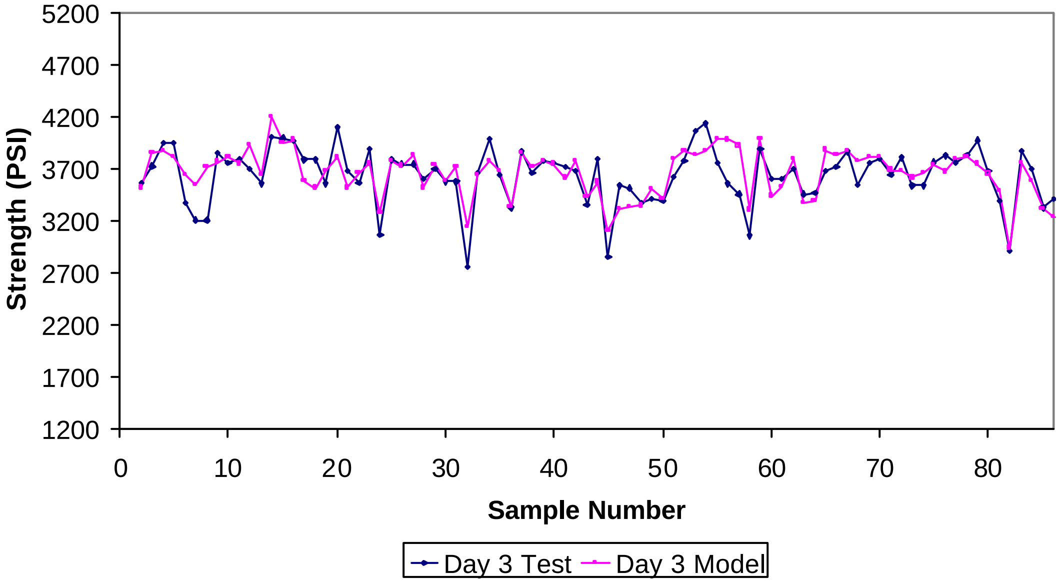

Figure 13 Predicted v Actual 3 Day cement strength using XRD and PSD

One of the findings to date on both the Australian and American Continuous XRD installations is that gypsum de-hydration varies enormously even when cement mill exit temperatures are constant. This depends on clinker temperatures in particular that change the mill internal temperature profile even if the exit is controlled by water sprays. Varying gypsum hydration states will affect setting times and strength development.

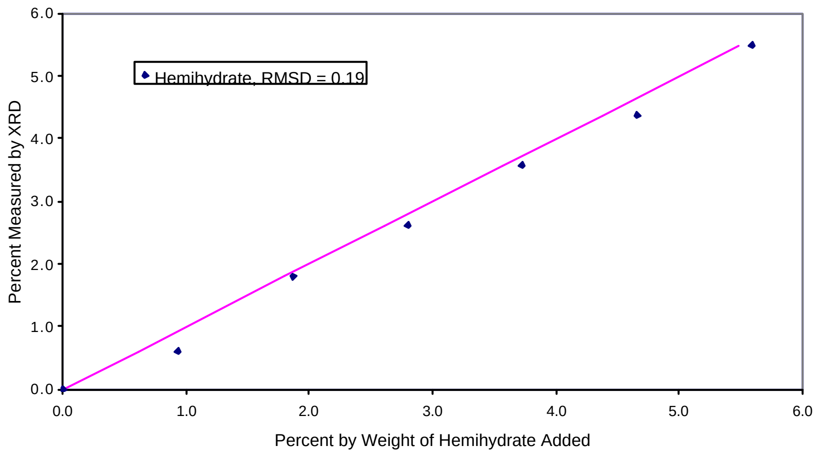

Figure 14 Weighed (known) gypsum hemihydrate addition to clinker compared with Continuous XRD analysis

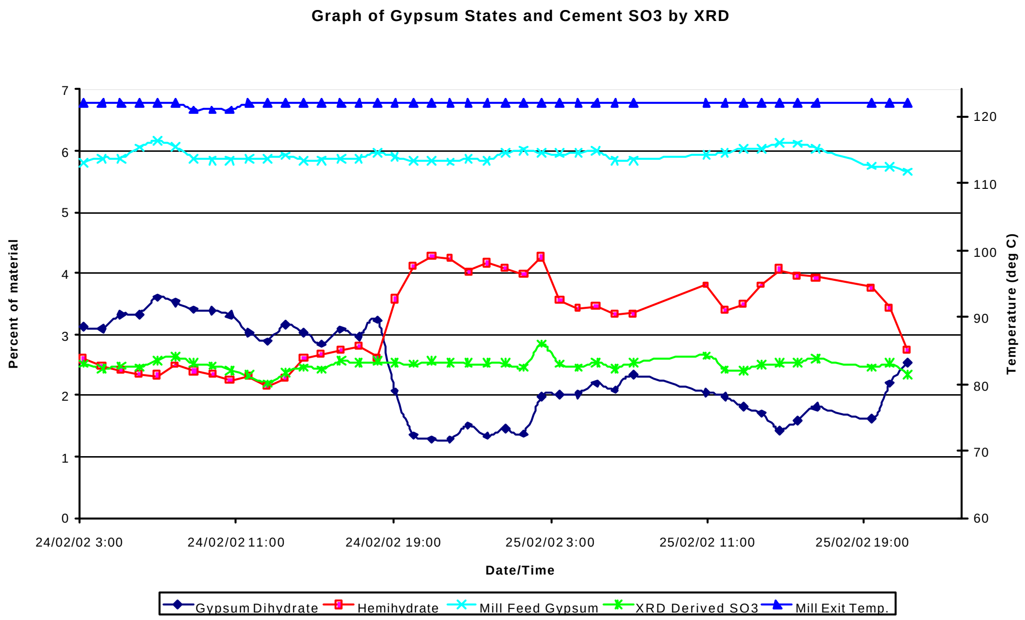

Figure 15 Continuous XRD recording of cement product gypsum states over a 48 hour period, during which cement mill exit temperature did not change but clinker feed temperature did change.

The Leamington plant has begun a program of manually controlling to a target gypsum hemihydrate level in order to get more consistent setting times. The next stage of implementation will involve setting time prediction and control directly. As for the strength prediction, the setting time prediction is also showing good agreement with later results from laboratory testing.

Kiln variables to be optimized include burner set up (heat flux, flame shape and length), burner insertion and angle, kiln rotational speed, excess air levels, free lime targets, degree of calcination ex the calciner, clinker cooler set up, waste fuel usage etc.

A program to address the issue of kiln optimization is the next stage being developed between the Leamington plant and FCT.

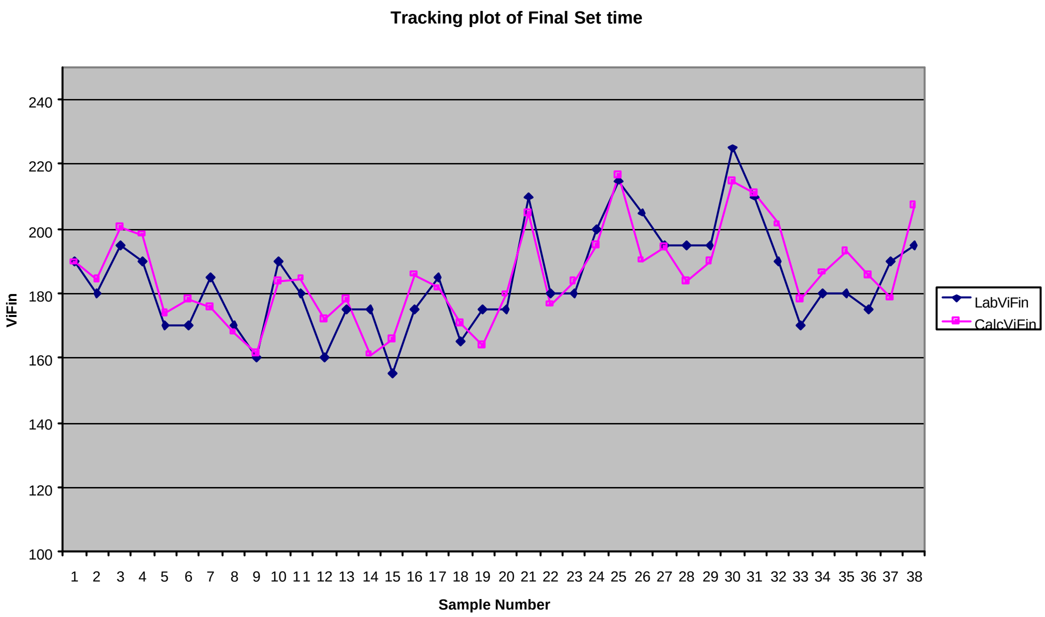

Figure 16 Predicted v Actual setting times, using predictive models based on Continuous XRD analysis.

5 Kiln Optimization

The continuous XRD installed at the Leamington plant has indicated some interesting issues with cement mineralogy. For example, significant levels of gamma C2S have been recorded frequently, indicating some slow clinker cooling. The next stage will be to determine what kiln operating condition is responsible for this and take corrective action.

Another point of interest is that the proportions of ortho-rhombic and cubic C3A are changing too. The significance of this or the cause are yet to be established, but are likely to lead to some improved operation and perhaps more consistent setting times.

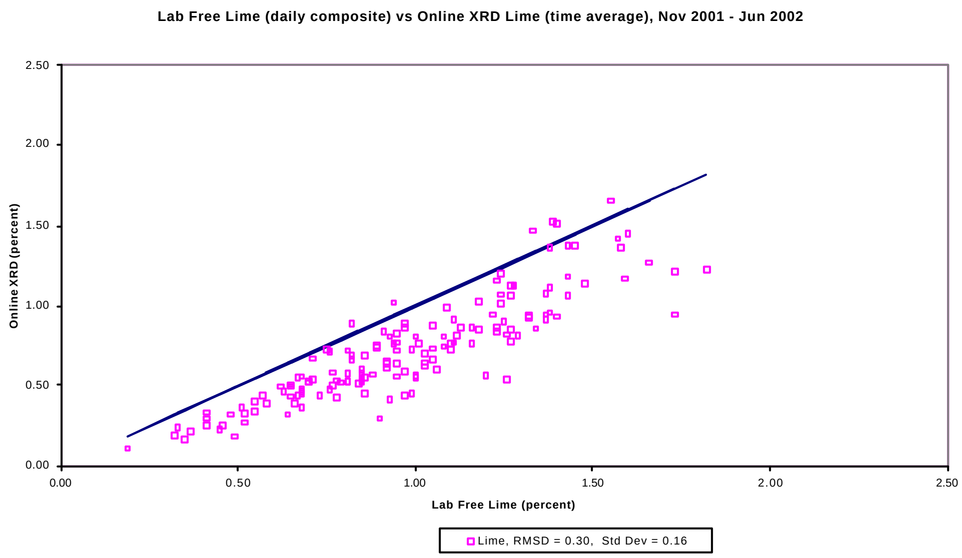

Figure 17 Continuous XRD monitoring free lime.

6 Strength and Setting time control Strategies

The control of vital cement performance parameters will arise from the predictive models being developed. This will be in the final stages of the work program, but it is anticipated that cement quality (strength and setting times for example) can be controlled to within tighter limits through changes to cement mill feed rate, water spray addition, mill feed proportions, separator operation etc.

It should be noted that the Continuous XRD can detect weathered clinker or mill spray malfunction through the lime (portlandite) present. Compensating action can be taken as required.

The benefit to cement milling is anticipated to be additional production at less kwh/t, because of reduced over-grinding. With less variability, and the ability to predict strength for example, feed rate can be increased when the cement strength is predicted to be above the target.

The more uniform product quality will be a bonus that may have significant value in some markets.

Investment Payback

The investment in any continuous on-line analyzer is significant, and needs to have sufficient return to justify the purchase.

In clinker and cement applications, the benefits of better process control will materialize through increased production, reduced specific energy use, reduced testing costs and improved market position. Continuous XRD monitoring of clinker and cement provides valuable information on product variability and predicted behavior, allowing the process to be tuned to provide the required product quality at minimum cost.

When limestone addition is being used, the XRD is an ideal instrument for monitoring and controlling the percentage limestone added. This ensures permitted levels are not exceeded, while maximizing the use of limestone in the cement product. The savings in clinker alone by better control of this can provide a justification for the on-stream XRD analyzer.

Using strength prediction models based on the cement mineralogy and particle sizing of the cement product, cement mill throughput can be optimized by reducing over grinding, again leading to reductions in kwh/t and increased production.

In clinker analysis, the ability to monitor and directly control free lime provides an easy justification with fuel savings and improved clinker quality.

Other additions to the payback equation can include marketing advantages, reduced plant testing and optimization of raw mix design and kiln and mill operation.

The payback will depend on each plants specific issues and the production tonnage. However, small efficiency improvements and reduced shift testing can have payback in the order of months for investment in Continuous real time monitoring of mineralogy by XRD and plant control arising from this.

Conclusion

In the continual drive to improve cement manufacturing efficiency, better monitoring and control is a key strategy for optimization.

The monitoring and control of the material flowing through the plant has not been adequately addressed in the past because of technology deficiencies.

A new piece of equipment has now been developed and proven in service that gives a cement operator vital information and insight in real time into the quality of product during the various processing stages of cement production. This equipment, along with other on-line real time measurement systems, now allows continuous on-line real time and automated product quality control.

Just a small improvement in production efficiency has justified significant expenditure for the industry in technologies such as automated fuzzy logic systems or on-belt raw material analyzers.

This new technology can be expected to yield at least comparable benefits, leading to attractive payback time in most cases expected to be less than 12 months.

References:

(1) Madsen I.C. and Scarlett, N.V.Y. Cement Quantitative Phase Analysis of Portland Cement Clinker – chapter 16 in Industrial Applications of X-Ray Diffraction, Marcel Dekker Inc (2000), ISBN 0-8247-1992-1

(2) Ketil Svinning, Sigrun K Bremseth and Harald Justines. X-Ray Diffraction studies on variations in microstructure in Portland Cement Clinker correlated to variations in production conditions in the kiln. World Cement, October 1996.

(3) Manias C, Retallack D, Madsen I. XRD for On-Line Analysis and Control, World Cement February 2000.

(4) Fullman T. , Walenta G. , Pollman H. , Giminez M. , Lauzon C., Hagopian-Babikian S. , Dalrymple, T. , Noon,P. Quantitative Rietveld Analysis of Portland Cement Clinkers and Portland Cements using TOPAS Software – Application as a Method for Automated Quality and Process Control in Industrial Production – Part 1.

(5) Schmidt, Rainer and Kern, Arnt Quantitative XRD Phase Analysis, World Cement, Feb 2001

(6) Manias C, Retallack D, Madsen I. Plant Optimisation and Control using continuous on-line XRD for Mineral Phase Analysis. ZKG March 2001

(7) Goswami G and Panda J.D.. "Application of XRD in a rapid quality control system of cement" Powder Diffraction 14 (2) June 1999

(8) Svinning K. , Justines H. , Viggh E. , Bremseth S. , Johansson S. , " Examination of clinkers from four Scandinavian plants with respect to microstructure and cement properties." Source unknown.

(9) Svinning K. and Bremseth S. "The influence of microstructure of clinker and cement on setting time and strength development until 28 days." Proceedings of the 18th International course on cement microscopy, Houston 1996.

(10) Bellotto M and Signes-Frehel M. "The role of Powder Diffraction in the Cement Industry: Recent Advances and Future Developments" Materials Science Forum Vols. 278 – 281 (1998) pp 846 - 851

(11) Svinning K., Ingeroyen, O. and Dalsveen K. "Optimization of a response variable y constrained by principal directions of variations in the observation X-matrix" Journal of Chemometrics 2000, 14: 699 – 709.

(12) Godek, A., Crutchfield, D., Blackham, F., Storer, P., and Manias, C. "Pro-Active not Re-Active" World Cement, Feb 2002.

(13) Neubauer J and Pollmann H "Rietveld Calculation – A High Performance Instrument in Automatic Quality Control Systems of Clinker and Cement Production. International Conference on Cement Microscopy, 1997.