Deliverable 3.10

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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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D3.10: Application of imaging methods (using optical and electromagnetic methods) in real-time properties measurements across batch processing methods.
A cross-sectorial report.

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<u>Dissemination level</u>

PU	public	☒
PP	restricted to other programme participants (incl. the Commission Services)	☐
RE	restricted to a group specified by the consortium (incl. the Commission Services)	☐
CO	Confidential, only for members of the consortium (incl. the Commission Services)	☐

CONTENT

1 Introduction

Within WP3 sensor technologies have been developed for the three different industrial sectors of polymerisation, silicon production and steel making. To optimize batch processing, development and application of real-time measurement of product properties was in the focus of all sectors. Since the requirements and boundary conditions in the different industries are quite different, adaptations to the specific process are necessary, which will lead to different appearance of the final sensor. Nevertheless, the measurement principles of different sensors might be applicable also to other processes. Especially imaging techniques might be especially flexible, suitable and adaptable to different processes, since they are not in direct physical contact with the process. The cross sectorial applicability is examined for the Raman spectroscopy and the coverage efficiency sensor within this deliverable.

2 Coverage efficiency sensor

The coverage efficiency sensor is an optical sensor designed for real-time monitoring of the energy loss through the cover of the batch. The coverage can change from batch to batch and can have a significant impact on the process. Thus, the sensor can have a significant influence on the accuracy of temperature control of high temperature processing.

The protective housing of the sensor is made of stainless steel with air cooling, which was designed to withstand the harsh ambient conditions above liquid melts in industrial environments of steelmaking. The efficiency of the sensor can be adapted to the radiation power of the cover by introducing an optical filter. The field of view can be adapted to the specific requirements by the size of the aperture in front of the detector. A CAD drawing of the sensor is shown in **Figure 1**.

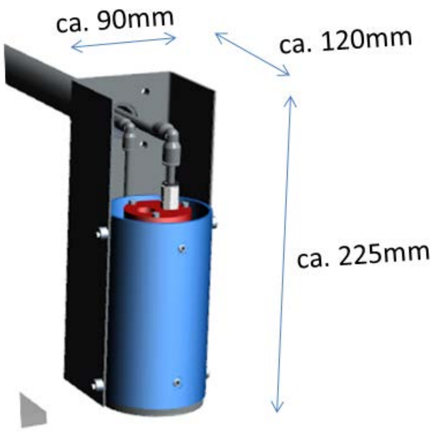

Figure 1: A CAD drawing of the coverage efficiency sensor.

The sensor was tested at a laboratory induction furnace containing liquid steel. In the industrial phase it was also applied and tested in another sector of industrial environments, namely in silicon production. Different batch covers were analysed and the radiated energy was measured for different surface temperatures. It was shown that metallic and oxidic surfaces have distinct differences in the emissivity, which can also be used for discrimination of different covers, see **Figure 2**.

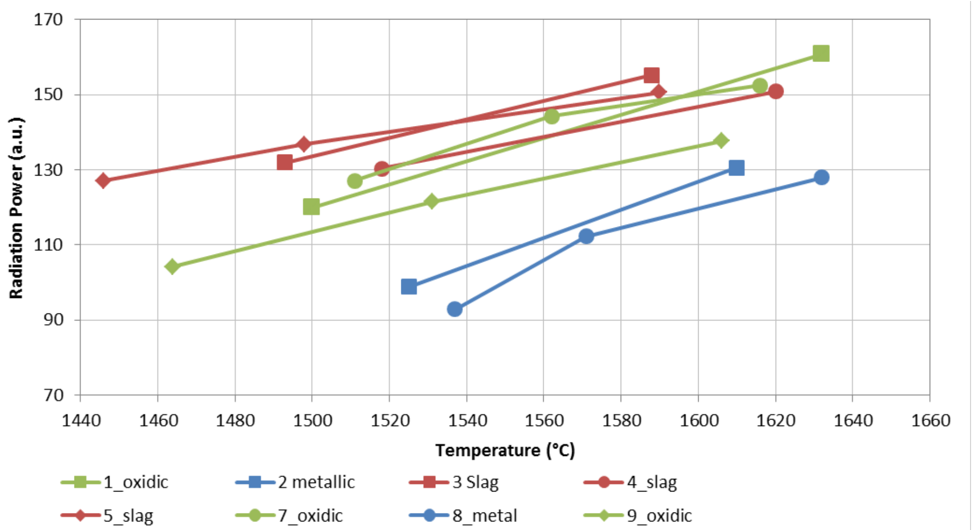

Figure 2: Radiation power of different Si covers versus temperature.

The coverage efficiency sensor is, in principle, also suitable for temperature monitoring in polymerization processes. Although the temperatures involved are much smaller compared to silicon production and steelmaking, thermal radiation is nevertheless emitted from the product surface and container walls. However, the maximum of the thermal radiation is shifted in the spectral range to a longer wavelength. The spectral sensitivity of the detector of 1 to 50 µm covers the spectral range of infrared cameras, which are used to visualize deficits in the heat insulation of houses. Here also the absolute value and the differences of the involved temperature are comparably small.

Thus, the coverage efficiency sensor designed for application in steelmaking and Si production, will in principle also be applicable for monitoring of polymerisation processes. However, the signal to background ratio will be quite small and the noise in the signal will be comparably large, such that decent effort in improvement of the detector electronics and algorithms for data evaluation is expected.

3 Raman spectroscopy

Within RECOBA Raman spectroscopy has been applied for the characterization of the polymerization process. Spectra were acquired and a model was set up to deduce the monomer concentration. Online monitoring of the monomer concentration is important for control of the reaction kinetics. Furthermore, an indirect hard modelling for the monomers of different sizes was set up. It was possible to model the Raman spectra with good agreement. Based on a data-driven PLS model the concentrations of different components could be predicted. The theory of Raman scattering of polymers was extended to describe scattering due to polymer chain structure and length. This theory is the foundation for the new particle size and particle size distribution sensors that will be developed after the RECOBA project.

The principle of Raman spectroscopy is based on measuring the difference between the wavelength of excitation light and inelastically scattered light, where scattering occurs via a variety of mechanisms, such as scattering due to molecular vibrations or due to interaction of light with solids. It is used for analysis of chemical composition in solid, liquid or gaseous phases. It is also used for structural characterization of solids and can be used for measuring temperature, since the shift in the wavelength is temperature-dependent.

There exist a variety of sampling techniques. Within RECOBA project a Raman fibre optic probe was used: the probe contains fibres and a lens designed for a particular type of the sample fluid. For example, a spherical probe head is used in the case of the highly scattering solutions. The probe is placed directly inside a reactor vessel. An image of the probe used in RECOBA demonstration at BASF is shown in **Figure 3**.

Figure 3: An image of Raman fibre optic probe used for in situ monitoring of emulsion polymerization within RECOBA demonstration.

In silicon production the ambient temperature in the melt of more than 1500 °C are much higher compared to the typical temperature of polymerisation of less than 100 °C. The melt is very reactive, which means the probe will not be inert but it will be destroyed during the measurement. Therefore, a conventional immersion probe cannot be used.

It is feasible to use an optical system for remote Raman sensing: a diffused light beam is focused by a long focal length lens onto a sample. In this case there is no direct contact of the probe with the sampled medium. This principle has been used commercially, but not in metal melt applications. The challenges here are the surface cover from slag and a continuous background radiation due to the high heat radiation from the melt.

Product properties which might be analysable by Raman scattering are surface temperature, melt composition and atomic structure of the melt surface. However, at present there are no examples of such analyses in open access literature.

4 Conclusion

The optical sensor technologies developed for real-time sensing of the product properties are in principle applicable also for different industrial sectors. Sometime the appearance of the sensor needs to be adapted to take into account the different ambient conditions. Such as the coverage efficiency sensor developed for steelmaking could be applied also for Silicon production. The Raman spectroscopy is in principle applicable to remote sensing of gas phase or surface chemistry compositions in silicon manufacturing. However, this has not been developed so far to a practical solution and there are no reports on this technology in the open literature.