**Deliverable 8.7**

EC's Framework Programme for Research and Innovation Horizon 2020 (2014-2020)
Grant agreement no. 636820

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

Start of the project: Jan 1st, 2015
Duration: 36 month

# Selection of best technology for temperature monitoring of solidification process

Due date: August 31, 2016
Actual submission date: August 31, 2016

Lead contractor for this deliverable: ELKEM

Author:

| Company | Name |
|---|---|
| **BASF** | |
| **TKSE** | |
| **ELKEM** | Kjetil Hildal |
| **UCAM** | |
| **RWTH** | |
| **VSCHT** | |
| **PMAT** | |
| **BFI** | |
| **CYB** | |
| **MINKON** | |

Dissimination level

| | | |
|---|---|---|
| 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) | ☐ |

## Table of contents



## Figures



# 1 Introduction

The casting process is extremely important for the product quality of silicon. For production of silicon to the chemical industry, it is important that the cast silicon has the proper microstructure in terms of grain size of the primary phase, as well as a homogeneous distribution of secondary phases and intermetallics. A homogeneous distribution of secondary phases is normally achieved with sufficiently rapid cooling rates. A side effect of rapid cooling rates is that the size of the primary grains of silicon becomes smaller, which can increase the crushing losses during subsequent handling and crushing of the cast silicon. Thus, there exist, at least in principle, an optimal microstructure that is both sufficiently homogeneous, but also keeps crushing losses at a minimum.

Certain secondary phases are more beneficial than others in the customer`s process. The nature and morphology of the intermetallic phases can be to a certain degree controlled by cooling rate and chemical composition, but this is a rather complex field due to the myriad of phases that are known to form in this multicomponent system. For example, it is known that Al-rich phases such as $Al_8Fe_5Si_7$ , $Al_3FeSi_2$, $Al_2CaSi_2$ promote the reactivity in the customer process, here defined as the amount of the gaseous species dimethlydichlorosilane produced per metric ton of metallurgical silicone entering the customer process.

Ensuring a homogeneous quality is perhaps even more important than achieving the perfect combination of secondary phases, as this will make it easier for the customer to adjust his process parameters. Just as for the refining process, a homogeneous quality from the casting process requires good measurements of critical process parameters as well as a dynamic model that can be verified in a proper scale. It must also be possible to adjust process parameters if the predicted outcome is off spec.

In the Recoba project, we will attempt to improve the casting process by developing a mathematical model of the casting process where the importance of critical process parameters can be examined. In particular, we aim at investigating the effect of initial temperature using a combination of a two-dimensional model of the heat transfer in the casting process as well as temperature measurements at the right location. In this document, we will discuss where such temperature measurements should be taken and the type of technology that should be chosen. The criteria will be cost, user-friendliness, technology readiness, robustness, accuracy and adaptability (to existing infrastructure).

Eventually, upon identification of critical process parameters for casting, these parameters will be included in the on-line predictive control system.

# 2 Casting of silicon

## 2.1 Overview of casting in Elkem

Elkem uses different techniques for casting molten silicon and other silicon alloys. Some of these are layer casting, ring molds, stationary chill molds, chill mold carousel and chill mold train. These methods differ significantly in terms of equipment complexity, robustness and operating and investment costs. However, there are certain features that are identical for all of the methods.

- The mold is covered with fine-crushed Si of the same quality to avoid direct contact between mold and molten Si.
- The thickness of the cast can be controlled during casting.
- The temperature of the incoming molten alloy is not measured during the casting process.
- The duration of the casting process is the time it takes to empty the ladle, typically 15-20 minutes depending on the volume of the ladle.
- The chemistry of the alloy is known at the beginning of the casting process, but no further chemical sampling is done during the casting process. In theory, volatile elements such as Ca could evaporate from the alloy in the ladle during casting.

As an example case, we will focus on the casting process currently used at Elkem Thamshavn. The main principles are to a large degree transferable to other variations of silicon casting, as indicated above.

At Elkem Thamshavn, the ladle is transported in a crane after refining is completed. The ladle is hanging from the crane at all times, and a secondary lift is used to raise the bottom of the ladle so that the silicon can pour out of the ladle into the 2 by 2 meter casting moulds covered with crushed silicon (Figure 1), typically 1-5 mm size. Four or five moulds are used per ladle, i.e. the crane has to move sideways from one casting mold to the next, see Figure 2. The thickness of the cast Si is about 100 mm. After solidification is complete, the large plates of solidified silicon are removed with a forklift and transported to a storage bin.

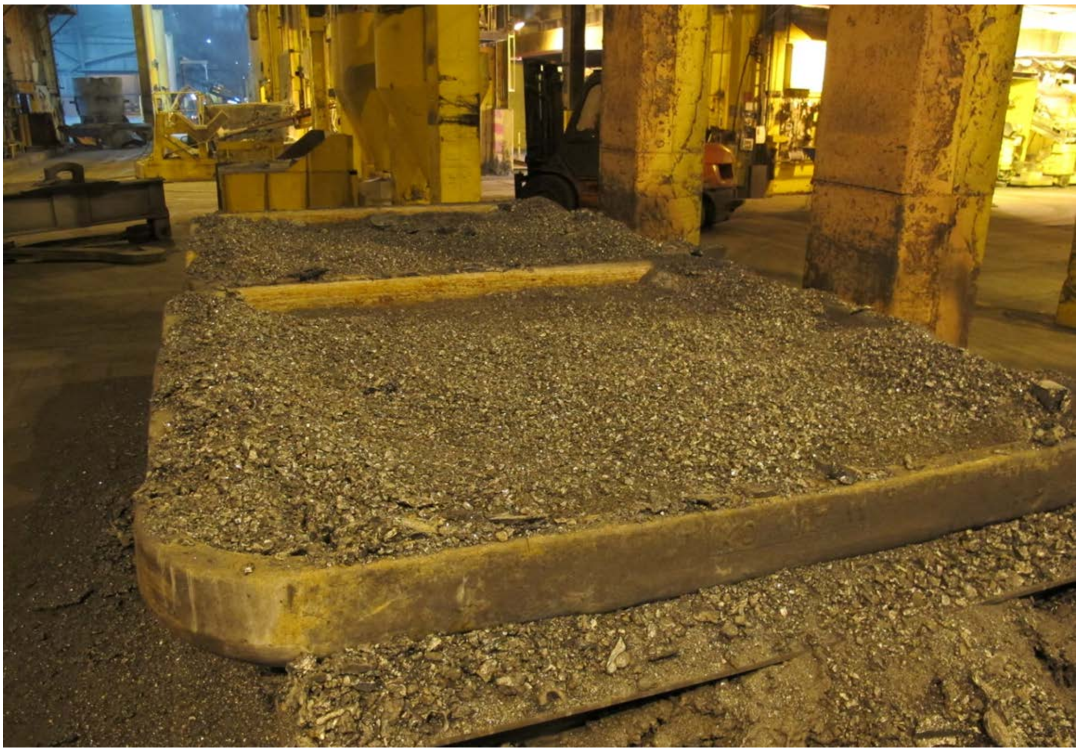

*Figure 1 - Casting moulds at Elkem Thamshavn.*

*Figure 2 - Casting from a crane at Elkem Thamshavn.*

## 2.2 Casting parameters

The development of a mathematical model for casting is a huge task. It is not the intention of this project to develop a complete model that can predict the final microstructure of the cast silicon. However, modelling the heat transfer during casting is relatively straightforward. Assuming that we can at some point establish the proper effect of cooling rate on the final microstructure, we attempt to control the process parameters that decide the cooling rate and the heat removal during casting. Eventually, when the optimal cooling rate (as discussed in the previous chapter) has been determined one can run the process accordingly.

Several process parameters will have an effect on the final product quality, such as alloy composition, temperature, mass flow rate, viscosity, casting thickness. Heat removal from the molten silicon is determined by both contact with the substrate beneath as well as the atmosphere above. Some key properties for the cooling substrate are temperature, size distribution, heat conductivity, thickness, chemistry. The heat removal from the top surface can be by radiation only or increased by convective flow of gas (typically air) or additional cooling such as water mist or water spray. At Elkem Thamshavn, heat removal from the surface is by radiation and natural convection.

# 3 Process measurements

In this chapter, we will discuss potential locations for process measurements that are beneficial for modelling the casting process.

## 3.1 Ladle

The material flow from the ladle to the casting mold is characterized by a specific chemistry, a temperature and a mass flow rate. All of these parameters are in principle dynamic in nature. The change of composition during casting is by experience rather small and is to a first approximation considered constant. In principle, the composition change could be measured with a standard dip sample, taking samples at the beginning and at the end of the casting. However, there are several practical issues concerning access to the melt during casting. Perhaps the easiest way of sampling is to take a sample from the melt as it flows out from the ladle.

The temperature of the silicon leaving the ladle (and thus entering the “casting process”) can be measured in several ways.

### 3.1.1 Dip temperature measurement in ladle

The normal way of using a hand-held probe is technically feasible, low cost and accurate. However, the access to the ladle as it is continuously tilting over is very poor. The intense radiation from the molten bath and ladle effectively prevents the operator to be located in front of the opening. Thus, he can have no direct visual contact with the silicon inside the ladle as the measurement is done. This method has practical limitations due to limited access and multiple issues regarding safety for the operator carrying out the measurement. The long-term strategy for Elkem is to remove all personnel from hazardous areas, including by or near a ladle filled with molten silicon. As such, dip measurements are disregarded as means of obtaining the temperature of the silicon before casting.

### 3.1.2 Continuous temperature measurement within ladle

The development of the Dyntemp® system by BFI and Minkon has opened up the opportunity to measure the temperature in the molten silicon in a far more elegant way than dip measurements. The basic idea is to insert the fiber optic wire with the refining gas through the bottom plug. A temperature can then be obtained throughout the casting process, if desirable. The involvement of humans is kept to a strict minimum as the Dyntemp system is fully automatic.

### 3.1.3 Temperature in outgoing stream

A remote technique is technically feasible in order to measure the temperature of the molten silicon as it leaves the ladle. The main challenges is the presence of silica fumes (see Figure 2) around the tapping stream, which obscures the view. Historically, this has presented problems for commercially available pyrometric-based techniques, such as IR-cameras.

Other measurements that a remote, pyrometric technique are considered inappropriate for measuring the temperature in the outgoing stream of silicon due to the heat exposure, lack of robustness and inadequate infrastructure.

#### 3.1.4 Temperature measurements at casting mould

After the molten silicon has been poured from the refining ladle to the casting mould, the temperature can in principle be measured both from above and at the bottom of the cast.

A remote measurement technique, such as an IR camera, can measure the surface temperature of the cast. An example is given in Figure 3. There are some technical challenges: (i) secure camera position,(ii) clear view of the surface (fuming is going to be an issue), and (iii) the surface emissivity will change with time due to oxidation. The cost of such an installation is also relatively high. On the other hand, the advantages are a no-contact sensor system, proven technology and measurements over a large area, which will yield good data for a mathematical model of the casting process. The IR-technique is possible at temperatures around 1400 C, but it can be expected that the accuracy of the measured temperature is sensitive to calibration.

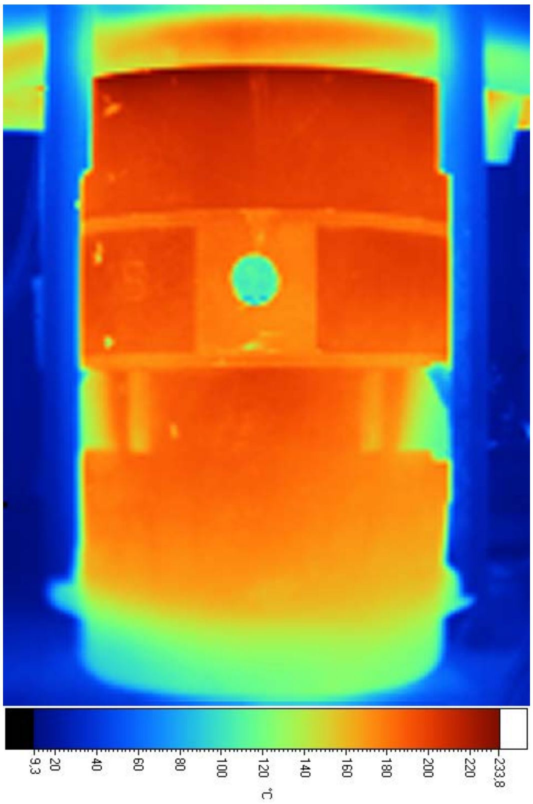


*Figure 3 - Temperature distribution at ladle surface taken with IR camera.*

A second possibility is to equip the casting moulds with thermocouples, positioned such that the tip of the thermocouple is just below the layer of fines protecting the iron mould from the liquid silicon. This technique is frequently applied in laboratory scale casting experiments. It provides for a robust and inexpensive method for accurate temperature measurements. However, in a large scale industrial setting this method has some severe drawbacks. The initial investment and infrastructure modification is considered acceptable, but during normal operations the system need continuous follow-up and inspection. It is likely that the resources currently available in terms of shift labour is insufficient to ensure smooth operation of such a temperature measurement system.

# 4 Conclusion

Based on the current casting processes for liquid silicon, two techniques are considered for further evaluation in the Recoba project.

The continuous temperature measurement system Dyntemp will be tested to verify silicon applicability. This system can be installed on existing infrastructure; specifically the fibre optic sensor can be introduced through the bottom plug with the refining gas. This temperature measurement will give the initial temperature of the silicon cast.

A remote, IR-based camera technique can be used for either measuring the temperature in the stream out from the ladle or to measure the temperature of the cast silicon surface as it cools down in the casting moulds. The stream measurement will give the initial temperature of the silicon, while the surface measurement will give the temperature during casting and as such, this is the preferred application.

Further tests, both in a laboratory scale and pilot scale, will reveal the applicability of such systems to the current silicon production process.