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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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## Pilot scale/industrial scale test of temperature monitoring of solidification process

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| 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) | ☐ |

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# 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 keep 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 to a certain degree by controlled by cooling rate and chemical composition, but this 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.

A review of possible techniques for monitoring temperatures relevant for casting quality was given in deliverable D.8.7. In this document, the results of the pilot scale test of the Dyntemp system applied to molten silicon are presented. The successful implementation of the Dyntemp system to ladle refining would significantly improve process control and result in direct benefits in terms of yield and quality.

## 2 Test of Dyntemp system

Elkem Solar, Kristiansand, operates several large induction furnaces for processing of molten silicon. One of these furnaces bears some similarity to the ladle refining process in that the process temperature is somewhat higher than the desired final temperature. In order to decrease the melt temperature, solid silicon is charged to the batch prior to casting, similar to ladle refining.

### 2.1 Laboratory experiments

A set of calibration runs were performed to match the temperature measured by Dyntemp system and standard thermocouples, see Figure 1 and Figure 2. Since the composition of the melt in the industrial furnace changes due to alloying with Ca, it was decided to calibrate for two different compositions of molten silicon. The behavior was similar to steel except for a small positive offset. Proper system calibrations (Table 1) were done prior to the test in the industrial furnace.

Table 1- Results from labscale calibration runs with the Dyntemp system.

| | Pure Silicon | | Silicon with 3% Ca | |
|---|---|---|---|---|
| | TC | DynTemp | TC | DynTemp |
| Σdelta / K | 0,0 | 7,0 | 0,0 | -1,8 |
| Sigma / K | 7,7 | 6,0 | 2,8 | 3,1 |

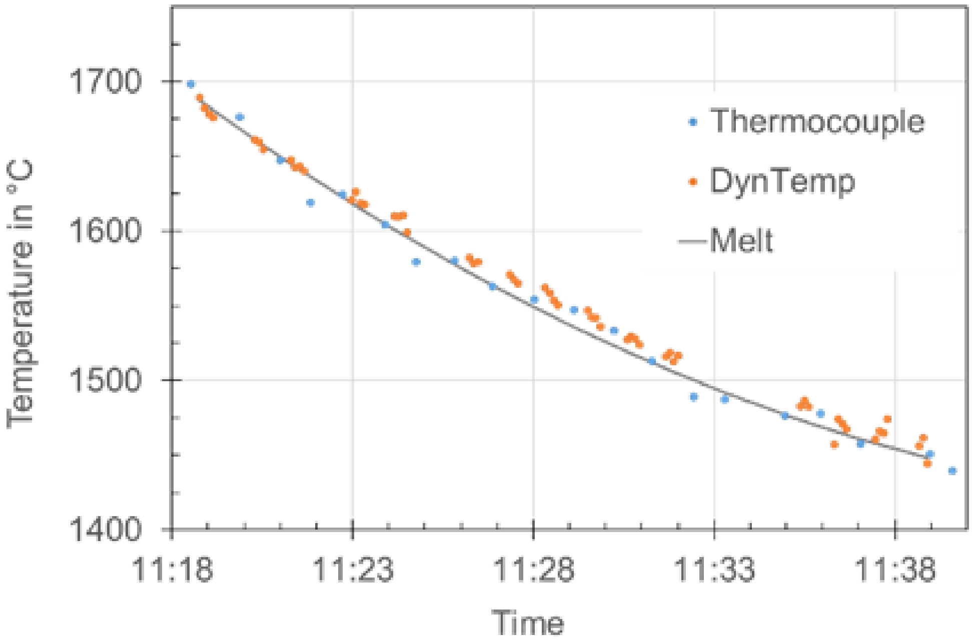


Figure 1 - Calibration trial for liquid silicon.

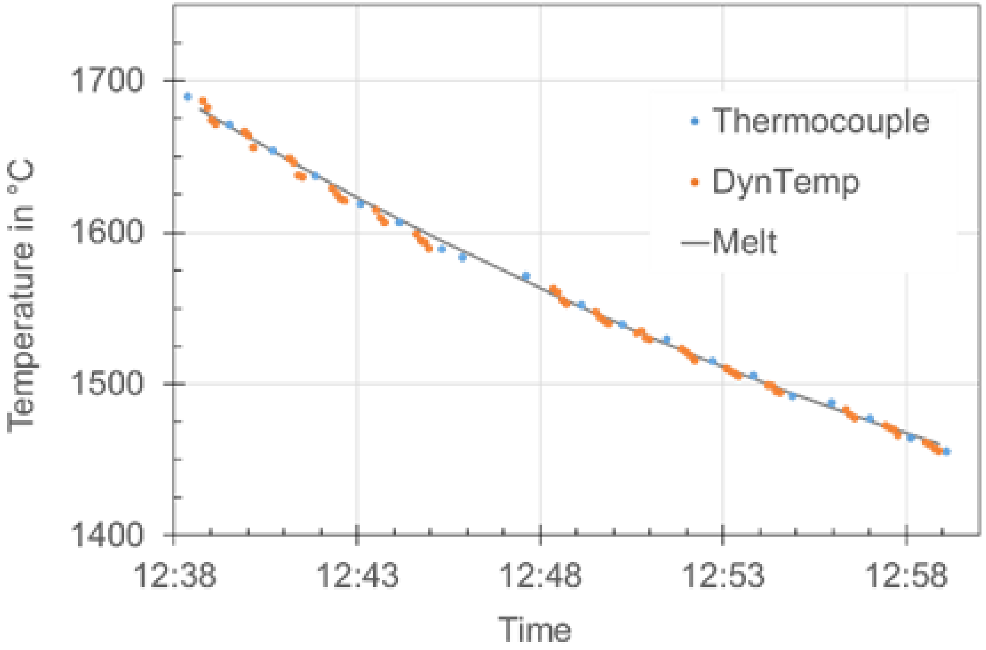


Figure 2 - Calibration trial for Si-3wt% Ca.

## 2.2 Industrial-scale trials

The fiber optic cable was introduced through the graphite stirring lance using a temporary connector. The length of the stirring lance is about 3 meter, introducing the stirring gas near the bottom of the vessel. It is possible, albeit not practical, to change the position of the lance tip by reducing the length. In these trials the original length of the lance was maintained. The trials took place during normal operation of the induction furnace, which resulted in some waiting and shortened measurement runs to accommodate the process operations. The measurements with the continuous fiber optic sensor are shown in Figure 3:

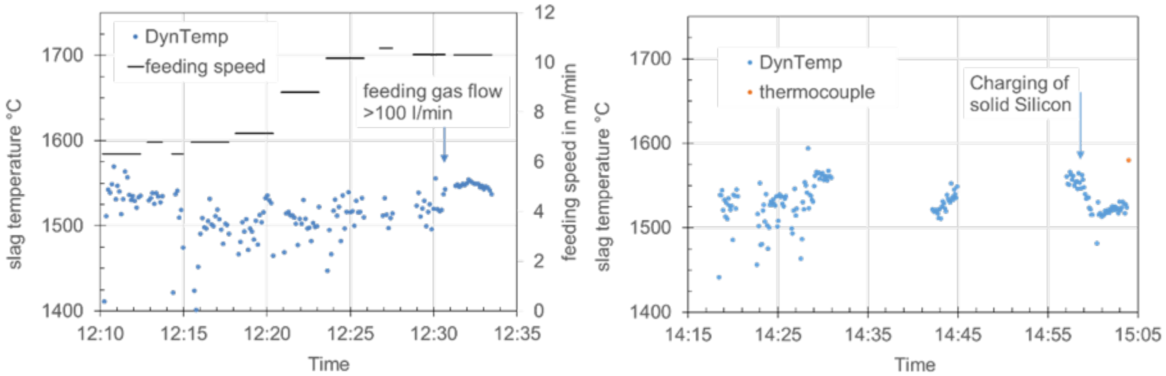


Figure 3 - Results from measurements in industrial scale furnace.

The duration of the measurements was limited by the bottled Argon gas supply (5 liters) with feeding flow gas of 30 liters/min. It was seen that a relatively high feeding rate of gas was required to secure stable temperature readings, up to 100 liters/min as can be seen from the left diagram in Figure 3. At the right diagram, it can be seen that the drop in temperature caused by addition of solid silicon was captured by the system. Also, the temperature was continuously recorded for about 20 minutes once the proper adjustments to feeding gas were done.

## 3 Conclusion

The Dyntemp system was successfully applied to the alloying furnace of Elkem Solar. A continuous temperature reading was obtained for about 20 minutes. Measured process temperatures were consistent with standard dip-measurements. The application of the Dyntemp system will enhance process control for casting purposes as the initial temperature of the melt before it enters the casting unit will be known also during the casting. Today, with standard equipment, the temperature of the melt can only be measured before the casting starts. It is possible that the adaptation of a continuous temperature measurement system to the refining ladle will allow for measuring the melt temperature during the entire casting process.