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Experimental Observation of Convection during Equiaxed Solidification of Transparent Alloys

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Abstract. 3D samples of NH_4Cl-H_2O solutions were solidified under defined experimental conditions. The occurring melt convection was investigated by Particle Image Velocimetry (PIV). The occurrence of NH_4Cl crystals was observed optically and first attempts were made to quantitatively measure its number density, size distribution and sedimentation rate by PIV and Particle Tracking (PT). In order to prove the reproducibility of the results several experimental runs with equal and slightly modified conditions were analyzed.

Introduction

Amongst other phenomena simultaneous liquid flow and solid movement is an important phenomenon in equiaxed solidification. Convection in the liquid melt is mainly caused by thermo-solutal buoyancy forces, while the movement of free solid crystals is due to the density difference of solid and liquid under the influence of gravity.

The two phase model proposed by Ludwig et al. has been successfully applied to globular equiaxed solidification [1-4]. The model considers nucleation and growth of equiaxed grains, motion and sedimentation of grains, feeding flow and solute transport by diffusion and convection. It allows the prediction of macro segregations and the distributions of grain size. Whether the Eulerian multiphase model will be successfully applied to the solidification and phase separation processes is strongly dependent on the definition and implementation (through user defined subroutines) of source terms, interaction terms, and other auxiliary terms into the conservation equations. Thus, experiments with the same boundary conditions are essential to verify the numerical predictions.

As a transparent analog for metallic alloys, the ammonium-water (NH_4Cl-H_2O) solution has proved its convenience to study convection and solidification phenomena [5 and ref. therein]. In this study we present a new experimental setup developed to measure particle sizes and velocities together with the convectional flow field and the temperature of the cell wall as boundary condition.

Experimental setup and procedure

Particle Image Velocimetry (PIV) is a whole-flow-field technique providing instantaneous velocity vector measurements in a cross-section of a flow. The use of modern CCD cameras and dedicated computing hardware results in real-time velocity maps. Tracking of single particles, further on referred to as Particle Tracking (PT), can be applied with the same experimental setup as PIV but with different data evaluation software. For the PIV and PT experiments presented in this study we used a double cavity Nd-YAG Laser (frequency doubled, $\lambda = 532$ nm) and the commercial software package "FlowManager", provided by DANTEC dynamics [6]. The principles of PIV evaluation are illustrated in Fig 1, taken from reference [6].

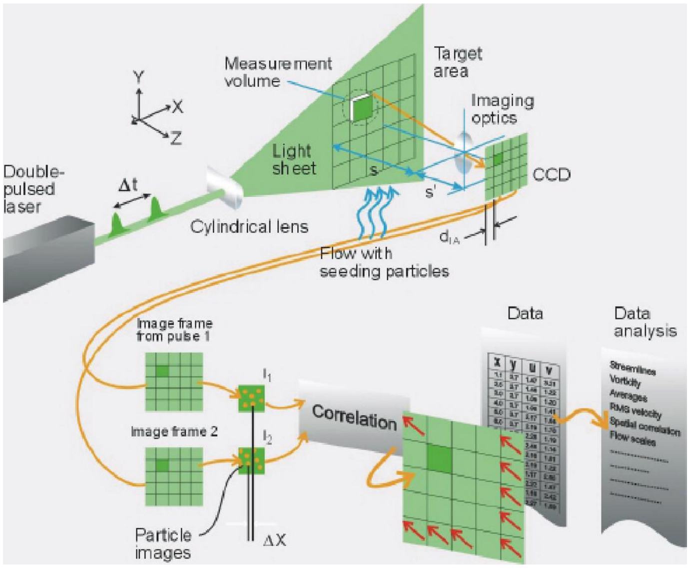

Figure 1. Principles of Particle Image Velocimetry (PIV)

A square measurement cell with 10x10x3 cm inner diameter was designed with three copper walls and two transparent sides made of glass. Front and top view of the measurement cell is shown in Fig. 2 together with a sketch of the setup for stereoscopic PIV. For the experiments presented in this work only one camera was mounted normal to the observation window. The camera lens was covered by a filter so as to let only the laser light which was scattered from particles in the measurement cell pass.

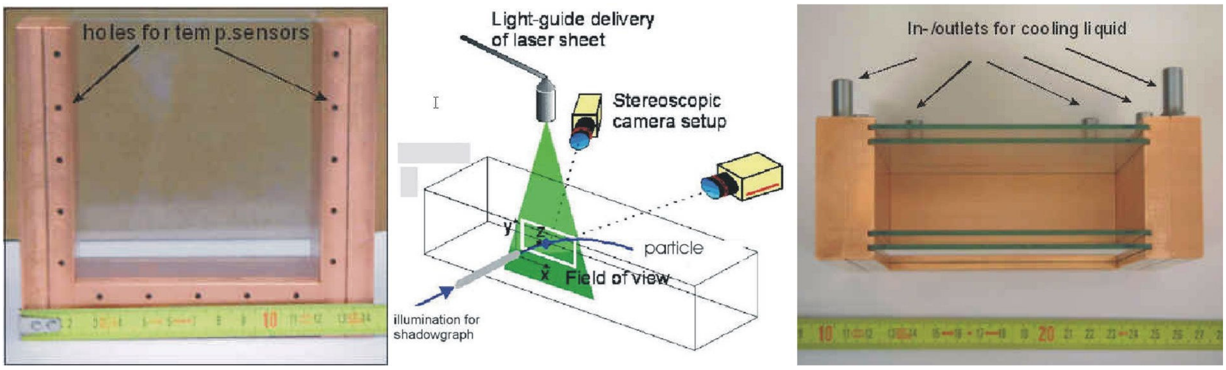

Figure 2. Front and top views of the measurement cell and sketch of the experimental setup.

The three copper walls of this measurement cell can be cooled and heated separately or together in a controlled manner. The coolant used consisted of a mixture of water and automotive anti-freeze. It could be heated/refrigerated between +150 and -35°C and pumped by an external cooling bath (HAAKE C30P) through silicone tubes into the cell walls. Inside the copper walls the coolant followed a meander path which led to a uniform temperature at the inside of the cell walls.

The temperature of the cell walls and for some experiments inside the measurement cell were measured and recorded via a 16 channel thermocouple reader (Stanford SR 630) equipped with NiCr-Ni (type K) thermocouples. For temperature recording a MATLAB based routine was developed to synchronize the PIV measurement with the temperature measurement of up to 16 thermocouples.

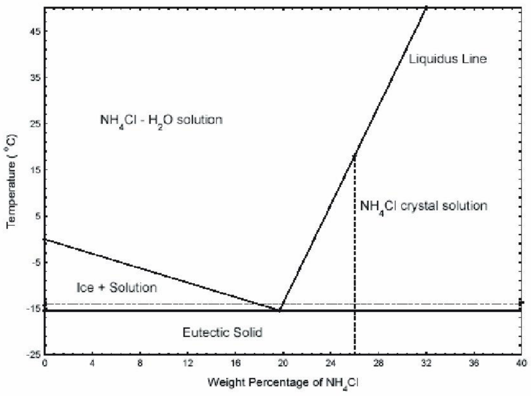

Figure 3. Equilibrium diagram of aqueous ammonium chloride

The equilibrium phase diagram of aqueous ammonium chloride is shown in Fig 3. The measurement cell was filled with a hypereutectic NH_4Cl-H_2O solution (~26 wt%) at 40°C, a temperature well above the liquidus. After 10 to 15 minutes, when cell walls and liquid had reached a homogeneous temperature as measured with thermocouples, the 3 cell walls were equally cooled to 10°C. NH_4Cl crystal formation started when a temperature below the liquidus was reached.

To visualize the solidification process several series of PIV images were taken with the same initial NH_4Cl-H_2O concentration and initial temperature. The measurement series were started when the formation of NH_4CL crystallites had become detectable, i.e. several minutes after the start of the cooling of the cell walls. Subsequently, PIV images were recorded every second until the solidification process stopped, i.e. no change was detectable in the images for several minutes. For real time observation of the process, PIV images were taken every second, the images can be analyzed separately and displayed as a movie after the measurement.

Since the number of small NH_4Cl crystallites is not sufficient to enable good PIV results on the flow field of the liquid melt, polyamide particles (d~ several 100µm) have been added as tracer particles in the experiment which is shown in Fig.4 and discussed in the following section. The influence of these tracer particles on the nucleation process is still under investigation, therefore all the other experiments presented in this work have been performed without additional polyamide particles.

The resolution of the images is given by the CCD camera to 1600 × 1186 pixels (~2 Mpix); the images of an experiment can be exported (individually or as movie) from the measurement software in 8-bit grayscale format [6]. In these experiments the camera was set to a position where it imaged the whole width of the measurement cell including the side and the bottom walls, visible as straight lines in the images. For this camera position the cell width of 100 mm corresponds to 1500 pixels. If one attributes single pixel particles as noise and 2 neighboring pixels as the smallest detectable particles their diameter corresponds to a distance of ~130µm with this camera setup.

Visual observation of convection flow and solidification

Several minutes after the cooling had been initiated the formation of crystals was observed at the cell walls, forming a columnar zone. Figure 4 shows PIV results measured during this stage of the solidification process. The convectional field in the measurement cell in this stage showed two major vortices with downward streaming at the cold walls and upward streaming in the center of the measurement cell. The regions at the sides of the measurement cell had to be excluded from the PIV evaluation because the crystallization at the walls led to misinterpretations in the PIV correlation. To illustrate the convectional movement observed in the real time movie of the measurement, larger arrows (of random size) have been added in these regions, whereas the small arrows represent the PIV results.

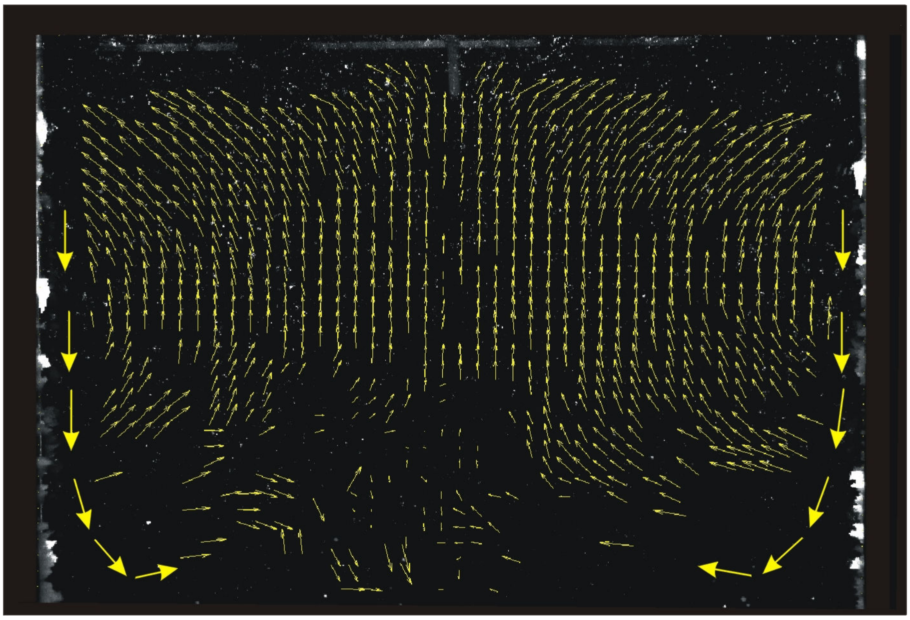

Figure 4. PIV results on convection before the development of plumes

Fig. 5 shows a sequence of PIV images taken during the solidification process. The measurement series was started when the formation of NH_4CL crystallites became clearly visible with the naked eye ~5 minutes after the cooling of the cell walls had been initiated. While the packing zone continued growing at the walls, small NH_4Cl crystallites which followed the movement of the liquid melt were observed in the inner region of the cell.

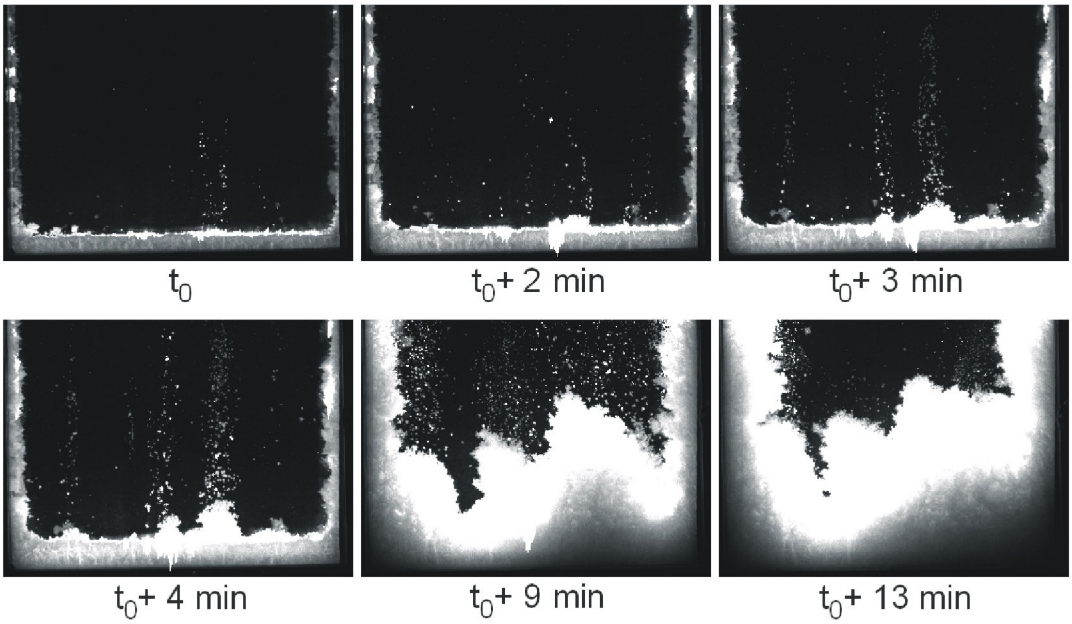

Figure 5. Sequential images of the solidification process

Further on, plumes were observed to develop in a random manner and it was found that between 3 and 5 chimneys formed in the measurement cell every time the experiment was preformed. In the presented experiments 5 chimneys can be distinguished with an average distance of 1.5 cm from each other and the cell walls, best seen on the image in Fig. 5 which was taken 4 minutes after the start. A comparison of the images shows that the number and size of crystallites increased with cooling time until all the ammonium chloride solidified, as shown in the last image of the sequence. Small particles followed the liquid flow until their size reached a value where gravity dominates and the crystallites sink and settle at the bottom. The growth of the crystallites and their movement can be observed best in and around the plumes.

To show the movement and growth of a single NH_4Cl crystallite in a plume, a section around a plume has been cut out the sequence of PIV images (from the same experiment as shown in Fig. 5) and the sections have been put together in Fig 6. The time at which each image was taken is labeled at the top and the position of a single particle (which has been followed in the live time movie) has been marked with a bright circle. In the first 5 images of the sequence, i.e. during the first 26 seconds, the particle moved up in the plume, during the following 22 seconds (images 5 to 8 in Fig. 6) the particle floats at more or less the same height. About 50 seconds after the start of the sequence the particle started to sink until it coalesced with the packing zone.

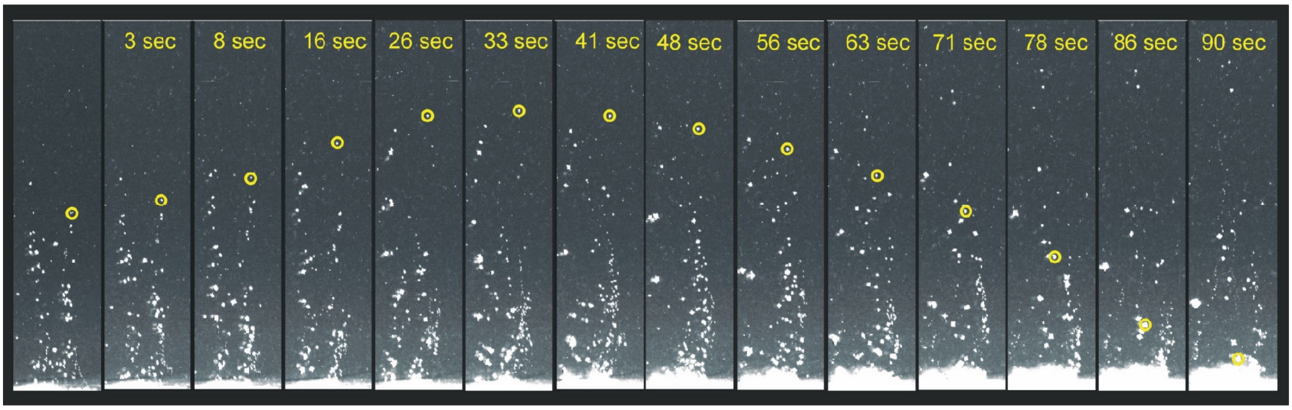

Figure 6. Following the movement and growth of a NH_4Cl crystal in a plume

Quantification of the PIV images

As described in the experimental section, the PIV experiments result in 2 Mpix grayscale images which can be used for further analysis. The standard PIV correlation software demands a high number of tracer particles evenly distributed over the detection region. Two images taken with a predefined time step and the correlating software result in a velocity vector for every detected particle, as shown by the smaller arrows in Fig.4. This “standard” PIV application does not implement particle sizes and leads to misinterpretation in the case of small particle numbers. DantecDynamics recently supplied a different correlation software called “Particle Tracking” which allows to evaluate velocity vectors for small particle numbers (not shown in this work) [6].

In order to extract information about the particle size distribution from the PIV images, we developed a MATLAB based routine which enables the user to extract a region of interest from the images. Next, one can set a threshold value to distinguish between the well illuminated particles in the laser light sheet and the ones in the background (see Fig. 1 and Fig. 2). In that way the routine imports the desired images evaluates the number and size of the particles in the desired region of the measurement cell and exports them in a table.

Figure 7 shows the result or this home-made routine applied on the first 4 images of Fig 5. The graph in Fig. 7 displays the number of detected particles versus the detected particle size. Each point of the graph corresponds to the number of particles detected in a size interval of 10 pix^2.

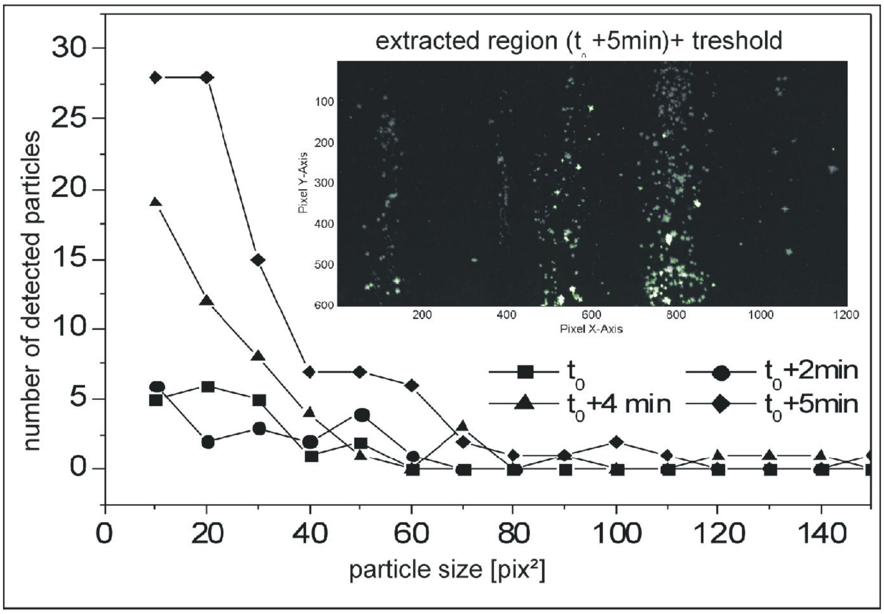

Figure 7. Extracting particle number and via setting a threshold

The detection region chosen for the evaluation is shown as an insert in Fig. 7. In this way we excluded the influence of the solidification at the sides and bottom of the measurement cell. As mentioned in the visual observation section, both number and size of the equiaxed crystals increased with time. At the start (t_0) only 19 particles were detected compared to 460 particles found 5 minutes later (t_0+5min). The average size of the particles at t_0 was 31 pix², which corresponds to ~0.1 mm², whereas the average size at t_0+5min was 203 pix² which corresponds to ~0.9 mm² at this camera setup.

Summary and outline

This study has demonstrated the use of Particle Image Velocimetry and Particle Tracking in a solidification experiment. We have illustrated that the convectional flow field, particle numbers and sizes can be quantified with this technique. In combination with the initial concentration and the measured temperature of the die these quantities will be taken as benchmarks for numerical simulations. The influence of different types of tracer particles on the solidification process will be the aim of future experiments as well as changes of the die geometry and the initial alloy composition.

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