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Cross-sectorial real-time sensing, advanced control and optimisation of batch processes saving energy and raw materials (RECOBA)

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1 Overview of RECOBA technology developments

The knowledge developed in RECOBA has led to development of new products and services. Key results fall into these categories:

1. New and updated real-time process control concepts for batch processes, including Nonlinear Model Predictive Control (NMPC) technology, Dynamic Real-Time Optimization (DRTO) and plant level scheduling algorithms.
2. New and better exploitable sensors that can be commercialised together with the control concepts and as stand-alone products.
3. New knowledge on modelling techniques for batch and semi-batch processes – in particular related to **steel, emulsion polymerization and silicon** production.
4. New algorthms for signal processing and calibration of spectral sensor signals.

The modelling results have been used in the development of control concepts and will to some extent be exploited together with the control concepts. Similarly, the signal processing algorithms are included in the exploitation plan for sensors. All results will be used in scientific teaching and training.

The new technologies have been demonstrated in three industrial case studies.

2 Industrial demonstration case studies

Polymerisation process

A demonstration case concerns semi-batch emulsion polymerisation, which is one of the most complex polymerisation processes. Real-time control of molecular structure and morphology of polymer latex particles has been demonstrated. This is possible using of new hard sensors, novel model based soft sensors and nonlinear closed loop model predictive control.

Liquid steelmaking process

The second demonstration case deals with the chain of batch processes for liquid steelmaking. Here new technologies for inline measurement of the liquid melt temperature were introduced in particular for the batch processes of vacuum degassing and gas stirred ladle treatment. Model predictive control concepts for energy and resource efficient melt temperature control are developed, incorporating the in-line temperature measurement as well as through process models for the whole chain of batch processes.

Silicon refining process

Silicon refining is a high temperature ladle process involving a liquid alloy, refining gas and refining materials. New temperature measurements have been successfully tested in the case study. A solution for model predictive control has been developed for optimal use of refining gas and materials, for improved control of process temperature and alloy composition.

3 Novel acoustic sensors

The University of Cambridge has focused on three different acoustic sensor technologies in the RECOBA project. Each of these technologies, Film Bulk Acoustic Wave Resonators (FBARs), Surface Acoustic Wave Resonators (SAWs) and Angular Acoustic Reflectance Spectroscopy (AARS), will be discussed separately in the following paragraphs.

3.1 Description of the sensors developed

FBAR

The FBAR consists of a piezoelectric thin film (ZnO or AlN) sandwiched between two metallic electrodes. The geometry of the structure will determine the resonant frequency of the device. When a mass binds to the surface, the resonant frequency drops. In RECOBA, several advancements have been achieved in the field of FBARs. Different techniques for the fabrication of transducers capable of operating in liquid have been developed and a patent regarding the use of FBARs with liquid samples has been generated.

SAW

The thin film SAW resonator consists of a piezoelectric thin film (ZnO or AlN) deposited on a silicon substrate and excited using interdigitated electrodes. The geometry of the IDTs and the properties of the materials determine the resonant frequency of the device. When a mass binds to the surface, the resonant frequency drops. In RECOBA, thin film SAW devices with different geometries and designs have been simulated, fabricated and tested.

AARS

AARS technology is based on the use of ultrasonic transducers and receivers. When the travelling acoustic wave interacts with the target particles, the ultrasonic angular reflection characteristics analysed in the frequency domain provide information about the sample. In RECOBA, a system for particle characterisation with ultrasonic backscattering method has been developed by PhD student Isabella Miele.

3.2 Applicability of the new acoustic sensors

FBAR

In RECOBA, FBARs have been successfully used to detect polymer particles. Latest experiments for viscosity monitoring reveal that the polymer particles attach to the surface of the sensor. Therefore it is not possible to distinguish the shift caused by viscosity and the one caused by particle attachment. However, once this challenge is overcome, a probe-based viscosity sensor using FBAR is very feasible.

SAW

In RECOBA thin film piezoelectric SAW sensors have been successfully used to detect polymer particles with a size of 10 µm present in liquid solutions. However, the frequency of operation of SAWs limits their sensitivity and their capability to interact with submicron particles. In addition to this, thin film piezoelectric-based SAWs suffer severe damping of the signal in liquids.

AARS

Detection of particles with sizes below 500 nm require transducers operating at very high frequencies. This increases the acoustic losses up to unacceptable levels. A system for particles smaller than 500 nm does not seem feasible at this point, but detection of particles above that range could be feasible with high sensitivity using AARS technology.

3.3 Potential use in other sectors

FBAR

The FBAR technology is very versatile and achieves sensitivities down to the fg level. In addition to viscosity measurements, different functionalisation layers added to the sensing surface of the device can be used to detect specific targets (e.g. PDMS for Toluene detection) in air and in liquid. It can also be used as a biosensor, to detect different types of biomarkers, proteins, bacteria, etc.

SAW

Even though SAW sensors can be used for viscosity and mass attachment measurements, we do not think they provide any advantages compared to FBARs.

AARS

Potential for detection of particles > 500 nm seems practical and could be implemented with relatively low cost using ‘off the shelf’ components, as well as acoustic particle characterisation in highly concentrated dispersions through backscattering method. In the field of biosensors, AARS technology has great potential for detection of cavitation bubbles in resonance (characterisation of microbubbles of medical ultrasound contrast agents).

4 Raman and TEM sensors

4.1 Description of sensors developed

Raman particle size sensor

Particle size measurement in nanometer scale under reaction conditions is highly challenging. The hypothesis of this sensor is based on the physical model of the effect of polymer chain growth on Raman spectra. This has been observed experimentally and developed into statistical model of particle size predicted from the observed Raman spectra. In RECOBA we have started developing the physical model of the effect of polymer chain growth on Raman spectra. The physical model will then be used to develop a practical sensors in the consecutive projects.

Liquid in situ TEM cell

There are several liquid TEM cells on the market. However, these are not suitable for process studies and in particular not suitable for studies of nanoparticles under controlled flow conditions. In RECOBA we have developed a concept of a microfluidic TEM cell that has a flow channel with specific flow characteristics.

4.2 Applicability of Raman and TEM sensors

The Raman sensor is proposed to be a process sensor: a probe inside a reactor vessel, connected to an instrument via a long fibre optics cable. This is a generic solution that can be applicable to many polymer processes. Within RECOBA BASF is implementing a Raman probe as a sensor for monomer concentrations. It will be a matter of implementing new set of computer routines for signal processing to enhance the output to provide also the particle information.

The TEM sensor is a generic research tool. It will be some time before liquid TEM may become applicable as a process sensor as this requires a significant reduction in the size of high resolution TEM instrument.

However, the output of this work is the potential new technical solution that may become a new service output from the University of Cambridge: design and implementation of bespoke TEM sensors for specific applications.

4.3 Potential use in other sectors

Both sensors would be applicable to any area of technology where particles in nanometer scale are being synthesised or studied. Specifically, this would be directly applicable in production of large molecule therapeutics, in bio-medical research on proteins, in synthesis of metal nanoparticles.

5 Temperature sensor for liquid metal applications

5.1 Description of sensors developed

The main sensor developed has been the Dyntemp system utilising (consumable) optical fibre as a black body radiation absorber and the medium for transferring the energy signal to a pyrometry system. This sensor is used for real time continuous temperature measurement, which was previously unachievable.

The fibre is delivered via a gas feed utilising either existing gas injection systems or it can be supported by a purpose designed gas supply unit. Whilst all the systems are similar in concept the variation between applications requires individual engineering and tailoring.

Additional sensors evaluated were radiation pyrometers and fibre bragg sensors for analysing system temperature/energy losses. In both cases these sensors were considered to be of limited value with respect to long term benefits.

5.2 Applicability of the Dyntemp sensor system

The Dyntemp is applicable for all high temperature molten metal/liquid processes where energy input and temperature critical chemical reactions take place. Proven examples are steel making and silicon production at all process stages after the metal becomes molten. Significant benefits relate to;

- Savings in energy usage through reduction of “superheat” (excess temperature levels generated to prevent early metal freezing/solidification)
- Reduction in refractory wear, which is directly related to temperature
- Reduction in use of alloys and deoxidants as excess temperatures and times also increase the rates of reaction and oxidation of these additions
- Reduction in process times through greater confidence in output data, models and subsequent lower energy inputs

- Increase in yield resulting from shorter process times and lower temperatures meaning less product being lost to oxidation from the atmosphere
- Finally there significant environmental benefits as a consequence of reduction in CO_2 emissions

5.3 Potential use in other sectors

Other examples, but not yet explored include; Glass production, ferro alloy processes, copper production, other high temperature metals and noble metals manufacture.

In the cases of ferro alloys and noble metal we have already received an approach.

6 Technology for Nonlinear Model Predictive Control (NMPC)

6.1 Runtime environment for advanced control and nonlinear estimation

Cybernetica CENIT is a runtime environment for implementing non-linear model predictive control applications. *Cybernetica ModelFit* and *Cybernetica RealSim* are two related tools used during application development.

Cybernetica CENIT – nonlinear model predictive control (NMPC) and Dynamic Real-Time Optimisation (DRTO)

Cybernetica CENIT is a powerful and versatile software suite for nonlinear model predictive control. It uses nonlinear mechanistic models, which makes it well suited for batch control applications creating robust applications.

The models used in CENIT are developed specifically for nonlinear model predictive control. Even though the model captures nonlinear dynamics of the process, there will always be uncertainty in a mathematical process model. The mechanistic structure of the models allows for very efficient compensation of this deviation in CENIT’s estimator algorithms. The combination of mechanistic models with on-line model adaption are crucial elements in the successful application of NMPC technology.

In RECOBA, we have demonstrated development of process models using data from regular operation in order to minimize impact on production.

Process models as well as application specific codes for control of the estimator and controller algorithms are implemented in a *Cybernetica Model and Application*

Component, which is linked into the CENIT system. This separation allows for very specific tailoring in order to best meet specific requirements of the particular plant to be controlled, still allowing the resulting application to build on general, well tested software kernel and a collection of advanced algorithms for nonlinear estimation and control.

6.2 Applicability of the Cybernetica CENIT control technology

Cybernetica CENIT is well suited for control of many demanding processes, such as:

- Processes that are highly nonlinear, requiring nonlinear models.
- Processes where important variables to be controlled cannot be measured on-line but have to be estimated.
- Processes with uncertain and time varying parameters, requiring on-line parameter estimation.

All the batch processes studied in RECOBA fall into these categories.

In RECOBA, applications for batch emulsion polymerisation and batch silicon refining have been developed and demonstrated. Other applications of the technology include:

- Model predictive control of phenolic resin production
- Model predictive control of amino resin production
- Optimization and model predictive control of PVC production
- Model predictive control of polyolefin processes (LDPE, HDPE, PP, various reactor technologies)
- Model predictive control of aluminium electrolysis cells
- Model predictive control of batch refining of other metals (manganese and steel).

6.3 Potential use in other sectors

The technology is generally applicable to the control of a large variety of processes. Cross-sectorial transfer of tools and principles are possible; the technology is developed particularly with that in mind.

The technology puts few limits on the cross-sectorial transfer of results; the limiting factors are market development / market access and lack of engineers with the specific knowledge of other industry segments.

7 Demonstration case: Semi-batch emulsion polymerisation

7.1 Description of new technology

Batch processes are usually operated with time-based recipes. However, due to process fluctuations, e.g. due to changes in raw material quality of changes in the cooling water, and due to process disturbances, the production efficiency is limited with time-based recipes. RECOBA aims at real-time monitoring of these fluctuations and automatically start corrective actions by predictive process models so that the full production efficiency can be achieved.

In a standard emulsion polymerization, the reactor is heated up to the reaction temperature which is maintained during the whole batch. Due to exothermity of the polymerization, the reactor needs to be cooled during the polymerization period. Real-time reaction control with good prediction capabilities helps to improve raw material and energy consumption, shorten batch time, and improve product quality.

For this, new type of inline sensors and predictive models have been developed. These sensors and models enable to visualize and predict important parameters of emulsion polymerizations like e.g. polymer composition, particle size, and morphology.

7.2 Applicability in the sector and beyond

It has been shown for representative emulsion polymerization products, that the developed technology is capable of prediction and controlling polymer composition and product morphology. For other emulsion polymers, model parameters for the kinetic polymerization models need to be adapted and calibrated to the specific system. Also, process models need to be adapted to individual reactors. The methods for these tasks are existing and straight-forward. Also, the morphology model needs to be trained and adapted to other systems. For other products, it will be necessary to describe also other product parameters, but as morphology prediction is one of the most complex part, there will be a broad variety of emulsion polymer systems to which the developed methodology can be used, be it for offline optimization or real-time online control. It needs to be mentioned that fouling is always an issue in polymerization so that this needs to be tested for each product individually. For this, further research might be needed to predict fouling and then develop automatic cleaning systems.

The developed methodologies can also be extended to other polymerization systems, e.g. suspension polymerization. When having bigger particle sizes, particle morphology might be detected with simpler techniques than TEM measurements.

Within the chemical industry, many other batch processes can make use of inline concentration measurement with Raman sensors and of real-time process control. For other particle-based processes like precipitations, the developed inline TEM sensoring is very interesting.

Finally, also other industry sectors like pharma industry can benefit from the developed technology, and RECOBA has proven that real-time process control can be utilized in all batch processes.

The developments have been established and demonstrated at BASF in a lab environment. Demonstration in pilot scale is prepared and will be finalized before due date of the final report of RECOBA.