Real Time Mineralogical Analysis for Automated Control and Process Optimisation
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Introduction:

Process Control of operating plant is based on gathering relevant information from the plant in a timely fashion and making decisions on adjusting operating parameters based on this information. In order for this to be effective,

- The information must be relevant and linkage clear to the parameter to be controlled
- The information must be true, correct and reliable
- The information must be timely enough to be applicable to the now state of the plant

In many industrial plants, the above requirements do not hold true, and operators are compromised in being able to operate plant in the most effective way. For example, in many processes, the mineralogy of material streams is a key piece of information, yet this has been difficult to get in a timely manner. This paper deals with a new development which now makes this possible.

X-Ray Diffraction

X-ray diffraction (XRD) analysis of poly-crystalline materials has been available since its development in 1916. The Bragg Father and Son team from Adelaide University won the Nobel prize in Physics in 1915 for their work on Diffraction of X-Ray Diffraction with Crystals, and Bragg’s law, \( n\lambda = 2d \sin \theta \), is the underlying formula applied to the analysis methodology. XRD is the technique of directing a stream of monochromatic X-rays at a surface and collecting the resulting pattern diffracted from the crystal structure of each phase in the material being analysed.

The technique has the unique ability to provide identification and quantification of the crystalline components in a sample of interest, such as cement, lime, alumina or any mineral mixture. In many mineral processing plants, the mineralogical information is key to the performance/quality of product and the performance/efficiency of the plant, so the analysis technique can meet the first criteria of providing relevant information for control of the process.
In order to address the other two criteria for process control – reliable, true and accurate information, as well as information in a time frame relevant for process control, FCT has developed COSMA (Continuous On-Stream Mineral Analyser) in conjunction with leading scientists from CSIRO. Using XRD and Rietveldt techniques, COSMA produces real time reliable analysis of the complete mineral content of process powder streams. The continuous real time nature of the analysis makes it suitable as the primary sensor for automated control of process plant to improve both quality of product and efficiency of operation.

There are very significant differences between COSMA and conventional XRD analysers hitherto available. Firstly, XRD analysers thus far have used a scanning goniometer to sequentially collect the diffracted X-Rays and build the diffraction pattern over time. The process involves preparation of a small sample that is irradiated to produce the diffraction field that is analysed over some time (5 – 30 minutes typically) before the analysis result is generated. They are batch analysers taking a finite time to analyse a small sample of a few mg to represent the bulk material. Often, by the time a sample is taken, prepared, analysed and information fed back to the operator, it is of historical interest only as the process may well have changed substantially from when the sample was taken. So the criteria about timeliness of information for control action is under strain already.
COSMA is designed to operate alongside a process stream, taking continuous sample to flow through the machine, and provide an analysis of that moving powder stream virtually continuously, and trending the data with updates every minute or less. COSMA uses a fixed curve detector that is position sensitive over the full 120 degree arc. This detector captures the entire XRD pattern all of the time without any need to scan. Apart from the obvious advantage that a complete pattern is collected all of the time, there are no moving parts that can give reliability concerns.

COSMA therefore provides continuous analysis at the process stream in almost real time, thereby facilitating automatic control action directly from the analysis.

As a second point of difference, traditional XRD analysers require a tablet of a few grams to be prepared for analysis that represents the entire production stream. With such a small sample, it is critical that the sample taken represents the entire stream it is taken from, that the tablet preparation is exacting with no contamination and the analysis is correct. This is not so easy when only a few grams of sample per day might be analysed.

COSMA takes a continuous stream of around 300 gm/minute, or up to 500 kg/day of material to analyse. The amount of sample analysed makes it easier to ensure the analysed material is representative of the whole stream. The equipment is fully automated, so there is also less possibility for errors to occur. The sample preparation method for COSMA is also less likely to lead to problems of preferred orientation of crystals or of material changing through laboratory fine grinding of sample.
The criteria of a more reliable, true and accurate information on which to base control action is therefore addressed.

This paper will use the cement production process to demonstrate the application of XRD to optimization of mineral processing, but it is likely that this same equipment will generate considerable benefits for many other mineral processing industries.

To this point, XRD has had little use in the cement industry at large except for some investigative and research activities. One of the difficulties has been the analysis of the complicated diffraction patterns due to the high degree of overlap/interference between the diffraction patterns of the clinker minerals. With development of more powerful computers and increased sophistication of software, these difficulties are being overcome and its relevance to the industry is fast gaining acceptance.

In summary, the newly developed and patented instrument utilises a unique combination of:

(i) simultaneous, rather than sequential, data collection through the use of a unique patented position sensitive curved X-ray detector. A further advantage is no moving parts.

(ii) whole-pattern analysis using the Rietveld method, providing a complete analysis of the spectra every minute, and

(iii) a sample preparation and presentation system that provides for a continuous flow of material through the analyzer such that a very large number of crystals pass the analysis region compared to the laboratory based units.

This configuration allows a whole pattern to be collected all of the time, so that as the sample is moving continuously past the X-Ray beam, a complete pattern for analysis is produced every instant. In practice, the pattern produced is averaged every minute to provide a new analysis

This breakthrough in XRD analysis now means that a continuous sample of material can be fed to the instrument from the cement mill or kiln, enabling ‘on-line’, real time control actions to be taken.

Several COSMA units are in operation in the US, Canada, Italy and Australia and have proven reliable in operation and accurate in analysis, producing data for real time control and optimization of the cement manufacturing process.
The Cement Process:

Fig 3 COSMA Analysis Equipment

Fig 4 The Cement Manufacturing Process.

- Direct Mineral Phase Analysis
- Continuous Real Time Analysis
- In Plant On-Stream Location
- Linked to Plant PLC
- Data Trending
- Elemental Analysis also
The cement manufacturing process consists of 5 principle unit operations. They are:

- **Quarrying** where the main ingredients are limestone (for calcium mainly) and shale (for silica mainly).
- **Blending** of limestone and shale often with small amounts of additional materials such as bauxite and iron ore to fine tune the chemical composition of the raw materials mix.
- **Raw Materials Milling**, to produce particles fine enough to promote the solid-solid reactions that need to take place in the kiln.
- **Kiln Burning** where the raw materials at temperatures of around 1400 °C react to form calcium silicates, aluminates and ferro aluminates from the solid-solid reactions in a liquid phase of alumina and iron compounds.
- **Cement Milling** where the clinker coming from the kiln is ground with additives such as gypsum (and depending on the cement type, limestone, flyash or slag), to produce a fine powder that will react with water in concrete or mortars to produce the matrix bonds that are responsible for the strength of concrete and mortar.

In the cement manufacturing process, it is critical to establish the correct chemical composition of feed going into the kiln. The raw material source of calcium, silicon, iron and alumina (main elements of cement and clinker) is not as important as having the precise proportions needed. These precise portions govern primarily the mineralogy of cement clinker produced that in turn determines the product performance.

![Fig 5 Mineralogical Changes with Temperature in a Cement kiln](image-url)
It is worth noting that major mineralogical changes take place in the kiln, where the clinkering reactions take place that will determine the properties and behavior of the cement and concrete eventually produced. The clinker mineralogy will depend on the kiln feed chemical composition, and this is critical, but it will also depend on the type of heat treatment in the kiln, which is just as critical. A real time measurement of clinker mineralogy coming from the kiln provides the information required in a timely manner for automatic kiln control action and production optimization for energy consumption, emissions and product quality. The fuel usage, flame characteristics, and feed chemistry can all be adjusted and controlled to optimum values based on real time mineralogical information provided by COSMA.

**Fig 6** Minerals Typically of interest in the cement industry and continually analysed by COSMA in real time

Figure 6 shows trends from a plant installation of COSMA. In this case, the reactions taking place in the kiln are monitored, with the percentage of free lime (CaO), alite and belite (tricalcium silicate and di-calcium silicate respectively) indicate the kiln feed chemistry as well as the adequacy of the heat treatment in the kiln. In this case, it shows for a period that the clinker reactions did not go to completion as the high free lime values were outside specification, so more heat was required to complete the reactions. The high free lime means that this was wasted potential for producing more calcium silicates that give cement its hydraulic strength. It also means the high free lime could cause cement to expand and produce unsound concrete in the presence of water.

It should be noted here that the continuous information allows trend curves to be produced for each monitored variable, giving an operator a much clearer view of how a process is changing and what control action may be necessary. A spot analysis gives an isolated set of numbers without any context of process state.
The final processing stage of cement milling is also a good application location for COSMA. Various additive materials to clinker in the final cement milling stage alter the final cement mineralogical composition. Continuous monitoring of this also provides optimized use of additives as well as quality prediction parameters to produce the required cement quality at a minimum cost.

The benefit in this situation comes from:

- Better control of proportioning of additive materials to a cement mill, such that the correct quality is reached with the most economical mix of feed materials to the cement mill
- Monitoring and controlling of gypsum dehydration in a cement mill, which has a major impact on performance of the cement setting times
- Real time quality indication of parameters such as strength to allow some control to a set point and reduced over grinding with associated energy savings
Mill exit cement after a power trip, July 28, 2004

Fig 8 Real time monitoring of various cement materials in finished product

Fig 9 Model for Prediction of cement Strength using Mineralogy and PSD
Fig 10 Real time monitoring of gypsum states in finished product

**Site Installation:**

Plant installations can be made very simple and reliable, with few moving parts in the equipment. The equipment has several unique features, including a fixed curved detector for gathering the diffracted X-Ray spectrum, and a unique sample transport and preparation system.

Fig 11 Cement screw sampler and simple feeding arrangement to and from COSMA
In some applications, the material to be analysed will be too coarse for XRD analysis and will need to be ground to a powder. In such cases, a sampling and grinding stage needs to be included to prepare the material before feeding in to the COSMA hopper. From there on, it is the same equipment and system.

1. Clinker sampling point
2. Clinker sampler
3. Crusher
4. RSD
5. Grinder (P50 50 microns)
6. Sample receival hopper
7. Sample return (pneumatic)

Fig 12 Schematic of sampling and milling system for coarse material

The requirements for a system installation and maintenance are not onerous. Typically, an air conditioned room will be required near the plant sample location with a transport system (preferable gravity) to transport material to the COSMA hopper located on the side of the machine. After analysis, COSMA has its own pneumatic pump to return analysed sample continuously back into the process. Small amounts of compressed air, an electrical supply and cooling water for the X-Ray tube are the main requirements to be supplied to the COSMA room. An internet connection is also required for remote monitoring by FCT.
Fig 13 COSMA room located beneath conveyor gallery for analysed material in a United States Plant

Fig 14 COSMA room located near cement conveyors for an Italian Plant
Potential Benefits:

COSMA has been conceived and designed as a plant control tool. The benefits will in a general sense arise from the quality, nature, timeliness and reliability of the information provided on which to base control action. The value of this will be very plant specific, and so needs to be individually assessed for each industry and each plant.

In the cement industry, the following gives an indication of what is possible.

Clinker Applications:

In clinker applications, COSMA looks at the kiln product to determine the mineralogy of clinker which indicates the performance that would be expected from that material. In particular, free lime, (CaO) is a key indicator of the degree to which kiln reactions have progressed. If not gone far enough, the clinker will make expansive and unsound cement with weak strength properties. If the reaction has gone too far, then there is a waste of energy and production rate for little benefit, an increase in clinker hardness that will have downstream detrimental effects, a negative effect on kiln refractory life and excessive NOx emissions.

Typically, the optimum “free lime” in clinker will be about 1.5%, whereas an average production number for kilns is more like 1.0%.

For a typical 1 million tonne per year plant, the following potential benefits can be achieved through continuous and real time analysis of clinker product allowing better control of free lime from an average of 1.0% to 1.5% say.

- A 5% fuel saving resulting in approx $750,000 per year saving (fuel price dependant)
- A 5% increased production, resulting in $2.5 million/year extra profit
- A 3% energy saving at the cement milling stage resulting in $100,000 per year power cost saving
- A 25% reduction in NOx emissions

This is obviously a significant saving.
Cement Mill Applications:

Clinker from the rotary kiln is mixed with gypsum and other additives such as limestone, flyash, slag, pozzolans or kiln dust and ground in a mill to a fineness of around 5% residue on 45 micron.

COSMA located on the finished product stream from a cement mill provides information on finished product quality as well as an analysis of what has gone into the blend.

In particular, a combination of this can allow better control of additive materials to to produce the cement with the most economical combination of materials.

For a 1 million tpa plant for example, up to 1% more additive materials added will save 10,000 tpa of clinker, the most expensive material in the cement mix. This would result in:

- A saving in the order of $350,000 per year substituting cheaper material for cement clinker
- A potential $750,000 per year extra profits if an additional 10,000 tpa cement can be sold.
- Approximately 10,000 tonnes per year saving in CO₂ emissions.

Conclusions:

The new patented COSMA equipment provides a real time continuous mineralogical composition of a moving powder stream. Its use in the cement industry has been demonstrated in practice and reported in this presentation.

Its application other industries is likely to also be straight forward, but the benefits will need to be assessed on an industry by industry and plant by plant basis. Potentially though, there is likely to be substantial efficiencies that can be derived by having accurate, reliable and timely mineralogical information on which to base plant operating decisions in mineral processing plant.
References: