Smart Sensors for Process Analytical Technology

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***Abstract*—Increased globalisation and competition are drivers for process analytical technologies (PAT) that enable seamless process control, greater flexibility and cost efficiency in the process industries. This research is carried out in collaboration with a project which aims to introduce an integrated process control approach, embedding novel sensors for monitoring in real time the critical control parameters of key processes in the minerals, ceramics, non-ferrous metals, and chemical process industries. The paper will review the development of a suite of affordable sensors along with smart sensor features and algorithms for easier integration, easier maintenance, metrological performance enhancement, process monitoring and control and sensor fusion for use within this versatile global control platform implementing PAT. Smart sensors will be investigated* that match existing offline solutions in performance while enabling size reductions, low power consumption, low unit costs, low maintenance costs and data fusion.**

I. Introduction

PAT involves the implementation of sensors to provide real-time data on a process to which data analytics is applied to gain better process understanding and improved product quality. PAT entails the use of affordable, non-destructive, at-line measurement techniques coupled with process analytics to deliver outstanding process understanding and enable predictive control to operate industrial processes at their optimum. A concept closely aligned with PAT is Quality by Design (QbD) by which key strategic product specific attributes are identified in order to devise a robust control strategy which is monitored and constantly updated for continuous process improvement [1].

This paper will review the selection of sensors for use in a number of case study industrial processes as part of their adoption of PAT and QbD. The main applications of interest encountered in this research include the measurement of moisture content and mass flow rate of powders and moisture content in polymer resins, as even though monitoring these parameters is critical to process control in many applications, they are not widely measured compared to parameters such as temperature or liquid flow. Consequently, there is a need to document the selection process from the limited range of sensors available for these difficult applications.

Smart sensors integrate traditional sensors with microprocessors and a communications interface. Smart sensors are ideal for PAT applications as (a) the intelligence provided enhances their measurement performance and their applicability in large sensor networks where decentralized control can greatly reduce network traffic and computational complexity at the fusion centre and (b) the communications interface provided enables sensor measurements to be transmitted in the desired format over a standard protocol, further reducing the complexity of data fusion.

Section II of this paper will examine the availability of affordable, versatile smart sensors for the target applications. Section III will look at some of their features that ease sensor maintenance and integration thus overcoming the pre-existing barriers to the adoption of PAT- high sensor cost, lack of internal knowledge within processing operations and the existence of dedicated sensors and data management systems for unique situations.

II. Application Specific Smart Sensors

The following sensor applications were identified by partners of the ProPAT project [1], to be realized to provide data on key strategic product specific measurands for process modelling and control and also to complement the multivariate data produced by Near Infrared Spectroscopic (NIRS) sensors and Particle Size Scatterometers also being developed by the project. The case-study industrial processes involved in the project include a pharmaceutical continuous tableting line, a milling process for ceramics and non-ferrous metals, a milling process for minerals and a chemical polymerisation process. The former 3 processes handle powders with the latter process manufacturing liquid resins.

A. Temperature of powders

As the absorbance spectra measured by NIRS is affected by temperature [2], a temperature reference is required to calibrate the multivariate data. In the aforementioned powder applications, the powder is constantly moving through the processes, by freefall or pneumatic conveyance, and the temperature of the particles should ideally be measured rather than the surrounding atmosphere. Noncontact temperature measurement lends itself to such an application.

Infrared (IR) Temperature sensors are commonly used for noncontact measurement of the surface temperature of the objects. They average the infrared radiation emitted by surfaces in their field of view, so in this application, an average reading of the stream of powder and pipe wall in the background is produced. IR sensors can be configured to measure substances of a particular emissivity [3] thus allowing only the powder (which will have a high emissivity) to be measured and the reflective background of the process (which for our applications is stainless steel) to be ignored.

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A number of different optical configurations are available to adjust the spot size dependent on the distance between the powder stream and the sensor. The main issue to address for IR and any other optical sensors would be keeping the lens clean as powder build up will mean the sensor will only measure the surface temperature of the coating that sits on the lens itself. A number of solutions are available including situating the probe clear of the powder, using pneumatics (either built into the probe [4] or from the process) to clean the lens periodically and using a sight glass with particular shape/material properties that prevent the powder build-up [5].

B. *Moisture content of Powders*

Moisture content is a critical control parameter in the project's pharmaceutical continuous tableting line application as it directly affects the products quality in terms of shelf-life and must be carefully controlled to maintain process stability during granulation [6, Sec. 7.1] and drying stages of the process. NIR sensors are commonly used to measure moisture content in pharmaceutical applications as water shows strong absorption bands [7], it provides noncontact measurement, it does not require sample preparation and it provides real-time data. NIR will be used for moisture content measurement in this project, as well as the measurement of chemical composition to control the critical rates of API in the product. However, NIR is not without its limitations. NIR has poor depth of penetration meaning the sample is not representative if the process materials are nonhomogenously distributed (sampling techniques can be used to overcome the problem [2]). Also, the multivariate data from NIRS is useless without calibration against supporting references [7]. Similarly to temperature, a real-time in-line, cost-effective moisture measurement was desired to complement the NIR data for chemometrics.

The availability of low-cost sensors for measuring moisture content in bulk solids is not widespread. Recent research has been dedicated to the utilisation of a number of principles including capacitance, electrostatics and microwave resonance.

Electrical Capacitance tomography (ECT) and Electrical Resistance tomography (ERT) have been employed [8], [9] and offer the advantage of producing a tomograph of moisture distribution rather than just perform point measurements. A drawback is that the integration cost is quite high (€10k plus) as a custom solution is required (the method has only recently been commercialised [10]). Also, the measurement is dependent on bulk density which is undesirable in a process with varying ingredients/product lines. Planar capacitive type sensors have also been developed, but are also very expensive [11].

Triboelectric probes are generally implemented for measuring the concentration of dust particles in air [12] and measure the electrostatic charge deposited by particles onto a earthed probe producing a current proportional to the mass flow and moisture content of the particles. A recent study found that the method was capable of moisture measurement, but only during the drying phase after the agglomerates produced from wet granulation had broken up somewhat [5]. Also, only point measurement is performed and many probes costing in the region of €3000 would be required to quantify process moisture distribution.

Microwave Resonance involves measuring reflection and transmission of microwave radiation to determine moisture content. The microwave field changes its polarity rapidly, only water molecules can follow this change as they are small and have a strong dipole and the energy required to move these water molecules is measured [7]. The method holds advantages over NIR including deeper penetration depth and no requirement for chemometric calibration [13] and commercial offerings are available at a moderate cost [14]. Similar sensors that penetrate the sample with invisible electromagnetic waves are acoustic and radio wave sensors. Acoustic sensors require moisture levels greater than 15% and are therefore unsuitable for the application [5]. Radio wave sensors were discovered that are manufactured by IMKO GmBh for moisture determination in grains and minerals and use TDR (Time Domain Reflectometry) by measuring the time it takes the waves to be reflected to measure the material's dielectric constant [15] which is dependent on moisture content [16]. The benefits of this unique measurement method [17], coupled with the selected wavelength of 600MHz to 1.2 GHz Band (which make it suitable for measuring larger particles than microwave methods [18]), and the fact that they are a third of the cost of microwave sensors make the method a very interesting solution for moisture determination in solids.

C. *Mass Flow Rate of Powders*

The mass flow rate of bulk solids is another parameter that is lacking a cost-effective solution even though it is invaluable to process understanding in many applications. Electrostatics, capacitance, thermal transduction, optics and acoustics have all been identified as principles by which inferential measurement of mass flow can be achieved [19]. However, these methods have yet to be commercialised. There is no shortage of sensors for the mass flow of liquids and gases, (turbine, Coriolis, ultrasonic and thermal to name a few) but unfortunately, these sensors are unsuitable for the mass flow of powders as they cannot distinguish between the mass flow of air and the mass flow of slower moving particles. Microwave sensors are the most prevalent in-line technique for particles conveyed pneumatically/in free-fall. They operate by emitting electromagnetic waves and analysing the reflected signal to produce a mass flow measurement (by the Doppler Effect, signals from the particles are frequency shifted – the amplitude and frequency shift of the signal is proportional to mass flow [20]). The microwave method was chosen for the application for its applicability to any powder material and its moderate cost [21]. Possible alternative solids mass flow sensors in order of commercial availability for the purpose include differential pressure [22] (requires flow impeding orifice/venturi tube), triboelectric [23] (not commonly implemented for the application, offers no cost advantage), ECT [24] (very expensive), optical [25] (available for use in particle concentration/turbidity measurement, applicability requires investigation), acoustic [26] and thermal sensors [27] (developed in research but not commercially available).

D. *Viscosity of polymer resins*

One of the applications for which sensors were sourced is monitoring the viscosity of unsaturated polyester resin. As with the previous powder applications, the viscosity sensor's data will support an NIR sensor in chemometric analysis, to determine chemical composition and monitor the progress of the chemical reaction. The applications main difficulty is the high process temperatures of up to 240°C which required a more expensive, heat resistant viscometer to be selected. Viscometers can reasonably be divided into 2 categories based on their measurement principle, Solid-state viscometers and Mechanical viscometers. The main difference between the two types is the presence of moving parts, which require more maintenance and possess a settable shear rate for better accuracy and applicability to non-Newtonian fluids, but a narrower viscosity range. A solid state torsional oscillating quartz viscometer was ultimately selected for its low cost and small size. The selected sensor excites a piezoelectric element at high frequency and monitors the electrical frequency response as the resonating crystal interacts with the material under test [28].

E. *Colour of polymer resins*

Colour sensors were investigated for monitoring colour during the polymerisation process of alkenyl succinic anhydride (C16/18 ASA) for paper sizing produced from the high-temperature reaction of isomerized olefins and maleic anhydride, where the resin's amber shade gets darker with process development. True colour sensors are suitable for such an application as they can determine colour difference with greater accuracy than the human eye [29] and when used with heat-resistant fibre-optic cables, can be easily applied to extreme applications. The selected sensor was chosen as it has 6 spectral filters [30] which would yield more information than the more common arrangement with 3 filters (for red, green and blue wavelengths). Such a multi-spectral sensor performs measurement of an entire spectrum (the filters' spectral sensitivities overlap) and can compensate metamerism effects [29]. The sensor has digital outputs that can be programmed to trigger when colour changes/deviates from a set tolerance. Fuzzy logic can also be used to quantify colour measurements and use them for industrial control applications [31].

F. *Moisture content in polymer resins*

This section outlines the selection of moisture sensors for monitoring the moisture content in the process in Section E. The moisture measurement is made before polymerisation when the polymer reactor is flushed with olefin and raised to 160°C to ensure clean and dry reactor systems. A sensor is required to replace a Karl Fischer titration test to determine if the moisture content has fallen below 100ppm.

Methods for moisture measurement mentioned in Section B (NIR and Microwave Resonance) are unsuitable for the application as they cannot measure at a low enough moisture level (without data fusion).

Intrinsic fibre-optic moisture sensors have been developed that can withstand high temperatures [32], measure low moisture content [33] and for measuring moisture ingress in polymers [34]. The change in refractive index of a sensing element (Fibre Bragg Grating), at the end of or along fibre-optic cable, as it absorbs moisture is measured. The sensors require the use of expensive analysers to implement.

Capacitive Moisture sensors that are used to measure humidity in air/moisture in oil can be made to withstand the high temperatures and sensors are available that can offer the required accuracy and range at lower temperatures. Measurement of moisture content at high temperatures is much more difficult though, as the dielectric constant of water decreases with increasing temperature [35] and at low moisture content, dielectric permittivity becomes more dependent on the fluctuations of the medium (which are many in a chemical reaction at high temperature) than on water content. Also, the technology capable of the required accuracy for this application cannot yet be manufactured robustly enough to withstand the high temperatures. As a result, the heat-resistant sensors available are accurate to only 300ppm [36]. The more sensitive sensors could possibly be used if they were removed from/cooled during the higher temperatures of polymerisation but manual interaction or having a heat sink on the process is nonideal.

Water Cut meters for measuring the water cut of oil in oil refineries (that also measure dielectric permittivity) are a possible solution as these meters are suited to low moisture contents and high temperatures (e.g. Max. temperature: 232°C, Resolution: 200ppm for 0-1% range [37]). The application is also quite similar as the dielectric constant for polymers is low, as it is for oil, compared to the high dielectric constant (approx. 80) of water.

Due to the difficulty of the application, a clear solution has yet to be identified. The measurement could possibly be achieved through sensor fusion (with an NIR sensor and a capacitive sensor) which has been implemented in industry to achieve better process understanding than that which is possible when considering the sensor outputs independently.

III. Smart Sensor Benefits

This section is concerned with features of commercially available smart sensors. The ISO/IEC/IEEE 21451.x standards, previously known as IEEE 1451.x, define the interface standards for smart transducers [38]. As many papers have demonstrated, the plug and play capability of smart sensors in a 'dynamic network' (a network with all sensors being plug and play) has improved flexibility, efficiency, integrity, reliability and network performance, and reduced cost by automating sensor configuration with self-describing behaviour [39]–[41]. Unfortunately these standards are not recognised outside of the automotive, aerospace and defence industries, a fact which will hopefully change in the near future if more awareness is raised and the Industrial Internet of Things (IIOT) drives activity [42].

A smart sensor is a sensor with some additional functionality it owes to the addition of a microprocessor [43]. Gaura and Newman give an overview to the degrees of this added functionality by which sensors can be classified as being smart, intelligent or cogent [44]. There have been many research papers focused on using the intelligence of smart sensors for sensor calibration/reprogramming [45]–

[47], trend recognition [48], fault tolerance [49], process diagnostics [50] and sensor fusion (temperature/pressure compensation) [51], [52] and distributed data fusion [53].

A. MEMS sensors

Ongoing development in MicroElectroMechanical Systems has brought many improvements to the smart sensor industry. MEMS sensors are manufactured at a micrometre scale and provide compactness, easier process integration, faster response time, low production costs, low power consumption. CMOS logic circuits are often integrated with MEMS sensors to provide the increased performance and reliability related to smart sensors and provide better reliability and adaptability [54], despite the inherent poor signal to noise ratio of MEMS devices [55]. MEMS lend themselves to wireless applications and allow more sensors to be deployed to gather more data and achieve a more detailed, distributed view of a process. Therefore MEMS have an essential role in the IIOT and the industry will experience huge growth [56].

Surface Acoustic Wave (SAW) sensors were investigated for the viscosity application in Section II.D but were deemed unsuitable as they are not manufactured to withstand the high temperatures of the application as of yet. SAW sensors use MEMS technology and can be used to measure a plethora of parameters (chemical vapours, moisture, temperature, force, acceleration, shock, angular rate, displacement, viscosity [57], flow, film characterization, pH levels, ionic contaminants, and electric fields) and offer the potential of being wirelessly powered by energy harvesting approaches [58]. Passive sensors open up the possibility for sensors to form part of the process (e.g. "flow tracker sensors") in order to report bulk properties that are very difficult to measure by static sensors. Future developments in Bulk Acoustic Wave (BAW) and SAW technology to withstand higher process temperatures may see the arrival to the market of a more affordable sensor for the application in Section II.D [59].

B. Algorithm implementation

No transducer is without nonidealities and no process is without interdependent process parameters. Algorithms can be implemented by a smart sensor's microprocessor to account for sensor nonlinearity, convert sensor signals to meaningful process measurements and even for sensor fusion, i.e. temperature compensation in intelligent sensors.

Performing processing at the sensor greatly reduces complexity at higher processing levels. Examples of such intelligence are now found in many commercial sensors and the movement of data processing to the 'edge' of a data acquisition system (decentralised intelligence) has been identified as a recent trend with many benefits [60].

C. Decision-making capabilities

Cogent sensors can use their memory capabilities to identify certain process conditions or trends in process development and signal these events to the rest of the network. Basu et al. detail an example for monitoring oil degradation [61].

D. Remote configuration

Process conditions can change over time, invalidating original calibration parameters / original program operation. Traditionally, recalibration involves performing manual adjustments to sensor hardware which is costly, time-consuming and hazardous in some industrial situations. As the size of sensor networks grows, so does the calibration burden, particularly process downtime due to the calibration procedure and breakdowns. A smart sensor's microprocessor and communications interface can be used to mitigate these costs. A commercial implementation has been demonstrated, that not only performs calibration over the Fieldbus, employing software control for better precision and predictability, but also uses sensor intelligence and some approved performance test to detect when a sensor needs to be calibrated meaning less frequent calibration is required [62]. The problem of trying to create a test system to handle a wider set of sensors is another challenge which is being looked at in the industry [60]. Algorithms/firmware can also be updated via a smart sensor's communication interface, e.g. in the selected sensor in Section II.B [15].

E. Machine learning

The implementation of self-learning algorithms can make sensors truly autonomous. Trends in process conditions can be recognised by algorithms trained by past data. Self-learning algorithms are often implemented as Artificial Neural Networks, a computing paradigm that mimics the human brain, composed of many computing cells or 'neurons' each performing simple operations and interacting with each other to make a decision.

The success of machine learning is down to 'Big Data' (very large data sets) and on the fact that more data beats wiser algorithms. The algorithms produced are tailored to their specific process and focus is taken away from developing the perfect process model meaning less time is spent by process experts manually fine-tuning a model which may have to change anyway. The acquisition and storage of vast amounts of data, the vanishing gradient problem [63] and overfitting (developing over complicated models that fit the training data precisely) are challenges associated with machine learning.

Ortega-Zamorano et al. highlight the difficult task of selecting the right neural network architecture for any particular application and proposes Constructive NNs (CoNNs) that generate networks that grow as input information is received, has a short training period and employs competition between neurons and filtering to prevent over-fitting [45].

Predictive/Adaptive Control involves using process understanding of the effects adjusting of the critical control parameters at any given time on future product quality in order to optimise process development. It involves the monitoring of the process in order to become familiar with typical process conditions. This procedure can be done by process experts (by Model Predictive Control (MPC) [64]) or be automated with the use of machine learning techniques such as Kalman Filters [65], Neural Networks [64], Adaptive Neuro-Fuzzy Inference Systems (ANFIS) [66], [67] or Evolutionary algorithms [68].

Predictive control is usually implemented at higher processing levels, however a smart sensor example is found in the radio wave sensor in Section II.B where the sensor applies Kalman Filters for applications with noncontinuous material flow to achieve more intelligent continual averaging and produce an analog output for process control [15].

IV. Conclusion

This paper has reviewed the availability of sensors for a number of unique and challenging applications in an endeavour to find affordable solutions and reduce the cost of the adoption of PAT. The functionalities provided by smart sensors have eased the challenges faced by the project, particularly in easing sensor integration and maintenance and reducing the complexity of the global control platform to be developed. This reduced complexity of data fusion is very advantageous in a PAT implementation as employing data fusion will further improve measurement performance and enable better process understanding. For example, mass flow can be better calculated using outputs from both the selected microwave sensor and the Particle Size Scatterometer developed by the project. Also, an NIR sensor can be calibrated in real-time against the supporting sensors. Future work in this research will investigate the development of the first layer of a sensor fusion platform: handling multiple sensor protocols and aligning sensor data.

With the proliferation of MEMS technology and more affordable sensors comes the revolution of the IIOT where large sensor networks can be implemented to continually monitor industrial environments with unprecedented levels of detail [60]. Such pervasive sensing applications have been described as a dream by Gaura and Newman, which is getting closer to being realised due to distributed processing, plug and play capability and improving MEMS manufacturing techniques [69]. The concept is in line with the PAT philosophy: to apply, in real-time, process measurements and data analytics, to deliver outstanding process understanding and enable predictive control to operate industrial processes at their optimum, both economically and environmentally, while ensuring high levels of quality.

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