

High-precision automated slag sample preparation for XRF and XRD applications

Analysis of steel slag is a significant requirement in the control of the steelmaking process and to maintain and expand the opportunities for slag reuse. In order to allow fast, highly precise analysis with a high sample throughput rate, thyssenkrupp has developed an automated slag sample preparation line from crushing through to preparation of pressed tablets for analysis by XRF and XRD.

The new system is in full use at the Duisburg-Bruckhausen steel plant where analytical performance has proved as reliable and precise as the benchmark fusion sample preparation method.

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With an annual production of approximately 12Mt of crude steel per year, thyssenkrupp is Germany's largest flat steel manufacturer. The Duisburg-Bruckhausen integrated site comprises iron and steel plants as well as a large number of rolling mills and other processing plants.

The laboratory for chemical process analysis is a key facility for quality control, with some 50,000 steel, iron and granulated BF slag samples analysed per month in order to control all steps of steel production and to supply high quality products to clients. The laboratory is fully automated, running 24/7 all year.

Slag samples are prepared by pre-crushing, grinding to powder and pressing the powder into 51.5mm steel rings to form tablets ready to be analysed by X-ray fluorescence analysis (XRF) or X-ray diffraction (XRD).

When the existing sample preparation was approaching the end of its service life, thyssenkrupp faced the need to replace the current installation and to cover all future needs of a steel plant laboratory. The ideal sample preparation for slag analysis in the steel plant laboratory had to:

- Meet the highest analytical precision, reproducibility and fast sample throughput
- Include reliable magnetic separation to remove all metallic iron (Fe) from the sample
- Withstand the extreme conditions in the steel plant, namely vibrations and highly abrasive materials
- Be ready to be integrated into the existing laboratory automation system
- Guarantee low carry-over contamination between different samples
- Have multiple adjustable parameters to cope with different types of slags analysed in the steel plant's processes
- Fit into the confined space of the existing laboratory

None of the crusher-mill press units on the market were designed to fulfil these requirements, so in seeking an automated solution, thyssenkrupp Steel Europe joined forces with thyssenkrupp Industrial Solutions, a leading partner for the engineering, construction and service of industrial plants and systems. For more than 50 years, this business has been offering innovative and leading laboratory automation products for the supervision of processes in the cement, mining and minerals industries.

AUTOMATED SLAG PREPARATION PROCESS

Today the polab Advanced Preparation Module (APMplus) is the core element of polab laboratory automation solutions so this was the starting point of a project in which an experienced plant manufacturer and an experienced user combined their know-how. They jointly modified the APMplus for use in the steel plant and added selected high-quality components to become the nucleus of a new steel industry sample preparation system. The result is a very compact sample preparation unit that is optimally aligned to the highly demanding steel plant laboratory process. As a special bonus, the limited floor space in the Duisburg laboratory proved to be sufficient for two systems, doubling the sample throughput and safeguarding against failure through having some built-in redundancy.

The two thyssenkrupp business areas designed, constructed and implemented the new sample preparation in close cooperation. The following description refers to the new system as installed at the Duisburg steel plant although other individual combinations are possible. For instance the system is open to every communications interface, can be controlled by any laboratory software, and can either be integrated into a fully automated

laboratory or operated as a stand-alone device in semi-automatic operation.

SYSTEM COMPONENTS

The system includes all process steps, from the receipt of slag sample to the ready-for-analysis pressed tablet. Sample preparation takes about 400 seconds, depending on grinding parameters. The innovative sample preparation method consists of an automated crusher with an in-line magnetic separator, a handling unit, a gravimetric dosing unit, a grinding unit, a second magnetic separator and a tablet press, all in a compact housing (see Figure 1).

It is integrated into the steel plant's existing automated laboratory by an automation interface. Communication takes place via OPC Unified Architecture and enables fully automatic operation. The existing proprietary software in Duisburg remains in use but the system can also be operated manually by a touch panel.

The operator has multiple options to predefine sample preparation parameters for individual materials, eg, primary metallurgical slags, secondary metallurgical slags and blast furnace slags. These include sample size, grinding speed, amount of grinding aid tablets (see later), the pressing force and the intensity of magnetic separation. This variety of adjustable parameters provides the necessary flexibility to handle the various slag types.

The system components are:

Automated crusher The sample material is pre-crushed in an automated crusher. Pre-crushing the material also prevents clogging of the system and a standard laboratory jaw crusher has been upgraded to meet the demands of the steel industry. Its robust construction is able to cope with unwanted materials that get into the process. The breaking jaws are made of highly wear resistant tungsten carbide. The crusher is equipped with a sample input compartment for handling and cup cleaning, a sample output with an in-line magnetic separator and a level control system. A suction/extraction unit assures accurate cleaning of the crusher from the material inlet and outlet. All points where material is transferred openly are equipped with efficient dust extraction.

As soon as the coarse magnetisable parts have been separated from the slag, the system checks if there is still enough slag in the cup for further processing. If the level indicator indicates insufficient material, the deposited magnetisable material is returned into the cup, which is then transported to a service area for visual inspection by a laboratory technician.

Magnetic separation is of the utmost importance for reliable slag analysis, eg, the BOS converter operator needs to know the amount of iron oxide (Fe_2O_3) in the slag as it relates to oxygen blowing efficiency. However, as metallic iron is always present in the extracted slag samples and

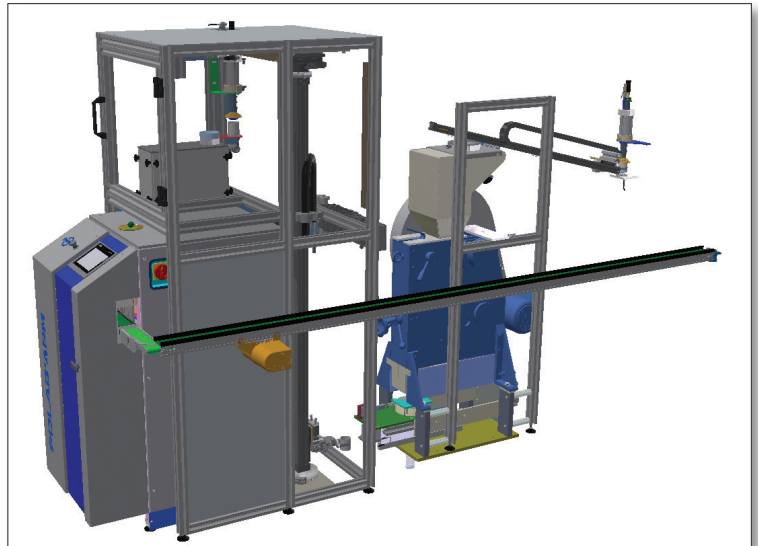


Fig 1 3D visualisation of sample preparation unit

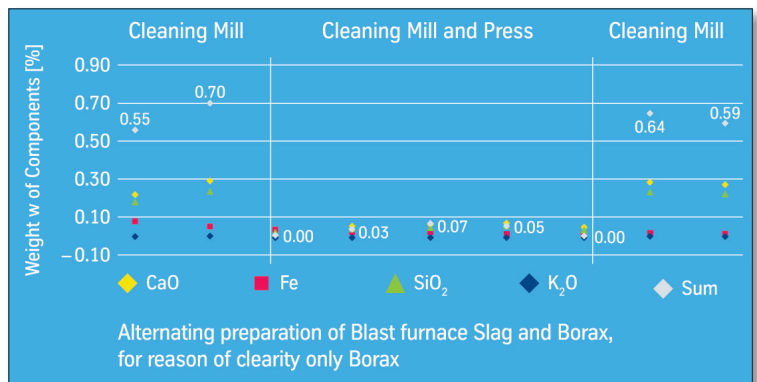


Fig 2 Borax sample analysis from alternating sample test

XRF cannot differentiate Fe_2O_3 from metallic Fe, the Fe must be removed before analysis.

Gravimetric dosing device The homogenised fragments leaving the crusher are next prepared for exact dosing. A handling unit transports the material from the crusher to the gravimetric dosing section, which is considered a highlight of the system. Gravimetric dosing has great advantages over volumetric dosing as some materials change volume and bulk density during processing, so volumetric dosing represents a compromise for different material parameters.

Gravimetric dosing, on the other hand, assures the definition of a correct mixing ratio (dosing) between sample and binder, even of materials with varying physical properties independent of volume and are dosed ready for tablet production.

The material is dosed to the nearest 0.1g – not easy under the conditions of a steel plant with frequent, considerable vibrations. The number of partial samples, which are needed for the cleaning process, are calculated by their weight. This is an outstanding feature and serves as an important basis when deciding if it is possible to prepare these samples.

polab APMplus sample preparation module The polab APMplus is the heart of the new automated laboratory system: The APMplus – only 600 x 1,230 x 900mm in size and 450kg in weight – features a grinding unit and a tablet press, integrated into a compact housing. It also contains a patented vibratory variable speed disk mill. The combination of grinding time and speed gives access to various grinding forces applied to different materials during sample preparation.

First, the grinding unit reduces the particle size of the pre-crushed material to analytical fineness. Because complete removal of all metallic Fe is so important, a second magnetic removal device is included. The coarse magnetisable parts have already been removed from the sample, but slag-coated small metallic iron particles remain which can only be removed after the sample has been ground. NB: by comparison, in fusion sample preparation, the sample material is merely sieved after grinding so the separation of iron from slag is not fully effective.

The sample is pressed into a steel ring, with the tablet press providing a pressing force of 47-93kN. The finished tablet is delivered at the side of the module and placed on a conveyor belt to the XRF or XRD analysers.

Resin tablets serve as a grinding pressing aid. These improve grinding of the sample to analytical fineness and also assure stability of the pressed tablet in the vacuum systems present within spectrometers. The new automation system facilitates a very economical use of

the grinding-pressing aid: in the Duisburg laboratory, the number of resin tablets required per sample dropped from 10 to 3, and has improved the OPEX of the installation considerably.

CONSISTENT CLEANING STRATEGY

One of the advantages of the new sample preparation system is that it can be efficiently cleaned, either as individual components or as a complete system. With its optimised cleaning strategy, the system prevents carry-over errors and contamination between different samples. A separate program can be run for each type of slag

Depending on requirements the cleaning can be adjusted. The system sends material batches to the sample preparation, which are then discarded either after the mill or as pressed tablets. Based on the slag charge's feed weight, the control unit decides how many cleaning samples can be used before the final tablet for the analyses. Usually a primary batch is used to flush the mill with the new sample material (blind sample) and is discarded with the dedusting system. In the case of very heterogeneous materials with very different properties, another blind sample is prepared in the mill, pressed and discarded. This cleaning is more intense.

TRIAL OPERATION RESULTS

Comprehensive tests have shown that the new sample preparation method has outperformed expectations. It offers a high processing speed and reproducible analytical results, separates metallic iron very reliably and avoids contamination effects by material carryovers.

To assess how good the cleaning behaviour of the setup was, a trial was conducted using blast furnace slag and a blank borax sample (see Figure 2). The two different materials were tested alternatively. As borax is not visible to standard XRF, all impurities are shown clearly in the blank sample. For clarity, only the borax results are shown in the figure. The result of this 'memory test' proved the efficiency of the automated cleaning procedures of the crushing, grinding and pressing section.

Today, the sample fusion process is regarded as the standard for sample preparation in a steel plant laboratory, thus fused beads served as benchmark to compare differential measurement of the iron value (see Figure 3). A chemically identical material was prepared both by fusion and the crusher APMplus system. The intensity of the mill magnetic field in the APMplus was varied and it was found that an intensity to only 10% of design capability was sufficient to reach the performance of fused beads in terms of standard deviation of repeated samples. The results confirmed the efficient performance of the magnetic separator of the APMplus.

In a series of tests with slag, the magnetic intensity

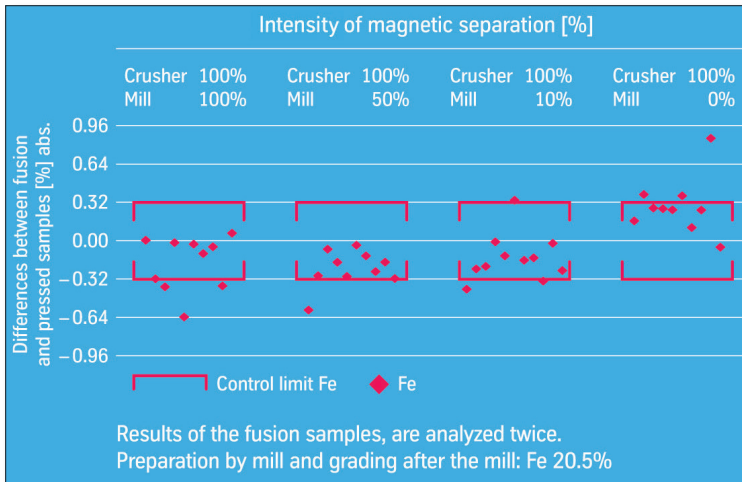


Fig 3 Effect of magnetic separation intensity on sample analysis difference with fused bead method (control limits set at 2 standard deviations of the fusion bead data)

was adjusted to between 0 and 100% (see Figure 4). For clarity only the CaO and SiO₂ are shown. The analysis did not reveal any significant differences in results, indicating that the APMplus separates the metallic iron, but no other elements.

The test series also indicates very low variability and very good reproducibility.

CONCLUSIONS

The innovative slag analysis system comprising sample preparation, crusher and polab APMplus, has been optimised for the demanding requirements in the steel plant laboratory. The automated system enables fast, highly precise slag analysis with a high sample throughput rate. Individual parameters can be predefined for each slag.

The compact dimensions of the unit are of particular advantage, enabling two units to be installed in the space previously needed for one in the Duisburg steel plant. With 40,000 slag samples per year, these additional capacities had been necessary.

The tablets from the new sample preparation meet the highest analytical requirements. The system takes less than seven minutes from receiving the slag to the pressed tablet, making it a viable alternative to conventional sample preparation methods. Fusion sample preparation is considerably more time-consuming, expensive and energy-intensive and less automated. **MS**

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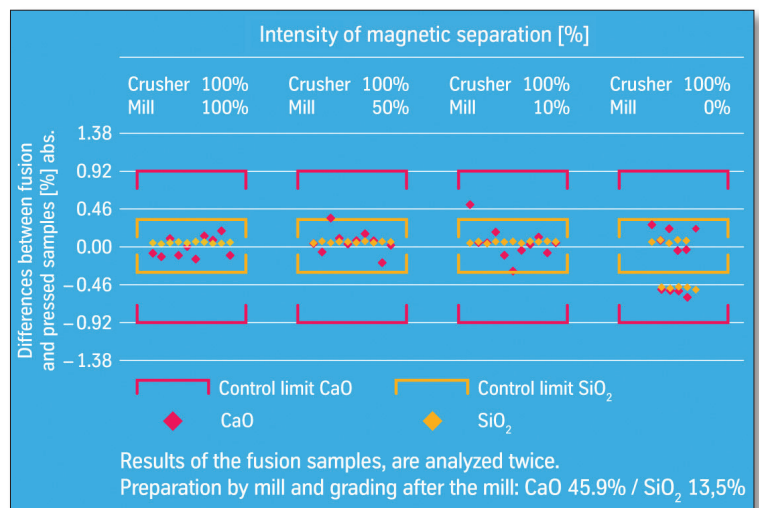


Fig 4 Effect of magnetic separation intensity on sample analysis difference with fused bead method