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STUDY OF PARTICLE SETTLING AND SEDIMENTATION IN A CRUCIBLE FURNACE

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Abstract

Particle settling and stratification in aluminium processing operations are of importance for cast house performance and product quality as they influence important factors such as furnace cleaning frequency, and design of launder troughs and inline equipment.

Analytical analyses of settling generally refer to Stokes' law describing the motion of a solid sphere in a liquid but neglect the dominating factor of thermal convection. A new option to monitor particle concentrations at different melt depths based on LiMCA technology was utilized to study particle settling within AMAP, the Open Innovation Research Cluster for Non-Ferrous Metals at RWTH Aachen University.

This paper reports the initial results obtained with the new LiMCA method in trials in a laboratory crucible furnace. The results are analysed with respect to the influence of particle size on settling kinetics and stratification and are compared to the above mentioned theoretical calculations.

Introduction

The product quality of flat aluminium products is largely influenced by the concentration of non-metallic inclusions transferred with the liquid aluminium into the product. Especially products with thin final gauges like 6 µm foil for aseptic application or products with high surface quality requirement like lithographic sheet require extremely low concentration and a complete absence of large non-metallic inclusions.

Substantial quality improvement has been achieved by the development of sophisticated inline melt treatment technologies [1]. Todays mostly used inline process setup applies gas fluxing with spinning rotor in a degassing unit and mechanical filtration by a ceramic foam filter or a bed filter (or a combination of both). Although the inline treatment has developed significantly in recent years, it is still a proportional reduction of the inclusion concentration and the quality of the product largely depends on the quality leaving the casting furnace [2].

Several options are available to improve the melt quality within the furnace, like gas fluxing with lances or spinning rotors or various salt addition practices. The procedure that is employed in almost all cast houses is settling, the sedimentation of the non-metallic inclusions by gravitational forces, which is proven to be highly efficient [3] at low cost as long as it does not limit the productivity. It is therefore crucial to understand the settling behavior in furnaces in detail to further improve the melt quality and finally the cast house products.

The settling of non-metallic inclusions has been studied in detail by LiMCA monitoring at the furnace exit of stationary and tiltable furnaces in operation [2],[4] and it has been proven to improve the melt quality continuously over several hours as long as no stirring disturbs the process. Otherwise, the LiMCA monitoring in operation generally misses the initial period of the settling curve as it starts after the applied settling time and intermediate stirring, like shown in Figure 1 was used in test charges to evaluate in detail the initial settling kinetic after an homogenization of the melt [2].

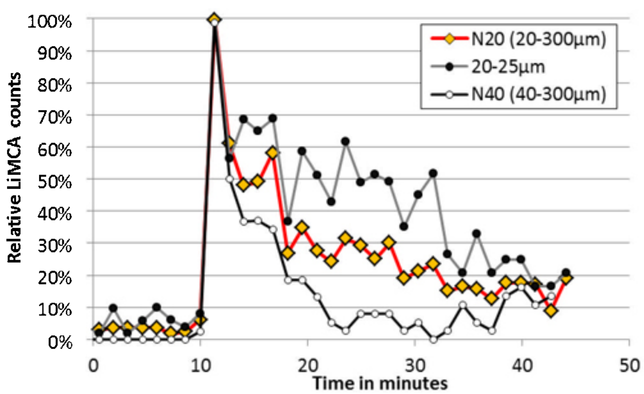

Figure 1. LiMCA settling curve at the furnace exit during casting and intermediate stirring by gas fluxing in the furnace. Note the rapidly decreasing numbers of larger inclusions by the N40 value in the initial stage

First analytical approaches to describe the settling of particles applied Stokes' law reflecting the descent of spherical particles in a quiescent melt based on the force balance between gravitational, buoyancy and drag forces [3]. The analytical data did not fit with the experimental results of the "static" model, which the author considered to be mainly an effect of convective melt flow in the furnace and the difficulty to apply appropriate physical parameters like the appeared density of particle clusters of spherical particles and oxide films [3]. Latter effect has been confirmed within studies regarding the particle size distribution in metal-matrix-composite materials [5].

The melt flow within casting furnaces and its influence on the particle behavior have been studied intensively by numerical methods confirming influences of natural convection [2],[3], particle type and size [2],[3],[4] and furnace shape and size [3],[4].

Sztur [2] used a simple numerical model and superimposed the terminal velocities of spherical particles with the melt velocity for a 35 mt production furnace and a 1 mt laboratory furnace. Firstly, he concluded that natural convection currents were of importance for production scale furnaces but had very little effect on temperature distribution of the laboratory scale furnace due to low temperature gradients of max. 6 °C and melt velocities of 3 mm/s. Secondly, he observed for melt with convection a two-folded particle settling behavior depending on particle diameter d:

- Large particles ($d>d_{Stokes}$) behave in accordance to Stokes' law,
- Small particles ($d<d_{Stokes}$) are transported by the melt flow with a velocity close to that of the fluid;

Here, d_{Stokes} refers to the diameter d where the Stokes velocity of a particle is identical to the velocity of the melt, which is e.g. in the range of 50 µm for a spherical particle with a density of 4.5 g/cm³ and a vertical melt velocity of 1 mm/s. Sztur [2] concluded that the two-folded settling behavior caused two stages in a particle settling curve as visible in Figure 1, an initial stage with rapid decrease due to the settling of large particles (N40 curve in Figure 1) according Stokes' law and slow decrease afterwards due to settling of smaller particles (20-25µm particles in Figure 1) superimposed by the melt flow.

The study reported here was undertaken to improve the understanding on the influence of melt flow on particle settling. For this, a new approach for in-situ monitoring of the particle settling in different depths using the LiMCA technology was applied and compared to analytical approaches based on Stokes' law taking particle shape into consideration and a numerical study of the melt flow within the crucible.

Procedure

A two-stage approach was undertaken to evaluate the settling behavior in a crucible furnace:

1. **Analytical settling model**
   A simple settling model was developed to describe the particle concentration of spherical and non-spherical particles in front of the LiMCA orifice to study the effect of different tube lengths on LiMCA data.

2. **Laboratory test**
   A new LiMCA monitoring procedure with elongated LiMCA tubes was employed to study in-situ the particle concentration in different depths in a laboratory furnace.

Analytical settling model

The motion of a particle in a fluid can be described by the force balance between gravitational, buoyancy and drag force. The particles settle with a constant speed, the terminal velocity, as soon as the relative weight of the particle just balances the viscous drag of the fluid.
For the simplest case of a spherical particle and low Reynolds number (Stokes' regime) the drag force F_D and the terminal velocity v_P can be described by Stokes' law [6]:

$$F_D = 3\pi \cdot \mu \cdot v_P \cdot d \qquad (1)$$

$$v_P = \frac{(\rho_P - \rho_F)\cdot d^2 \cdot g}{18 \cdot \mu} \qquad (2)$$

where ρ denoted the density of particle and fluid,
d the diameter of the particle
µ the viscosity of the fluid

Several approaches are available to modify Stokes' drag force description for higher Reynolds numbers, in the transition and Newtonian regime, for compressible particles, droplets and non-spherical particles [6]. Taking into account that thin and thick Al_2O_3-oxide films are two of the most frequent particle types in liquid aluminium an approximation for these shapes is necessary for an improved description of particle settling. The applied procedure here represents a worst-cast-scenario by an approximation of the settling behavior of the oxide films by the motion of a disc perpendicular to the plane of disc, where the drag force and the terminal velocity can be described by [7]:

$$F_D = 8 \cdot \mu \cdot v_P \cdot d \qquad (3)$$

$$v_P = \frac{(\rho_P - \rho_F)\cdot \pi \cdot d \cdot D \cdot g}{32 \cdot \mu} \qquad (4)$$

with D thickness of the disc

Figure 2 shows the terminal velocity of spherical Al_2O_3 particles (density = 3,95 g/cm³) and disc shape particles with a diameter-to-thickness ratio of 10:1 (e.g. 3 µm thickness and 30 µm diameter) and for 1 µm thick oxide films. The model predicts a significant reduction of the terminal velocity of more than 80% for the films with a thickness-to-v ratio of 1:10 and even higher for the 1 µm films representing thin oxide films.

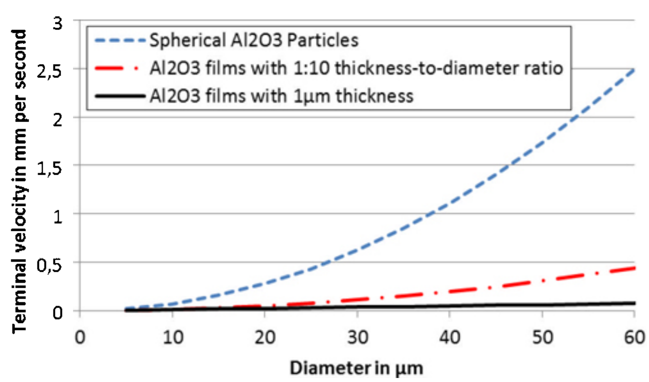

Figure 2. Terminal velocity of spherical and disc-shaped Al_2O_3 particles representing oxide films

In order to transfer the terminal velocity of (2) or (4) into a time and location dependent concentration, one has to calculate the flux of particles of size i leaving the plane x [3]:

$$\dot{n}_i'' = v_i \cdot n_i \qquad (5)$$

Under steady state condition an equivalent flux of particle will enter a plane x from above until all particles of size i vacated [3]. The plane of separation h identifies the melt level where al particles of size i are vacated above. Starting with a homogeneously mixed melt, the plane of separation is given by the melt surface and moves downwards with the terminal velocity of the particles of size i (in Figure 3 shown for two particles sizes).

Within the simple model applied in the current study the area of interest was extended to a (cubic) volume of 6 ml representing the typical amount of melt monitored within one LiMCA cycle (Figure 3). This monitoring window was positioned in the model according to the position of the orifice of the LiMCA tubes based on equations (2+4) in the melt. The concentration of particles of size i within the monitoring window changes over time with the movement of the plane of separation across the monitoring window until all particles of size i settled out of the monitoring area. The total particle concentration in the monitoring window changes then dependent on the monitoring position and particle type mix as the different particles settle with their individual terminal velocity depending on density, size and shape (Figure 3).

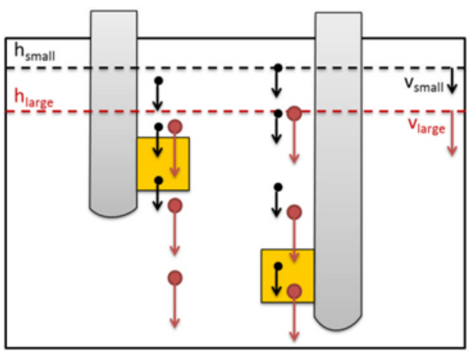

Figure 3. Sketch showing the model to transfer terminal velocity into LiMCA monitoring data

<u>Laboratory test</u>

The LiMCA technique is based on the principle of the resistive pulse / electric sensing zone technique (ESZ). LiMCA monitors the voltage across a 300 µm orifice in a non-conducting glass tube filled with liquid aluminium. As soon as a non-metallic inclusion enters the ESZ the voltage changes, which is then analysed in a post-processing step regarding the particle size and concentration.

The immersion depth of the LiMCA tube and therefore the position of the tube orifice can be adjusted by the LiMCA head in very limited range due to the thermal balance between overheating of the LiMCA head and freezing of the liquid metal in the LiMCA tube. Le Brun and Taina developed a procedure to monitor the concentration of non-metallic inclusions deeper in the melt by a 100 mm elongated glass tube [8]. They confirmed in laboratory and on-site side-by-side tests, that the elongation of the tube and the signal path along the electrode did not influenced the LiMCA particle monitoring.

The above mentioned new LiMCA monitoring procedure was employed in the reported study in a laboratory test setup with two LiMCA units in a resistance heated crucible furnace with 180 kg liquid metal based on 99.8 % pure aluminium ingots (Figure 4).

The two LiMCA units were as close as possible symmetrically positioned and one of the two units was equipped with an elongated tube and the other with a standard tube. In order to avoid any equipment influence on the measurement the positions of the LiMCA units and the elongated tube were switched several times during an extended monitoring campaign. For short sequences both units were equipped with either short or elongated tubes to validate the test setup.

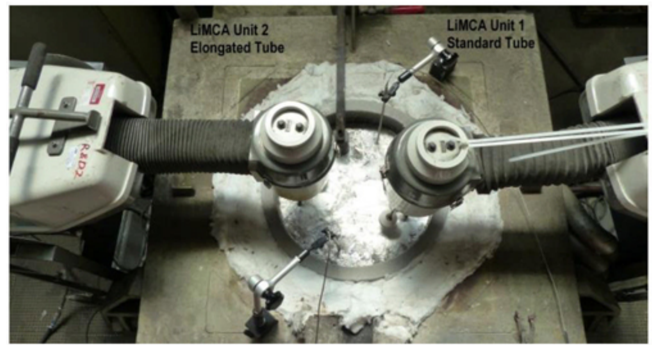

Figure 4. Laboratory test setup using two LiMCA units with LiMCA tubes in different lengths in a 180 kg resistance heated crucible furnace at RWTH Aachen

During the measurement period the melt was manually stirred several times to homogenize the particle distribution within the melt. The subsequent settling of particles was monitored by both LiMCA units about 5 cm and 15 cm underneath the melt surface. The melt quality was additionally monitored by PoDFA sampling from the surface in stirred and settled condition.

Results and Discussion

According to the sequence already introduced in the "Procedure" section the results will start with the "Analytical settling model" to predict settling curves for spherical and film shaped particles. In the next chapter "Laboratory test" these results will be compared to the rate of settling in the laboratory test and discussed in the "Discussion" chapter afterwards.

<u>Analytical settling model</u>

The analytical settling model has been employed to calculate settling curves for spherical and disc-shaped Al_2O_3 particles (density: 3950 kg/m³) in liquid aluminium (density: 2360 kg/m³, viscosity $1{,}25 \cdot 10^{-3}$ kg/m/s) with several assumptions, which will be discussed shortly:

- <u>Homogeneous distribution</u>: The model assumed a homogeneous distribution of all particles as initial situation representing a perfectly stirred melt
- <u>Monitoring position:</u> The change in the particle concentration was predicted for a monitoring window 5 cm or 15 cm underneath the melt surface representing the location of the LiMCA tube orifices in the laboratory tests
- <u>Monitoring window shape:</u> The monitoring window was set to a volume of 6ml and cubic shape. While the volume represented the real LiMCA sampling procedure, the shape was a rough assumption of the

sampled metal in front of the orifice. Nevertheless, the influence of the results should have been limited.

- Particle type: The model assumed only one particle type present (Al_2O_3 in the current study), which clearly did not reflect a real melt. This assumption might be acceptable as Al_2O_3 oxide films are the dominating particle type in clean 1XXX alloys.
- Particle shape: The model was applied for perfectly shaped spherical particles or rigid thin disc particles flowing perpendicular to the plane of the disc. Both setups vary significantly from the particle shapes present in a real melt but might reflect upper and lower limits for the real terminal velocity. It is especially difficult to describe oxide films correctly, which e.g. are flexible or settle down like a leaf falling from a tree.
- Particle size: The particle size of the oxide films in the analytical model was defined by the diameter of the disc, while the particle size in the LiMCA monitoring is calculated from the height of the voltage peak assuming a perfectly spherical particle shape.
- Particle size distribution: Particles between 17 µm and 300 µm have been considered in the analytical model representing the monitoring window of LiMCA. The initial distribution of the particle sizes was fitted to the typical distribution present in liquid aluminium with higher number of small particles and less larger particles. This was done by assuming the start concentration of each 1 µm wide size bin to be 5 % smaller than the 1 µm smaller bin.

$$c_{i,0+1\mu m} = 0.95 \cdot c_{i,0} \qquad (6)$$

The settling curves calculated by the analytical model 5 cm and 15 cm underneath the melt surface are shown in Figure 5 for spherical Al_2O_3 particles and in Figure 6 for disc shape particles with 1 µm thickness. The figure shows virtual N15, N20, N30 and N40 curves by summarizing the concentrations 17-300µm, 20-300µm, 30-300µm and 40-300µm respectively.

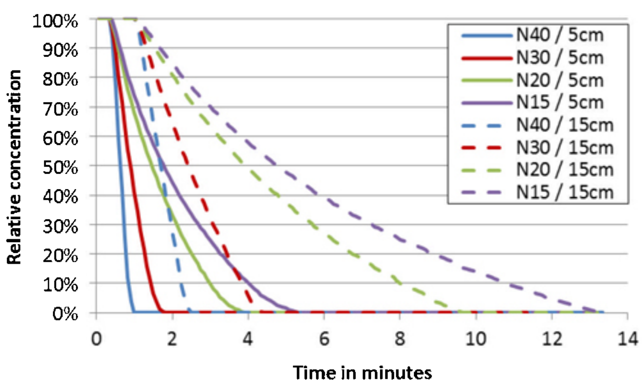

Figure 5. Virtual settling curves for spherical Al_2O_3 particles 5 cm and 15 cm underneath the melt surface calculated with analytical model

All four calculated states (monitoring position and particle shape) show as expected an influence of the particle size on the settling velocity with lower settling rates for smaller particles represented by the delay of N15 or N20 settling curves compared e.g. to the N40 curve. This influence was predicted to be more dominant for spherical particles as a result of the steeper increase of the terminal velocity over the particle size for spherical particles than for disc shape particles as visible in Figure 2.

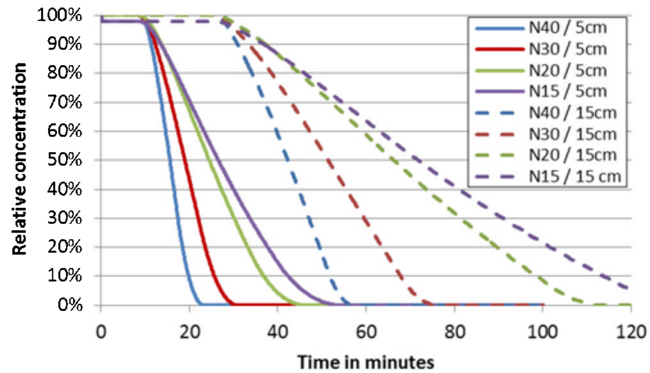

Figure 6. Virtual settling curves for disc-shaped Al_2O_3 particles of 1 µm thickness 5 cm and 15 cm underneath the melt surface calculated with analytical model

All settling curves showed a delay in the start of the particle depletion in the monitoring window resulting from particles present above and travelling into the monitoring window. Such a delay has never been reported in LiMCA monitoring and was likely an effect of the assumption of single particle type and shape present in the melt and the exclusion of convective melt flow in the model. In real aluminium melts larger, denser and more spherical-shaped particles start to settle immediately (at least faster than the LiMCA monitoring) and other particles are transported by the melt flow.

The analytical model predicts an influence of the particle shape on the settling curve as visible by the different time scales in the two figures. This means the settling curve of the melt would be largely influenced by the presence of film-shaped particles. This could also explain the two-stage settling discussed by Sztur [2] and shown in Figure 1 with fast initial settling of the spherical particles and slow settling of the film shaped particles afterwards.

Interesting in Figure 5 and 6 is the influence of the monitoring position on the settling behavior. In both cases larger immersion depths result in an extended delay in the depletion start and lower settling velocities. This is also an effect of particles travelling into the monitoring window from above. The relevant particle reservoir is larger for a larger immersion depth.

Laboratory test

The laboratory crucible tests using the LiMCA technology were accompanied by PoDFA monitoring of stirred and settled melt to identify the particle types present. A generally low particle concentration of < 0.01 mm²/kg was monitored for both situations. Al_2O_3 oxide films were the most frequent particle type with ~ 60 % of the identified particles, followed by TiB_2 particles with 30 %. The latter particles added with the grain refiner to the metal source were small in size and could not be monitored by LiMCA. The assumption of 100 % Al_2O_3 oxide films in the analytical model was therefore reasonable to represent the particle level monitored by LiMCA. Nevertheless, agglomeration products of oxides and borides were not considered in the analytical solution but have been present in the laboratory setup.

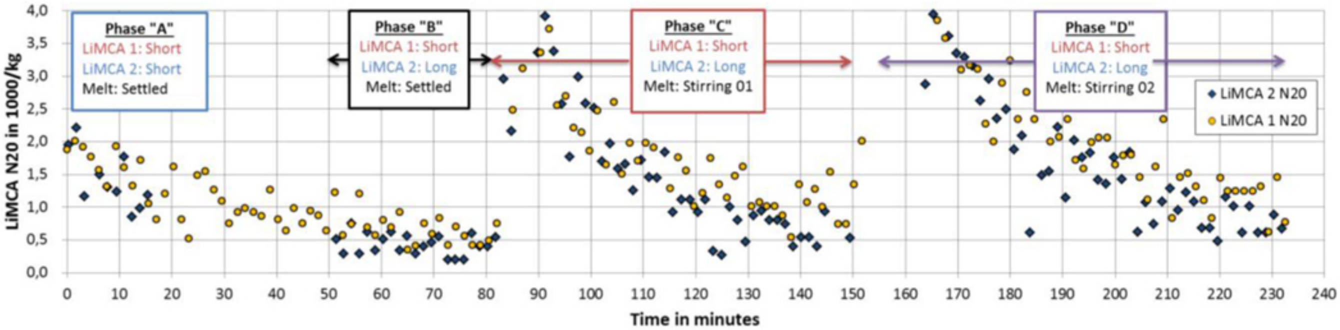

Figure 7. LiMCA N20 run-chart for two LiMCA units with standard and elongated tube; high particle level induced by manual stirring

The continuous in-situ monitoring of the particle concentration by LiMCA in two different heights is shown in Figure 7 over a period of 4 hours. Shown is the LiMCA N20 value reporting particles in the size range between 20 µm and 300 µm. The test was started with standard LiMCA tubes installed on both units in a validation sequence in Phase “A” to evaluate possible influences of the LiMCA units on particle monitoring. It was concluded by the good agreement between the two units, that the test setup was suitable for the intended evaluation. Afterwards an elongated tube was installed on LiMCA 2 and this arrangement was kept for the rest of the monitoring period. Within this trial period the melt was manually stirred after 85 minutes and 155 minutes to homogenize the particle concentration in the melt and the following settling curve was monitored on both positions (Phase “C” and “D” in Figure 7).

A generally low particle concentration was monitored by both units over the complete monitoring period, which agreed with the PoDFA monitoring done in stirred and settled melt. Although the LiMCA monitoring showed some spread of the data it seemed that the elongated tube showed a lower particle concentration in settled melt than the standard tube. In order to compare the settling behavior of the two units in more detail with the analytical solution, exponential curves were fitted to the LiMCA data in phase “C” and “D”. The rate of settling was calculated for both units taking the constant in the exponent of the fitted curve. This procedure was done for the LiMCA N15, N20, N30 and N40 data to quantify the settling behavior of different particle sizes in both positions.

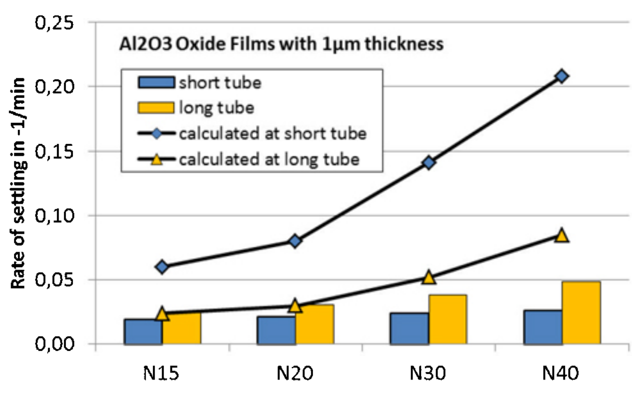

Figure 8. Rate of settling for LiMCA values from laboratory trials in different depths and calculated exponents from analytical model for Al_2O_3 oxide skins with 1 µm thickness

The rates of settling for the four chosen particle size classes on both monitoring positions are shown in Figure 8 in comparison to the respective calculated values from the analytical model for 1 µm thick Al_2O_3 oxide films. The influence of the particle size on the rate of settling was confirmed at both monitoring positions with faster settling of larger particles. A reasonable agreement between analytical model and monitored settling was evident for the elongated tube especially for smaller particles, which indicated suitable assumptions in the analytical model for this position. Nevertheless the influence of the immersion depth found in the laboratory trials was just the opposite of the situation predicted by the analytical model and the rate of settling largely disagreed, which indicated further influences on the settling for this position.

Discussion

A significant effect on particle settling not incorporated in the analytical model was the melt flow in the crucible due to natural and thermal convection. This influence was studied in a numerical model using the commercial fluid flow software Flow3D. The special situation of the chosen crucible furnace with electrical heating from three sides and a cold front face was represented by voids in Flow3D to define different ambient heat separated by baffles used as heat sink.

Figure 9 shows the calculated melt velocities in a vertical cut through the center of the crucible. The positions of the orifice of the two LiMCA units in the plane are shown in Figure 9 by red dots. A symmetrical melt flow was calculated for the shown plane with upwards directed flow close to the crucible walls and downwards directed flow at the center due to the heat input. The calculated melt flow velocity of up to 3 cm/s exceeded the terminal velocity for Al_2O_3 oxide films of all monitored sizes in a large portion of the plane. For these areas the settling of the particles was likely influenced by the melt flow. Even smaller spherical particles would be transported by the melt as already predicted by Martin [3].

The two LiMCA units monitored in the laboratory test in largely different flow regimes as visible in Figure 9. The elongated tube monitored in an area of slow melt velocity. In this area of almost quiescent melt the assumption of the analytical model of an adjusted Stokes’ law was likely suitable to predict the settling of the particles and resulted in the good agreement between the analytical model and the laboratory test. The short tube monitored in an area close to the melt surface with elevated melt velocity. Particles were likely constantly transported by the melt flow into the monitoring window resulting in the large difference between analytical prediction and monitored data

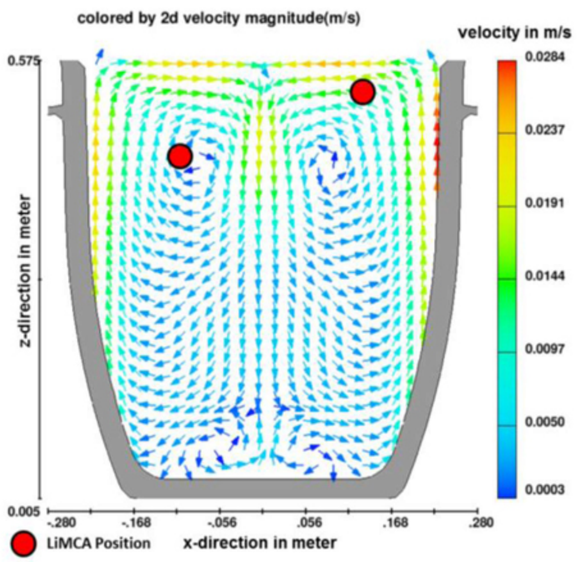

Figure 9. Results of numerical study of melt flow in resistance heated crucible furnace

Summary and Conclusion

The settling of particles in a crucible furnace was studied by an analytical model using Stokes' law including an adjustment for non-spherical film-shaped particles and in-situ monitoring in a laboratory test setup using a newly developed LiMCA procedure with LiMCA tubes in different length [8].

The results of the current study can be summarized in following points:

- The adjusted description of Stokes' law with the assumption of thin disc-shaped particles moving perpendicular to the plane of the disc predicts a more than 80 % reduction of the terminal velocity and lower particle size sensitivity of Al_2O_3 films compared to spherical Al_2O_3 particles.
- The analytical model predicts for the 6 ml monitoring window in accordance to operational experience a significant influence of particle size and shape on the settling curve but in contrast to the observed behavior an influence of the LiMCA monitoring position on the LiMCA data in Stokes' flow regime and a delay of the start of concentration decay.
- The laboratory setup using two LiMCA units with different tube length identified a settling behavior, which generally agreed with the basic considerations regarding the particle size and shape influence on settling and showed a reasonable agreement to the analytical model for disc shaped Al_2O_3 particles at the elongated tube position. Significant lower settling rates were monitored at the standard LiMCA tube position indicating influences by convective melt flow on the particle sedimentation.
- A numerical study of the melt flow pattern in the crucible furnace identified significantly different flow regimes for the monitoring positions of the two LiMCA units used. The elongated tube monitored in almost quiescent melt, where the assumptions of the analytical model reflected the laboratory situation. The melt velocity present at the monitoring position of the standard LiMCA tube exceeded the terminal velocity of a wide range of particle sizes of Al_2O_3 films influencing their settling behavior.

Following conclusions might be drawn from the studies.

- The analytical model used in the current paper can describe basic influences of particle types on settling of non-metallic inclusion in quiescent melt including the influence of particle size, density and shape.
- The new LiMCA monitoring procedure using LiMCA tubes in different lengths and the laboratory setup with a symmetrical arrangement of the LiMCA units allows in-situ monitoring of particle sedimentation and melt flow driven particle transport in a crucible furnace.
- Particle sedimentation in a crucible furnace is largely influenced by natural and thermal convection especially for melts containing larger portions of film-shaped particles (Al_2O_3) or particles with a density close to that of liquid aluminium.
- In the situation of a melt with laminar flow in Stokes' flow regime the position of the LiMCA monitoring influences the reported particle concentration.

Acknowledgements

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