Sampling for Industry 4.0 – Sensor signal acquisition inspired by the Theory of Sampling (TOS)

M. Lischka[a] and K. Berelsmann[b]
[a]HERZOG Maschinenfabrik GmbH & Co. KG, Auf dem Gehren 1, 49074 Osnabrück, Germany
E-mail: m.lischka@herzog-maschinenfabrik.de
[b]HERZOG Maschinenfabrik GmbH & Co. KG, Auf dem Gehren 1, 49074 Osnabrück, Germany
E-mail: k.berelsmann@herzog-maschinenfabrik.de

The Theory of Sampling (TOS) has become firmly established in many process industries over the last few decades as the basis for precise and accurate material characterisation. Increasingly, considerations go into the design of sampling and sample preparation equipment to ensure sample representativeness. With the introduction of digitalisation under the buzzword Industry 4.0, many options have emerged to monitor these processes. But also, in the other fields, such as process analytics techniques (PAT) and applied sensor technology, which are not directly attributable to sampling, new applications arise where the Theory of Sampling is never-the-less a very useful addition. Here we present two case studies in which TOS have delivered decisive improvements in data acquisition.

Introduction

The application of Industry 4.0 approaches has already taken a firm place in many areas of industry that can no longer be imagined without. Not only can predictive maintenance be organized and implemented, but the information obtained can also be used to gain new knowledge about processes and to control them in a targeted manner. Primarily, machine parameters from systems and their components can be read out and evaluated. Here, for example, torque values, acoustic signals, travel times, and temperature can be mentioned and supplemented by many others[1,2,3]. In addition to the information that can be obtained directly, additional sensors and measuring instruments are frequently added in order to obtain a more extensive data density.

When using these methods to produce relevant data, a basic understanding of the measurement process itself is required to record a meaningful representative data series. Process Analytical Techniques (PAT) must be designed specifically for each application so that a sufficiently detailed information density can be used.

Accordingly, each measured and recorded value must be representative. This shows clear parallels to the sampling of raw materials for their analysis, the basis of which has been scientifically anchored in the Theory of Sampling (TOS) for decades and is being used more and more. Nevertheless, the Theory of Sampling is less known in the field of process technology and sensor technology. Here, however, it could play a decisive role.

At this point, not only obvious questions like: "How do I measure, and at which point?" arise, but also far-reaching other issue, such as sampling rate, interference factors, data transport speeds, and measurement uncertainties have to be considered. These and other specific sources of error add up to the Fundamental Sampling Error (FSE) for sensors which, together with the variation of the measured system, defines the total measurement uncertainty (TMU)[4]. There are many similarities between physical sampling and digital sensor signal acquisition.

Below two cases are presented that show how a fundamental understanding of TOS helps to properly design, monitor (QA/QC) and use sensor measurement systems. Both case studies are developed to control sample preparation processes for chemical, mineralogical and physical characterization of raw materials.

Sample preparation for X-ray fluorescence analysis

X-ray fluorescence (XRF) is one of the dominant measurement techniques for chemical analysis of inorganic substances[5]. In order to enable measurement by this qualitative method, the sample material must be prepared in such a way that these optimal conditions for the analysis is matched in each preparation. Forms of sample preparation are used here:

- Grinding and pressing of the sample material into a steel ring (Case A)

- or chemical digestion at over 1000°C and obtaining a homogeneous glass bead. (Case B).

Both methods require a basic reproducible preparation process to reproduce the initial conditions during calibration of the analytical instrument. Smallest deviations can have consequently negative effects on the measurement result[6]. When grinding and pressing the sample material, the achieved particle size distribution is an important

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factor, while in chemical digestion the temperature measurement accuracy plays a major role[7]. To ensure a sufficient reproducibility of those processes, while tool condition monitoring comes to the fore.

Case A: Measurement on powder

For a direct measurement of the sample material, a grain size must be achieved beforehand which is suitable for the low penetration depths of the X-rays. Particularly for the determination of light elements, grain sizes ideally smaller than 45 µm must be achieved. So-called vibrating disc mills are often used here to grind the sample material to a correspondingly fine size. This preparation method is subject to a variety of influencing factors that affect the particle fineness achieved and, consequently, the measurement accuracy of the XRF measurement. The use of an acceleration sensor attached to the grinding vessel can provide important information for this purpose. This provides information about the condition of the mill, which is naturally subject to wear, and can reveal variations in the sample material.

Vibrating disc mill with acceleration monitoring

Due to the unique design of the vibrating disc mills shown in figure 1, a new possibility has arisen to monitor the grinding process completely. The experimental set-up explained below in connection with the applied sensor technology are already protected by patent[8]. The special feature here is the design of the oscillating unit, which is freely supported by springs, and an acceleration sensor fixed to it, which is why the smallest changes during the grinding process can be determined and analysed.

The oscillating unit consists of eight horizontally arranged tension springs and six vertical compression springs, which support and stabilize the movable inner rigid assembly (eccentric drive and grinding vessel). To start the grinding process, the three-phase motor drives an eccentric weight at speeds of 700 to 1500 rotation per minute (rpm), which consequently generates a vibration and transmits it to the grinding vessel. This vibration is directed into a circular path by the symmetrical arrangement of springs, thus ensuring a controlled and uniform grinding process. The grinding is performed by a grinding ring and stone inside the grinding vessel. The two grinding tools are accelerated by the circular oscillation and roll against the grinding vessel wall, crushing the sample material. In grinding with the vibrating disc mill, the comminution mechanisms of compressive, shear and impact stress are mainly effective[9]. Thus, this principle enables reliable and fast grinding of medium-hard, hard, brittle or fibrous materials to particle sizes below 45 µm and, at the same time, realization of the highest standard in avoiding contamination of the subsequent sample.

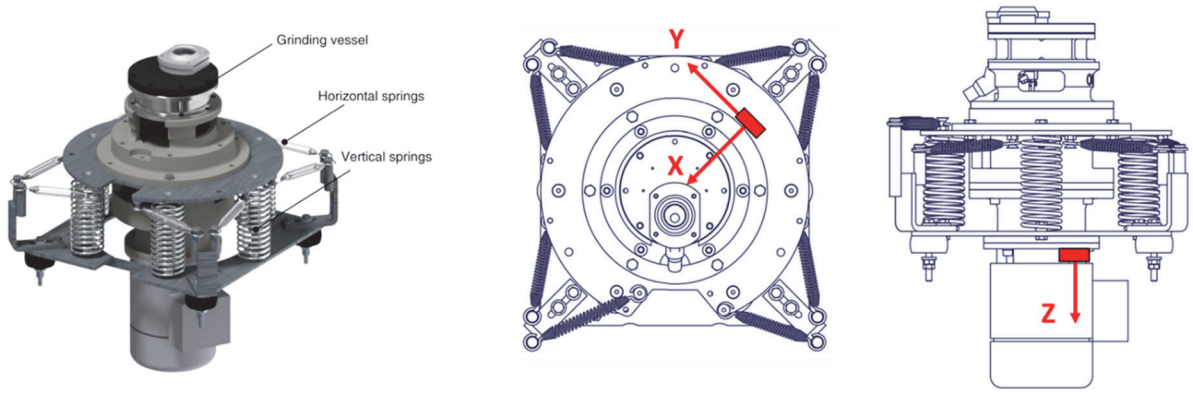


Figure 1: Swing aggregate of a vibratory disc mill with horizontal and vertical springs guiding the motion of the grinding vessel[10].

Many investigations have shown that not only changes in the mechanical components (wear of tension and compression springs, wear of the grinding set or motor settings), but also the specific properties of the material have a significant and characteristic influence on the vibration of the system. Therefore, to monitor the grinding process, a tri-axial capacitive accelerometer is fixed to the motor flange. The exact mounting position of the acceleration sensor on the vibrating disc mill is shown in figure 2. The sensor is mounted concentrically so that the X-axis points in the direction of the centre of the vibrating unit. To record the acceleration data, the data is read out and evaluated in real time. For the sampling rate of the measurement data, 100 values per second were specified.

In the following, two critical error potentials of the sensor-based monitoring of this oscillating unit are presented and how the application of TOS can assist in their avoidance.

Importance of a suitable position of the accelerometer

Imprecise location of accelerometer. A wrong sampling method (sampling location, execution, material quantity, sampling frequency) is known to influence the significance and accuracy of the subsequent analysis. The same is also the case for sensor technology as applied to the vibrating disc mill, where many factors (including the choice of sensor, recording frequency, positioning of the sensor) must be considered. Just as with TOS, it is important to identify the correct location or position for sample or data acquisition during acceleration measurement prior to subsequent analysis. Here, the more complex the process, the more difficult and important this decision is.

Due to the manifold and partly also random influencing factors on the vibration behavior of the vibrating aggregate, such as: Starting position of grinding ring and grinding stone in the grinding vessel (influences start-up behavior), filling weight of the material, grain size of the material, temperature, speed, grinding aids, etc., a high degree of complexity is given for this application. Therefore, instead of the actual direct investigation of grinding processes, the determination of a suitable sensor position was dealt with first, in order to be able to fall back on reliable and valid measurement data subsequently.

By considering the vibration to be expected theoretically and checking possible sensor positions in practice, the position shown in figure 1 was determined as the reference position. It became clear that the positioning of the sensor (i.e., the removal of the sample) plays a fundamental role. This is impressively shown by the two acceleration measurements in figure 2. The diagram shows the acceleration data in the X and Y coordinate system of two identical sensors during the same no-load operation at a speed of 1400 rpm, only located differently.

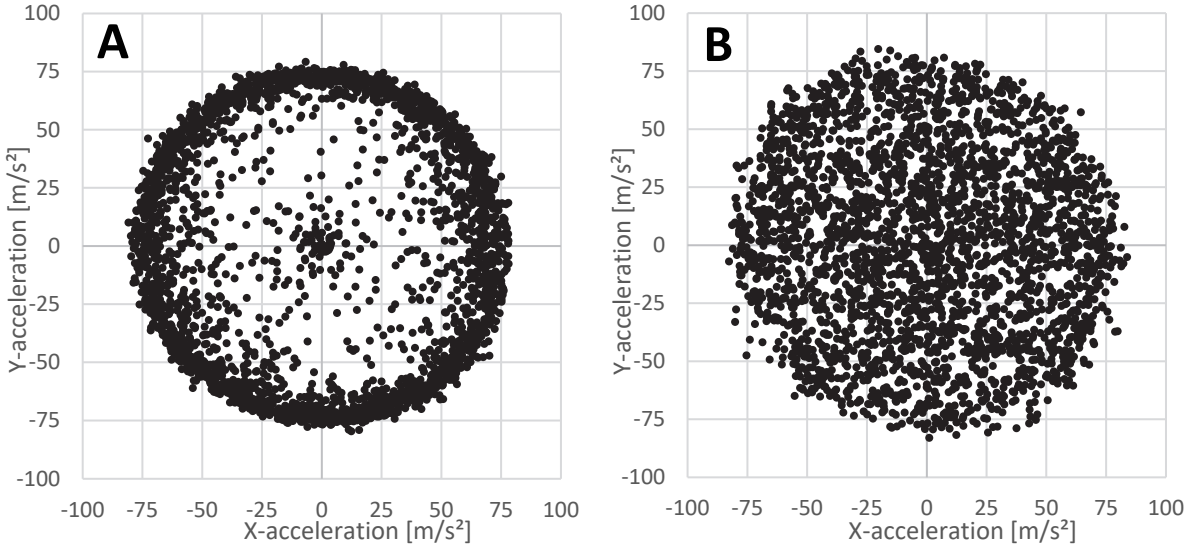


Figure 2: Measurement of acceleration in x- and y-axis at an unloaded milling run with 1400 rpm with sensor in (A) suitable measuring position according to figure 1 and in (B) unsuitable measuring position (near an attachment point of a horizontal spring) to evaluate the concentricity of the disc mill[10].

Based on the theoretical consideration of the oscillation system, an ideal circular path of the system is to be expected during an idle run, (free movement of the grinding stone and ring without the grinding of material). However, due to the tumbling behavior of the spring-loaded grinding vessel, not every position is suitable for mounting the sensor to detect this circular motion. In position A (at the motor flange), the expected circular motion including the partial deviations can be seen very clearly. This is contrasted by the recorded measurement data (B) from the sensor position at the attachment point of the tension springs. Although a circular motion can also be guessed there, this can by no means be described as uniform and makes a detailed statement about the condition of the oscillating unit in comparison with the theoretical reference value difficult or impossible. The deviating measurement data from sensor position (B) are, in addition to the tumbling behavior of the oscillating unit, probably also generated by the spatial proximity to the grinding vessel, since impact and shock movements of the grinding set additionally have an influence on the measurements of the acceleration data.

With position (A) a suitable sensor position was found for this application, which can realize an accurate monitoring of the grinding process. This is demonstrated in the following section on a practical example for potential benefits of a tool monitoring system.

It should be noted that the sensor must always be mounted in the same fixed orientation (position and inclination), otherwise there is no comparability between grinding processes. Even small angles or changes in direction influence the recording of the acceleration data, so that the potential statement of the TCM system about the condition of the

vibrating unit would be subject to error. Therefore, one should also follow one of the principles of TOS and implement a constant control of the sampling or data recording, since the greatest error occurs at this point in the process.

In data acquisition, as in sampling, the principles of TOS apply: the method must always be precisely adapted to the particular application, otherwise errors may occur, which may propagate to an error in the subsequent analysis.

Selection of the sampling rate

While in the case of sampling from a lot or from a material stream, attention must be paid to the frequency of material sampling to obtain a result that is as representative as possible. The question of the sensor's sampling rate is indispensable in the case of sensor condition monitoring. For both cases it is true that an incorrect methodology at this point will most likely have a significant impact on the analysis result (measurement result of the XRF or condition monitoring of the TCM system).

To illustrate the importance of this check, two variants for the choice of sampling rate are presented and evaluated below. For this purpose, two idle runs were performed at 1400 rpm and the acceleration was recorded in the first step with the standardized sampling rate of 100 Hz and in the second step with 23.3 Hz, which approximately corresponds to the excitation oscillation of 23.3 Hz.

The investigation includes the analysis of the vibration in the X-direction. For this purpose, the measured data points of the X-acceleration for both sampling rates were first plotted against the grinding time. Subsequently, a sinusoidal curve representing the ideal acceleration curve for a uniform vibration was added. The ideal acceleration curve thereby approximately describes the vibration behaviour in the so-called idle state.

The general form of the sine curve contains some parameters which change the course of the graph with regard to certain characteristics.

$$A(t) = A_0 * \sin\left(2\pi * \mathrm{T} * (\mathrm{t} + \varphi)\right)$$

Where:

- A_0: Amplitude of the oscillation [m/s^2]
- T: period duration [s]
- φ: phase shift [s]
- t: time [s]

This sinusoidal curve and the measured acceleration values can be used to demonstrate why the "sampling parameters" should be precisely matched to the application and checked before evaluation/analysis.

The effects of an incorrectly selected sampling rate can already be seen in the graphical representation of the measurement data on the vibration curve. In comparison with the idealized vibration in no-load operation, the sampling rate can be immediately identified as the source of the error, but usually only statistical data, as shown for this example in the following table, are available for evaluation or analysis.

Table 01.: Statistical evaluation of the measurement data with the sampling rates 100 Hz (A) and 23.3 Hz (B.)

	Fig. A	Fig. B
Sampling rate	100 Hz	23.3 Hz
Number of data points	51	12
Standard deviation	51.6 m/s²	0 m/s²
Average acceleration	0.5 m/s²	-25 m/s²

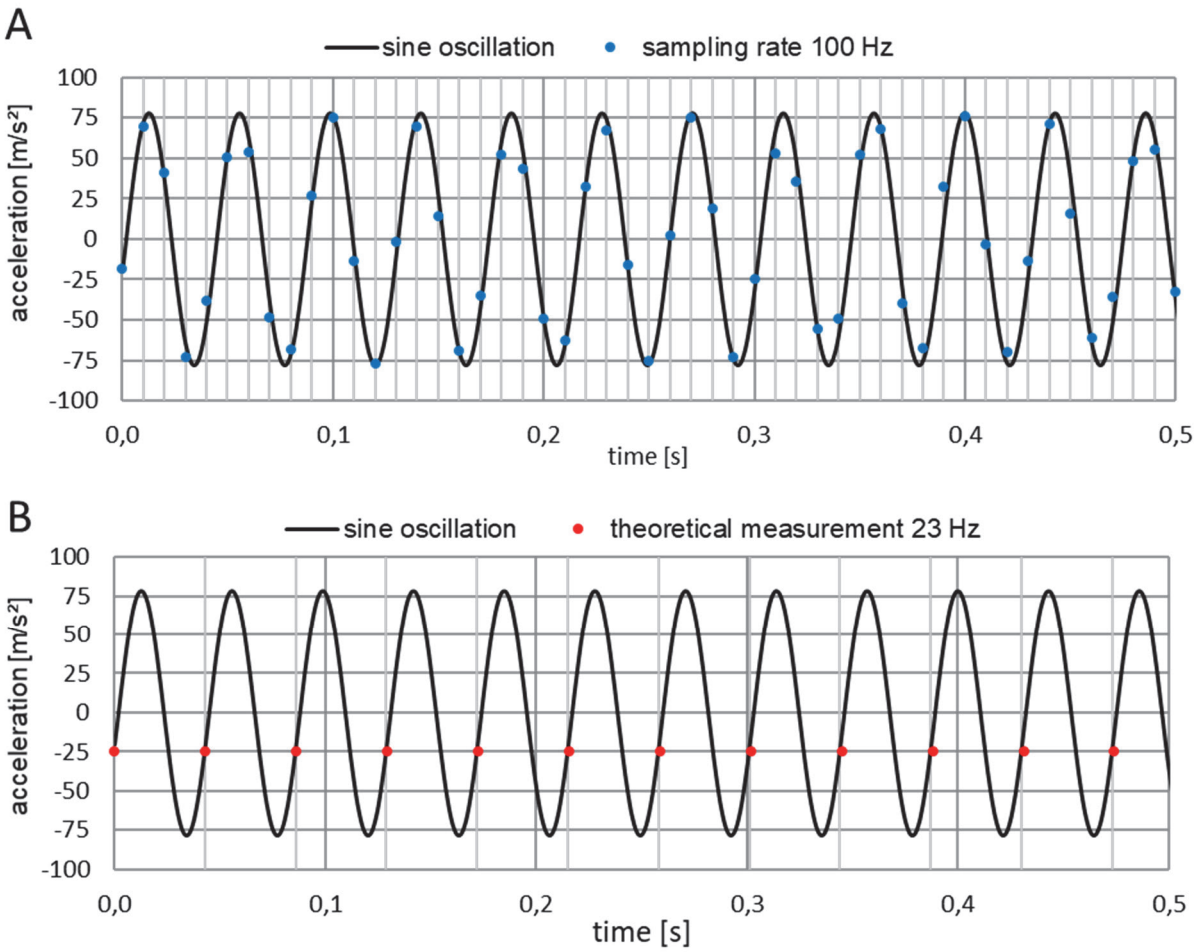


Figure 3: Display of the measured values for acceleration in the X-direction for an idle run with approximated sinusoidal oscillation with a sampling rate of (A) 100 Hz and (B) 23.3 Hz over a time range of 0.5 seconds.

On the basis of the static values, one can see an opposite course, although the acceleration or oscillation is actually identical. While the statistical values of figure A are approximately representative of the present oscillation (mean value of all acceleration must be 0 m/s² for circular movements), the values from figure B indicate a constant acceleration of -25 m/s² and thus manifestly not a circular movement.

To avoid this misinterpretation, the principle of sampling frequency (sampling theorem) must always be considered before using a sensor. The Shannon-Nyquist theorem states that the sampling frequency must be greater than twice the fundamental frequency of the sensor so that the fundamental frequency can be measured correctly [11]. At the present maximum speed of 1500 rpm, this results in a fundamental frequency of 25 Hz for which the sampling theorem is satisfied.

Also, for this point of consideration of the data acquisition at a vibrating disc mill, a consideration of the basic principles of TOS can support in error prevention. It is always important to increase the significance of a measurement as much as possible.

Practical example for potential benefits of a tool monitoring system

With a representative and detailed measuring system, the movement path of the grinding vessel can thus be continuously monitored. To guarantee an optimum service life for the mill, this data can be used for predictive maintenance. The following data set of six recorded runs of the mill clearly shows how data analysis can help predict machine failures, such as in this case caused by a cracked motor flange anchor bolt. The first signs of developing damage were evident five days before the actual event. Ideally, the X/Y vector of acceleration should reflect the relatively stable circular path of the grinding vessel (Graph 4 A). Slight deviations from the ideal condition can already be seen here. Nevertheless, not as clearly as they become visible in graph 4 B. Here the acceleration values show two pronounced negative anomalies, which are opposite each other on the circular path. Over the next few days, the acceleration drop at these two positions increased and the oscillation behaviour of the grinding vessel

was more and more disturbed by the speed changes. The reduced power transfer due to the slowly loosening motor from the grinding vessel also impacted the achieved particles size distribution of the material to be pulverized. The material was not ground sufficiently which would have had significant consequences for accuracy and precision of the subsequent analysis.

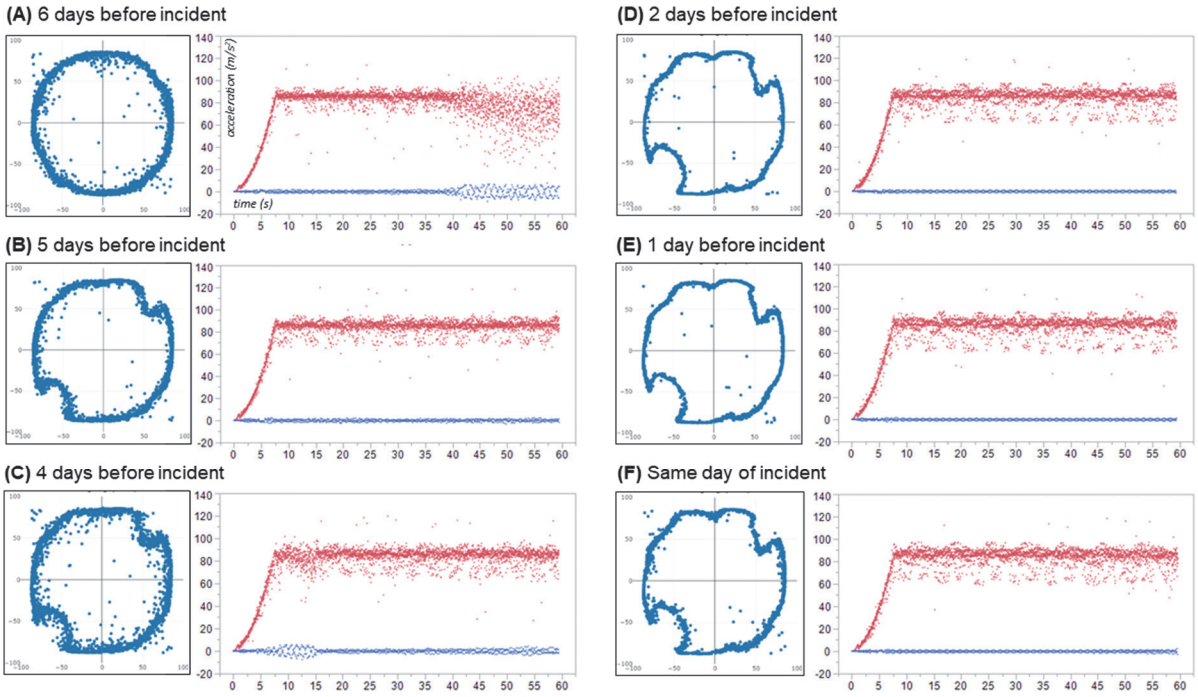


Figure 4: Display of acceleration x-/y- vectors (left box) and RMS values (red dots, right box) in the period before incident with breakage of anchor bolts between drive motor and swing aggregate. Six days before (A) only small deviations in the acceleration could be observed. In the subsequent period of time (B-F) a significant progressive deviation occurred.

When the anchor bolt finally broke off, there was a direct loss of velocity of almost 50% (see figure 5). With the acceleration curves recorded, it is now possible to interpret these anomalies using statistical methods. This data set can be used in the future to inform personnel that an inspection and, if necessary, precautionary maintenance must be carried out. With these measures, machine failures can be effectively avoided and a high availability of the system can be realized.

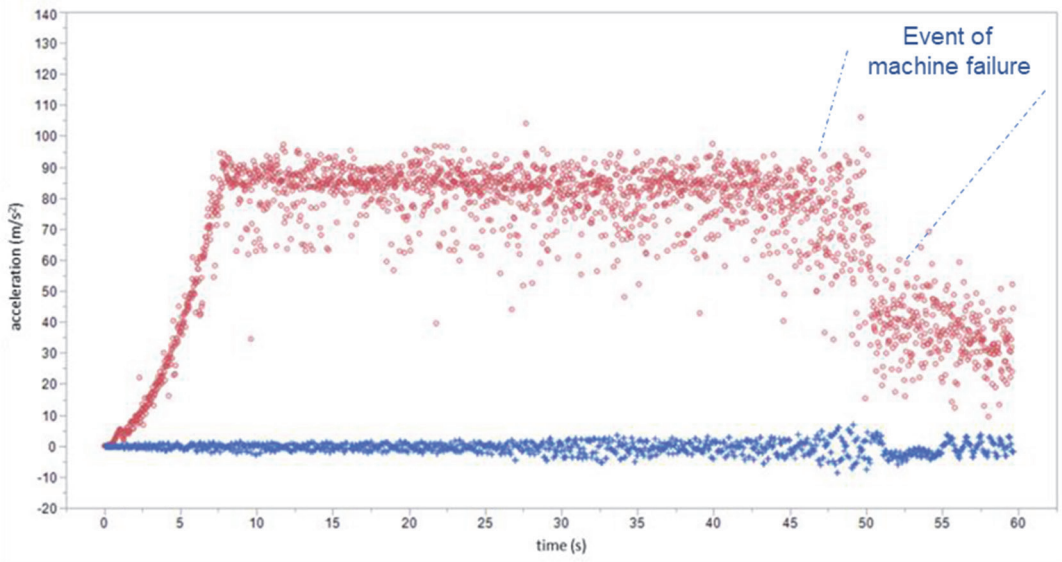


Figure 5: Display of the RMS of the x-/y- acceleration (red dots) and z-acceleration (blue dots) showing the breakage of the anchor bolts.

Case B: Measurement of a borate glass

The chemical digestion of the sample material in a lithium borate has many analytical advantages and creates, among other things, a matrix-independent sample preparation[12]. In a fixed mixing ratio, glass former/flux and sample material are digested for several minutes at over 1000°C, homogenized, and then poured into a dish. Three different heating methods are used here in laboratories, electric resistance furnaces, gas burners or induction digestion units, to heat the platinum/gold crucible (95% Pt/5% Au) to the desired temperature. For classical applications (electric/gas), thermocouples can be used for direct temperature measurement, providing good temperature resolution. For more complex digestion methods, where different temperature levels are required during fusion, induction heating is particularly suitable because the temperature control can be adjusted with almost complete flexibility. This is due to fast heating and cooling rates. Those fusion methods also tend to be sluggish and maintenance prone due to the corrosive vapours of the chemicals often used in the process where induction heating is much more reliable compared to other heating methods. The disadvantage of this heating method is that temperature control during digestion can only be performed indirectly via infrared pyrometers and strongly depends on a well-known emissivity of the measured Pt/Au crucible.

Methodology

A general setup of the fusion unit is shown in figure 6 and the characteristic values of the infrared pyrometer used are summarized in table 2. The pyrometer measures the thermal radiation emitted by the crucible and computes the temperature with

$$T = \sqrt[4]{\frac{P}{\varepsilon \times \sigma \times A}}.$$

Where T is the temperature [K], P is the total radiated power [W], ε is the emissivity, σ is the Stefan-Boltzmann constant [W m^{-2} K^{-4}] and *A* is the area [m^2]. The only unknown variable is the emissivity, which for platinum depends on the surface condition and temperature.

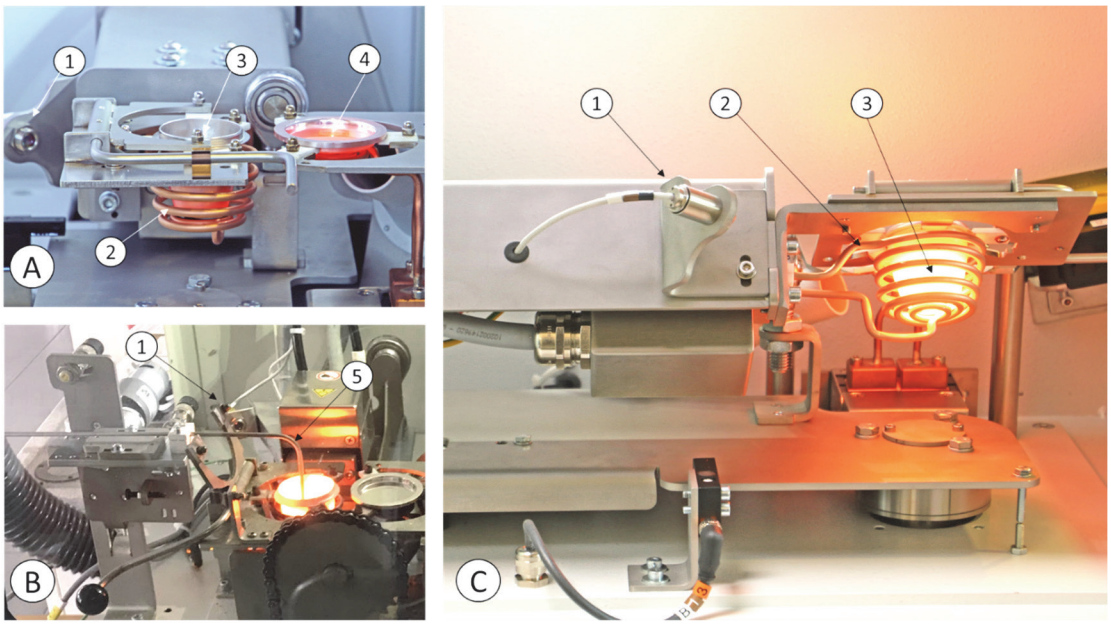


Figure 6: Induction heating unit with:
(1) IR pyrometer
(2) Induction coil
(3) Pt/Au crucible
(4) Pt/Au casting dish
(5) Thermocouple for calibration.

In order to allow temperature calibration and determine the emissivity (ε), a patented methodology has been developed, which allows the emissivity of the crucible to be determined indirectly from the temperature of the melt[13]. Here, a NiCr/Ni thermocouple (see table 2) is positioned in the borate melt and the emissivity is adjusted by an algorithm until the pyrometer reading matches that of the melt. In this way, a specific emissivity can be assigned to each crucible used. To account for the additional temperature dependence of emissivity, calibration is performed

at two (or four) temperature levels, taking into account the fusion temperature range used in the specific method to be applied.

Tabel 2: Main features of the used IR pyrometer used for process control and the K-Typ thermocouple used during the temperature calibration of the IR pyrometer.

IR pyrometer crucible - 1MH Used for process control			**K-Typ thermocouple SCAXL-125U-6-SHX** Used for calibration	
Temp. range:	650-1800°C		Type:	NiCr/Ni
Spectral range:	1 µm		Temperature range:	0-1145°C
Optical resolution:	75:1		Uncertainty:	2,2°C or 0,0075 x T_m
Accuracy:	± 0.3% T +2°C			
Reproducibility:	± 0.1 % T+1°C			
Resolution:	0.1°C			
Detection time:	1 ms			

Once the emissivity's $(\varepsilon_i, \varepsilon_j)$ for both temperatures (T_i, T_j) have been determined by the control system, a linear equation can be drawn up which shows the relationship between emissivity and temperature. For a more precise calibration the regression can be extended to four temperatures used for calibration.

The general term for the linear equation with ε on the Y-axis and T on the x-axis is

$$\varepsilon = m\,T + b\,.$$

The following applies to the emissivity and temperature pairs$\{(T_i|\varepsilon_i), (T_j|\varepsilon_j)\}$. The slope m_{ij} of the linear regression line is

$$m_{ij} = \frac{\varepsilon_j - \varepsilon_i}{T_j - T_i}.$$

The y-axis intercepts b_i or b_j are defined by

$$b_i = \varepsilon_i - m_{ij}\,T_i\,,$$
$$b_j = \varepsilon_j - m_{ij}\,T_j.$$

The emissivity ε_θ of a target temperature θ is calculated by

$$\varepsilon_\theta = \frac{\varepsilon_j - \varepsilon_i}{T_j - T_i}\,\theta + \left(\varepsilon_i - \frac{\varepsilon_j - \varepsilon_i}{T_j - T_i}\,T_i\right) \equiv \frac{-\varepsilon_j T_i + \varepsilon_j \theta + \varepsilon_i T_j - \varepsilon_i \theta}{T_j - T_i}.$$

Even the emissivity can be determined using this method, but there still remains other factors that can impact accuracy and precision during the fusion process.

With the aim of achieving a temperature accuracy of +/- 5°C, the measurement system was investigated further in numerous measurement series and multiple sources of error were identified:

- Dependence of the emissivity on the crucible surface and the measured temperature, which changes during the life cycle of the platinum crucible.
- Positioning/orientation of the crucible in its holder.

Time dependence of surface heterogeneity

The heterogeneity of the platinum crucible surface can be attributed to various reasons and increases during a life cycle. The constant exposure of the material to high temperatures leads to grain growth in the Pt/Au matrix[14]. In addition, during melting, chemical reaction between sample material and crucible may occur, especially when the samples contain so-called platinum poisons or reduced phases[15]. Depending on the initial surface properties the emissivity decreases. The aging rate is therefore dependant on the sample material, digestion method and crucible composition[16]. Additionally, a higher wearing rate can be observed new crucibles at the beginning of their usage. Accordingly, this defines the recalibration interval to ensure a representative measurement over the entire life cycle (see figure 7). To keep the calibration effort as low as possible, characteristic data (e.g. frequency and energy from the high-frequency generator) can be read out from the high-frequency generators in order to monitor the condition of the measurement system via statistical process control.

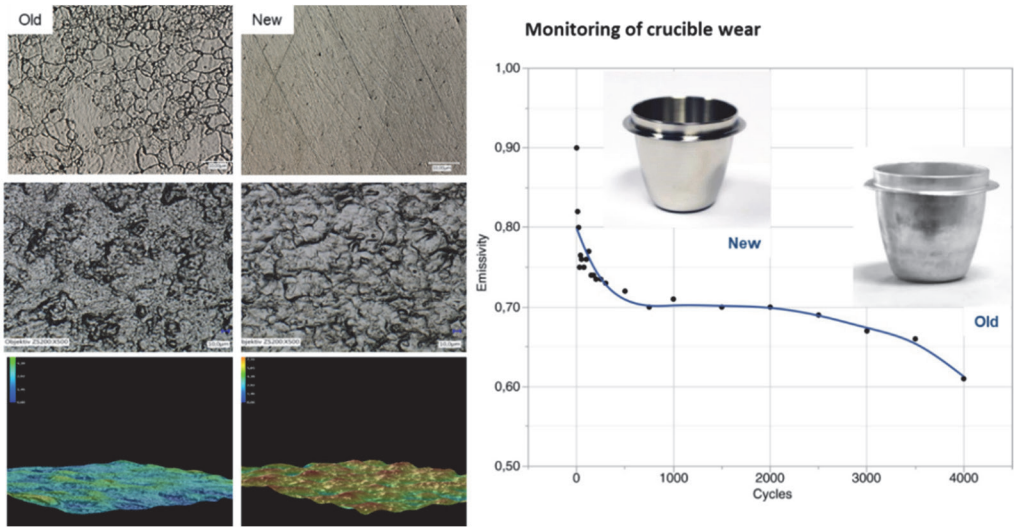


Figure 7: Change of the emissivity of the crucible surface over 4000 heating cycles. Where reactions in the Pt/Au matrix leading to matrix alteration and changes in the surface roughness.

Addressing in-situ surface heterogeneity

In addition to the aging of the crucible, the positioning of the crucible in the digestion unit also plays a decisive role for the measurement accuracy. Especially after a certain period of use, the crucible surface shows extensive heterogeneity resulting in a broad variation of the emissivity. Depending on how the crucible is positioned, this results in sometimes considerable differences in emissivity and thus in the temperature that drives the control system. Figure 8 shows an example of the typical temperature variation for an old crucible. For the data series, the crucible was rotated 360° twice over in 10° increments, and the temperature of the melt was determined at constant emissivity (ε = 0.556) and introduced energy (22%).

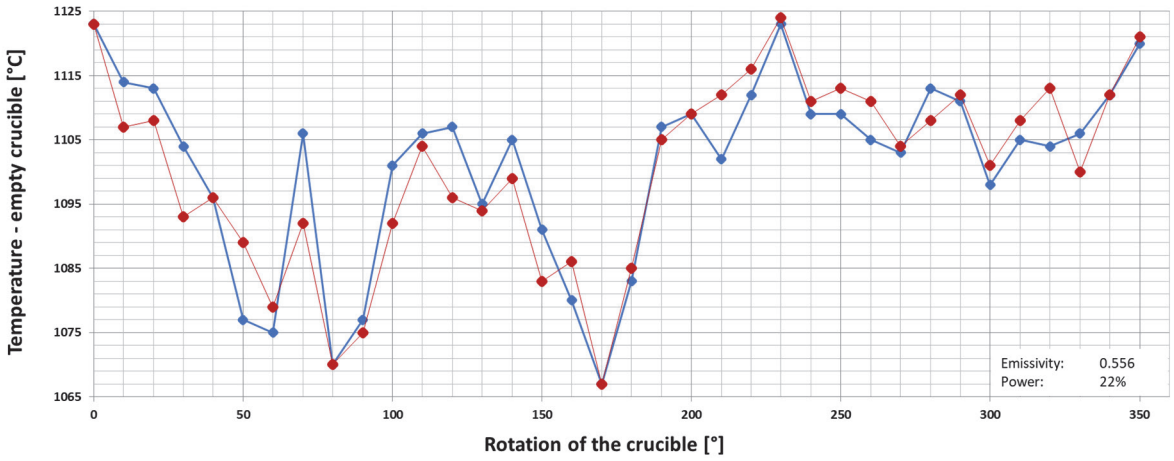
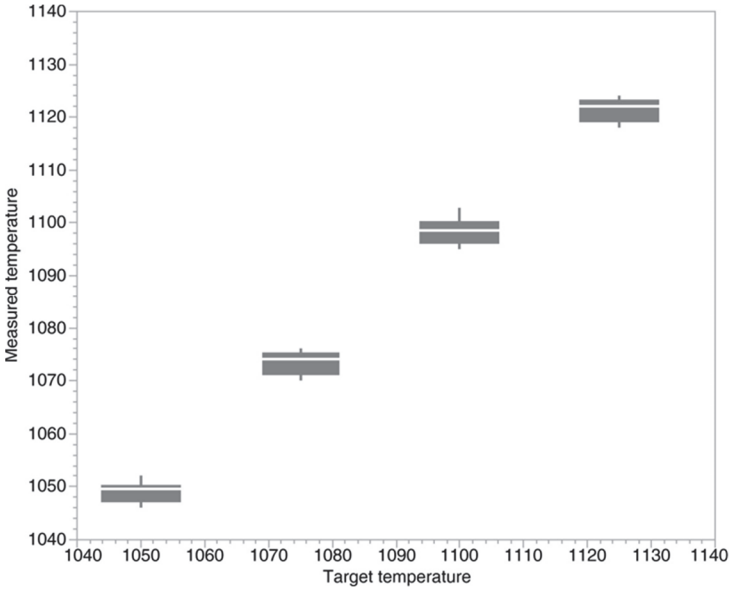


Figure 8: Measured temperature variance for an old often used Pt/Au crucible heated with fixed emissivity (ε = 0.556) and energy (22%). The crucible was rotated in 10° intervals completing 360° twice.

In order to minimize the fluctuations of the emissivity over the crucible surface, a solution had to be found that positions the crucible as precisely and repeatably as possible in the digestion unit. In benchtop units, this is done manually by the operator, who uses markings on the crucible and digestion unit. For automated systems in which industrial robots are used to place the crucible, camera systems can be used for alignment. The bottom of the crucible is provided with a marking that can be used by the camera system to determine the exact orientation of the crucible. With this angle information, the robot can approach each crucible individually in such a way that, after it has been set down, the temperature is always determined at the same point on the crucible.

Measuring system accuracy

Taking into account the above-mentioned sources of error in the measuring system and the resulting measures, these factors can be successfully reduced to a large extent. This has been proved in a replication experiment, where the temperature calibration has been checked 20-times for each temperature that has been used in the linear regression (see figure 9). The mean temperatures recorded show a standard deviation of only +/- 2° K.



Target temperature [°C]	Mean Temperature achieved (±SD; Max-Min)
1050	1049.0 (2; 6)
1075	1074.0 (2; 6)
1100	1099.0 (2; 8)
1125	1122.0 (2; 6)

Figure 9: Temperature precision and accuracy for a four-point calibration, checked by a 20-fold replication test. The mean temperature has a standard deviation of +/- 2°K.

Conclusion

The two case studies from the sample preparation field for material characterization clearly show how sensor technology and machine performance indicators can help to monitor processes and obtain additional information on the process. The design of the measuring system and its recalibration interval play a decisive role in ensuring that the measured values obtained are indeed representative of the process and/or guarantee its long-term stability.

Considering case A, it becomes clear that when monitoring motion or, equivalently, vibration, the sampling rate must be synchronized with its frequency. Likewise, the optimal positioning of the sensor must be very precisely defined in order to eliminate interference and overlapping as far as possible. Case B, on the other hand, exemplifies the necessity of how the surface properties of the object under consideration play an important role in indirect measurement methods. These can vary at different stages of the process or change over time and require constant monitoring and recalibration. In both cases, the accuracy and repeatability of the measurement systems could be significantly improved using the theoretical approaches of representative sampling or recording of data.

The evaluation of the acquired data can contribute in two ways. First and foremost, the measurement series are used for process control and its monitoring in order to ensure high reproducibility. Furthermore, the same data can be used to give an indirect statement about the reproducibility of the sample preparation. Deviations in the measurement series, for example due to the constitutional and distributional heterogeneity of the sample material, can thus be detected and interpreted. Deviations thus provide additional value to the actual analysis and can also provide direct information about the sample material or be used as a quality proxy in process control.

ORCID iDs

M. Lischka https://orcid.org/0000-0001-7259-0079
K. Berelsmann https://orcid.org/0000-0003-0711-9875

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