Deliverable 8.4

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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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Selection of best technology for temperature monitoring of the ladle refractories

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<u>Dissimination 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

The refining of silicon takes place in a ladle made up of a steel casing, a layer of insulating refractory materials and a bottom plug supplying air and oxygen to the molten silicon, as previously described in deliverable D.2.3. Furthermore, the heat loss from the molten silicon at about 1600 °C to the ladle refractory has a significant impact on the overall heat distribution in the system. As shown in deliverable D.8.5, the temperature distribution in the ladle refractory depends on the cycle number. It may take as many as 5-8 fillings of the ladle before the temperature through the ladle has reached a quasi-steady state. This heat loss impacts the overall economy of the process in several ways:

- Heat lost to the ladle refractory could have been used to re-melt silicon fines and B-grade material
- The temperature drop requires oxygen ($Si + O_2 = SiO_2$ is strongly exothermic) to supply heat to the ladle, but this may cause the silicon to be over-refined in order to keep the desired temperature, decreasing the yield of Si.
- Loss of heat causes solid deposits (sculls) at the ladle wall that must be removed mechanically, causing wear and tear to the ladle refractories and reduces lifetime of the ladle and establishes the need for costly repairs and re-building of the ladle refractory.

Addressing these issues brings about solutions like

- Sufficient pre-heating of the ladle refractory
- Monitoring the refractory temperature
- Modelling the overall temperature distribution in the ladle
- Proper ladle design
- Refractory materials selection

In the RECOBA framework, Elkem, in co-operation with the rest of the consortium, will exploit various techniques for monitoring the refractory temperature and use the measured data as input to a heat distribution model. This model will give valuable insight on how to optimise the heat balance/temperature distribution in the silicon refining process and ensure that this batch process is run close to its theoretical optimum.

Measuring the temperature in a reliable and continuous manner is not straightforward for this particular process. The high temperature (1000-1600 degrees Celsius) narrows the available methods. The continuous movement of the ladle complicates any temperature measurement routine. Finally, the temperature should be measured automatically, without need for excessive labour work. This document will describe the options for measuring the ladle refractory temperature for the silicon refining process, and suggest the most promising for further testing in the RECOBA-project.

2 Fundamental methods for measuring temperature

In the following discussion we will make a distinction between *invasive*, *semi-invasive* and *non-invasive* methods (Childs, 2000).

For invasive methods, the sensing probe should ideally be in thermodynamic equilibrium with the medium of interest. The temperature is then determined by the physical parameters of the probe. In reality, there will be some deviation from perfect equilibrium but thermal equilibrium may be closely achieved.

As for semi-invasive methods, the medium of interest is covered by thermochromic coating whose colour changes with temperature are observed remotely.

In the case of non-invasive methods, temperature-dependent properties of the medium is observed remotely, without physical contact. Information about the temperature is acquired from spectral properties of the electromagnetic radiation emitted, reemitted or scattered by the medium. Optical phenomena such as thermal radiation, absorption and emission spectroscopy, Raman and Rayleigh scattering, luminescence and optical interference can be employed.

2.1 Pyrometry

The pyrometer measures the temperature of a surface from some distance. Modern pyrometers can be hand-held devices, which offers a great deal of flexibility. The pyrometer determines the temperature from the spectrum of the thermal radiation from a surface. Hot objects radiate electromagnetic radiation, and the distribution of wavelengths emitted by an object depends on temperature and is given by Planck`s blackbody radiation law.

Pyrometers can operate in different atmospheres in temperatures exceeding 2000 °C. The response time is short which makes these devices suitable for studying dynamic processes or moving objects. However, pyrometers require direct optical access to the hot surface, preventing the use in several applications. For example, the temperature distribution within a closed furnace cannot be determined with a pyrometer. The surface characteristics play an important role, and changes in surface parameters, i.e. emissivity, will lead to significant errors in the measurements. Emissivity changes can result from chemical reactions at the surface, like oxidation.

For the given application, it is clear that pyrometers used in an indirect setup only has limited use, but if a setup and configuration can be found that allows the point of measurement to be located within the refractory, pyrometric measurements offers an interesting option for monitoring the ladle refractory temperature.

2.2 Thermocouples

A thermocouple is an electrical device consisting of two different conductors forming an electrical junctions. A thermocouple produces a temperature-dependent voltage as a result of the thermoelectric effect, and this voltage can be interpreted to measure temperature. In contrast to the pyrometer, thermocouples must be in contact with (or very close to) the object for which the temperature is going to be measured. Thermocouples can typically measure within certain temperature ranges for maximum accuracy. At elevated temperatures, the presence of oxygen or other reactive gases will severely limit the application of exposed TC`s due to chemical reactions with the thermocouple wires. The practical temperature range of standard thermocouples is 0 to 2100 Kelvin. For high-temperature applications, type B can measure temperatures up to 1820 °C. The choice of thermocouple for a given application is based on temperature range of interest, accuracy needed, atmosphere and cost.

For the given problem, the thermocouple method has the advantage of being able to measure the temperature of the refractory directly, which is the preferred setup.

2.3 Fiberoptics

In cases where direct access to the hot surface is not possible, for example within a furnace, fiberoptic techniques offer advantages compared to pyrometric techniques as indicated in section 2.1.

2.3.1 Distributed temperature sensing (DTS)

DTS is a mature technology, well-suited for harsh environments where long lengths are to be monitored. In an optical fibre, light is scattered due to Raman, Brillouin and Rayleigh scattering. These scattering effects result in frequency shifts, which can be either positive (Stokes) or negative (anti-Stokes). The intensity of the anti-Stokes Raman component is highly temperature-dependent, which makes it suitable for temperature detection by measuring the shift of a well-defined laser pulse. Light is pulsed into an optical fibre and by accurate timing information the temperature profile along the fibre is obtained by comparing the two reflected Raman peaks.

The temperature resolution is moderate and depends on the distance from the laser unit and the averaging time. Furthermore, there are different ways of configuring the system, i.e. single-ended and double-ended configuration. The latter is superior in terms of accuracy and reliability.

The spatial resolution is typically about 1 meter, although resolutions down to 25 cm are also available. The spatial resolution is usually a better argument for using fiberoptic fibers rather than temperature resolution, since it offers an infinite number of sensors along one fibre.

The most important limitation of a DTS-system is the fibre material. A widely used material is silica. Since the material properties depend on the temperature, silica is limited to about 700 °C. Suitable coatings must also be applied to the fibre for mechanical protection. For use in the 500-700 degrees range, gold is used as coating material, which gives a high cost of about $200/meter.

2.3.2 Fabry-Perot interferometers

This technique utilizes an optical cavity defined by two partially reflecting surfaces. A light beam passing through the surfaces will set up a temperature dependent interference condition. It is not necessary to use a laser source, in fact, white light interferometry has numerous benefits over laser systems, not only of technical character but also price. Optical fibre based systems incorporating Fabry-Perot interferometers have attracted a lot of attention for high temperature measurement applications. Sapphire fibres are often used as basis due to the high melting temperature compared to silica. In fact, sapphire offers superior high-temperature properties compared to silica and can be used for temperatures up to 2000 °C while silica begins to soften at 1200 °C.

Within the family of FPI`s there are several modifications, but that will not be further discussed here.

2.3.3 Bragg gratings

Fibre Bragg gratings are described by a refractive index modulation written into an optical fibre. Conventional silica fibre gratings can be used for temperatures up to 250 °C, however, gratings that involve physical modifications of the silica fibre can operate up 1000 °C, which is still well below softening temperature. In D3.9 it was shown that these gratings do not degrade significantly at 600 °C for 15 days. Bragg gratings in sapphire fibres offer a huge potential since operating temperatures up to 1700 °C has been demonstrated and still keeping the integrity of the Bragg gratings. However, sapphire fibres are brittle and thus quite difficult to handle.

3 Requirements for temperature monitoring system

There are several factors that will determine whether a specific method is suitable to measure, directly or indirectly, the temperature (profile) in the ladle refractory.

- Cost (consumables, installation, investment, maintenance): subject to current investment requirements and cost/benefit analysis
- Accuracy +/- 10 °C
- Durability: 7 days
- Toughness: must be able to withstand natural ladle movement and minor impacts on the ladle
- Time resolution: One reading per minute
- Useful temperature range: 25-1400 °C, preferably.

The pros and cons of the various techniques as described in chapter 2 is summarized in Table 1.

Table 1. Summary of advantages and disadvantages for various refractory temperature measurement techniques.

Technique	Example	Cost	Suitability	Accuracy	Durability	Temp.range	Resolution
Non-invasive	Pyrometers	Moderate	Low	Moderate	Good	Sufficient	High
(Semi) – Invasive	Thermocouples	Low	High	Good	Unknown	Sufficient	High
	Fiberoptic sensors	High	High	Good	Unknown	Sufficient	High

4 Summary and conclusion

Based on the above discussion it is clear that on the one hand the harsh environment and high temperatures limits possible solutions. On the other hand, some mature solutions exist due to the continuous work in the field of temperature measurements and its fundamental importance for process industry. Furthermore, it can be expected that innovative solutions based on state-of-the-art fibre-optical techniques will continue to offer flexibility for process industry in terms of temperature measurement. Cost will likely be a crucial factor for any possible industrial implementation of refractory temperature measurement sensors in existing refining ladles.

5 References

Childs, G. L. (2000). Review of temperature measurement. *Rev.Sci.Instrum. vol 71, pp 2959-2978*.