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A Front-Tracking Measurement Technique for In-Situ Columnar and Equiaxed Structure Growth with Controlled Solidification

T. Hughes¹, S. McFadden^{2,1} and A. J. Robinson¹

¹ Department of Mechanical & Manufacturing Engineering, Trinity College Dublin, Dublin, Ireland.
² School of Computing, Engineering, and Intelligent Systems, Ulster University, Magee Campus, Londonderry, Northern Ireland BT487JL, United Kingdom

E-mail: xxx@xxx.xx

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Abstract

Growth rate is an important consideration in solidification as it influences features of the micro and macrostructure such as morphology and the columnar to equiaxed transition. Thus, accurately quantifying the growth rate is key to understanding the solidification process as well as verifying numerical simulations. In this work, an experimental apparatus has been developed to investigate in-situ directional solidification using a transparent analogue material; neopentyl glycol-35wt%(d)camphor. The facility enables the control of the mushy zone growth rate as well as the imposed temperature gradient during solidification, similar to Bridgman type furnaces. Image processing techniques were developed for automated tracking of position and growth rate of the solidification front using background subtraction on photographic image sequences captured in experiments. Growth rate measurements using this technique and the traditional method (liquidus isotherm speed) have been benchmarked against manual measurements of position and growth rate. Results show that automatically tracked growth rate compares well throughout experiments with errors less than 2.7%. Contrastingly, isotherm speed was in error up to 19%. Three columnar and two equiaxed morphologies were investigated and solidification fronts were characterised for their growth rates and thermal conditions.

Keywords: Solidification, crystal growth, front tracking

1. Introduction

Control of the solidification process, the development of microstructures during solidification, and the impact upon the resulting material properties continue to be an essential yet technologically challenging task for developing high-quality cast, welded and additively manufactured parts. Solidification conditions, such as temperature gradient and cooling rate, directly influence the microstructure and grain texture in the as-cast solid and therefore have a direct effect on the quality and mechanical properties [1], [2].

Directional solidification refers to the case where the solidification occurs along a known or preferred direction. Under laboratory conditions involving cylindrical crucibles, the solidification is controlled by managing the thermal conditions so that the growth direction is aligned with the crucible axis. Different crystal morphologies can arise depending on the process parameters imposed during the solidification process. The different crystal morphologies lead to different crystal textures and properties at the macroscale of the casting.

One such texture is called a columnar grain structure (see figure 1) where elongated grains (i.e. with high aspect ratio)

are aligned in a common direction. Another texture is called an equiaxed grain structure where grains have a lower aspect ratio (typically less than two) and random orientation. Columnar structures have been shown to provide superior creep resistance in metal alloys operating at elevated operating temperatures [3] while fine equiaxed structures provide a higher yield strength as described by the Hall-Petch relationship [4], [5].

A Columnar to Equiaxed Transition (CET) can occur during solidification when transient thermal conditions exist. For example, casting conditions that initially favour columnar growth (high temperature gradient or low growth rate) can change to favour equiaxed growth (low temperature gradient or high growth rate) giving rise to a CET. Growth rate and temperature gradient are therefore important considerations in studies on CET.

In particular, numerical models that attempt to simulate the thermal conditions for CET require deeper information about the process from experiments [6]–[9]. However, the validation of numerical simulations can be problematic due to a lack of detail regarding boundary conditions in the experimental work. Furthermore, traditional metallographic analysis of experimental samples is termed ex-situ or post mortem analysis; that is, the analysis takes place after the sample has been processed and has cooled to room temperature. In-situ analysis of the solidification as it occurs is preferable for model validation. In-situ experiments are, however, difficult to achieve due to the opaque nature of metals in the visible spectrum as well as the opaque ceramic furnaces that are used for metal processing. One approach is to use x-ray technology to observe crystal growth [10], but this approach is limited to thin samples and alloys that have sufficiently different x-ray absorption rates to give good image contrast.

Another approach to in-situ analysis is the use of transparent organic materials that solidify in a similar way to metals [11] and which form columnar and equiaxed microstructures. These materials are referred to as transparent model alloys or transparent analogues. Provided that transparent crucibles are used, they facilitate real-time visualisation of solidification processes using optical video microscopy. Experiments with these analogues have advanced knowledge and understanding in solidification research (see reviews [12] and [13]). One such transparent analogue is the binary system of Neopentyl Glycol and (d)Camphor (NPG-DC). Recently, microgravity experiments with this system provided benchmark data for numerical models independent of the gravity-driven phenomena such as buoyancy-driven flow and sedimentation [14]–[17]. Future work on NPG-DC alloys is planned onboard the International Space Station for investigating CET [18].

Figure 1 shows a schematic of directional solidification for columnar growth (left) and equiaxed growth (right). The schematic shows the liquid zone and the mushy zone (mix of

Figure 1. Graphical representation of directional solidification defining important positions and regions for columnar growth (left) and equiaxed growth (right). L, Liquid zone; M_C , Columnar mushy zone; M_E , Equiaxed mushy zone; $M_{E,I}$, Incoherent equiaxed mush; $M_{E,C}$, Coherent equiaxed mush; T_L , equilibrium liquidus isotherm; SF, solidification front; v_L , equilibrium liquidus isotherm speed; v_{SF} , growth rate.

liquid and solid), denoted L and M, respectively. Additionally, for the case of equiaxed growth, figure 1 delineates the equiaxed mushy zone into coherent and incoherent zones, $M_{E,C}$ and $M_{E,I}$, respectively. In equiaxed dendritic solidification, the nucleated equiaxed crystals can move freely in the melt and this is called the incoherent mushy zone. When the free-to-move dendrites sediment and grow to impinge on one another, the dendritic network becomes coherent (i.e. the dendrites' motion becomes restricted). Figure 1 shows the solidification front (SF) of interest in this study at the top of the columnar mushy zone (left) and at the top of the coherent equiaxed mushy zone (right). The SF proceeds upwards with a growth rate, v_{SF} . The equilibrium liquidus isotherm, T_L , advances with a velocity, v_L , called the equilibrium liquidus isotherm velocity.

Relationships between growth rate, morphological transitions and dendrite characteristics are well documented for transparent analogues [13]. Figure 2 shows how controlling the temperature gradient, and the cooling rate facilitates growth rate control in a static crucible. Two heaters either side of the adiabatic baffle zone (sometimes a heater and heat exchanger) maintain a hot and cold side thus providing a

Figure 2 Graphical representation of the assumed alignment of the mushy zone. Temperature profiles assumed during isotherm speed measurement at two time instants; t_1 and t_2 , and schematic of the adiabatic baffle zone (right) corresponding to time t_2 .

temperature gradient, G (shown in figure 2). By subsequently applying a cooling rate equally throughout the baffle zone the solidification front grows upwards and, following an initial transient period, the isotherm speed, v_I , provides an estimate of the growth rate as follows:

$$\frac{\Delta z}{\Delta t} = \left(\frac{\Delta T}{\Delta t} \right) \left(\frac{\Delta z}{\Delta T} \right) \quad (1)$$

$$v_I = -\dot{T} \left(\frac{1}{G} \right) \quad (2)$$

Additionally, the temperature profile can be used to give a basic estimate of the position of the mushy zone. Based on equilibrium conditions, the mushy zone may be assumed to exist between the liquidus and solidus or eutectic isotherms, as appropriate. In this case, the bounding isotherms (i.e. the liquidus and solidus) are typically calculated based on the original composition of the alloy. However, under steady growth conditions, even when the liquidus isotherm speed and the growth rate are well-matched, the liquidus isotherm position and the boundary of the mushy zone may not match. This mismatch may arise due to the crystal tips, which define the extent of the mushy zone, growing at temperatures below the equilibrium liquidus temperature (i.e. a tip undercooling must exist). Misalignment between the growth direction of columnar tips and the heat flow direction typically causes a greater mismatch between liquidus isotherm position and the crystal tips. In the case of an equiaxed mushy zone, nucleation of crystals occurs at some finite level of undercooling deeper within the mushy zone and the resulting coherent equiaxed growth front does not align with the moving liquidus isotherm. If the growth conditions are nonsteady due to transient thermal conditions, then the growth may deviate further from the set conditions. Mota et al. [19] have shown that after initial pulling in a Bridgman furnace, the growth rate can take 40 minutes to 2 hours to reach a steady state with a succinonitrile and camphor transparent analogue. It is clear that the application of the equilibrium liquidus isotherms to define the extent of the mushy zone can be problematic. This has implications regarding the interpretation of the physics as well as for validating simulations.

An experimental apparatus has been developed in this investigation that implements directional solidification using the NPG-DC transparent analogue in a transparent sapphire crucible. This allows for visualisation and videography of the mushy zone development as well as the grain textures. An image processing technique has been developed to track the position and speed of the solidification front with time, providing an averaged position and velocity of columnar tips in the bulk direction of growth. The image processing technique also tracks the growth rate for fully equiaxed growth by tracking the top of the coherent mushy zone. The results are compared with liquidus isotherm speed measurements and

manual reference measurements of position and growth rate. Importantly, the growth rate measurements are obtained with well-defined and accurately quantified experimental boundary conditions. The experiments thus provide accurate thermal input data for simulation research in the form of measured boundary conditions and output data related to the crystal morphology. This data will allow for code validation to take place.

This contribution aims to describe the application of a unique directional solidification test rig that includes thermal instrumentation and control in combination with automatic image processing to track position and calculate growth rates for columnar and equiaxed crystal growth fronts. The objectives are (i) to provide technical details on the development of the new experimental facility and to provide thermal calibration data (ii) to describe the development of a new image processing technique for tracking the position and growth rate of the solidification front (iii) to benchmark the newly developed automated tracking technique against manually assigned growth rate and front positions, and (iv) to compare the automatically calculated growth rates to the manually assigned growth rates and traditional isotherm speed estimation.

2. Description of Materials & Methods

2.1 Apparatus

Figure 3 shows a technical illustration of the experimental apparatus. It consists of a vertical sapphire tube of 450 mm length, with an inner diameter of 8 mm and an outer diameter of 10mm. Sapphire was chosen as the crucible material because it is optically transparent, allowing visualisation of the top of the mushy zone and, due to its high thermal conductivity (≈ 40 W/mK), facilitates heat transfer to and from the material with low (< 1 K) temperature difference across the crucible wall.

A resistively heated coiled nichrome wire is wrapped tightly around the outer tube wall with a layer of thermally conductive silicone to provide adequate thermal contact and electrical isolation. This region acts as the heat source in the upper section of the crucible. Electrical power is supplied to the resistive coil with an Automatik EA-PSI 8360-10 T DC power supply. The power supply is controlled with a dedicated computer (see figure 4), which is connected with an EA-IF-U1 interface card. Heat is extracted from the lower section with a 170mm-long tube and shell type heat exchanger. A precision chiller (Julabo A40) in series with a pump supplies chilled water to the tube and shell heat exchanger with initial setpoint temperatures followed by controlled cooling rates ranging 0.2 - 1 K/min.

Figure 3 shows the baffle zone between the heater and the chiller. A camera (Canon EOS 1100D, 12 Megapixel sensor)

Figure 3. Technical illustration of apparatus with highlighted view of an acquired image. In the upper right, the baffle zone is shown up close with solid growing directionally upwards

with a variable lens (Navitar 1.4 x – 13 x zoom) mounted perpendicularly to the sample vertical axis records image sequences of the solidification process within the dashed region of the baffle zone shown in figure 3. Thermocouples on the wall of the sapphire tube in the baffle zone monitor the temperature and are used to determine the thermal gradient, G , and cooling rate, $\dot{T}$, in the region.

End caps at either end of the sapphire crucible, shown in figure 3, serve both as fixtures of the test section to an aluminium frame (not shown) as well as providing a pass-through seal to and from the crucible. This facilitates an airtight connection of a pressure transducer along with conduit piping; one for adding and removing the model alloy and one to allow outflow when filling the crucible. At different heights along the wall of the sapphire tube, pairs of 0.5 mm diameter

blind holes have been drilled, diametrically opposed to one another, into which T-type thermocouples have been inserted with a small amount of thermal adhesive for non-invasive wall temperature measurement. Two thermocouples embedded in the wall of the crucible in the cooled region provides the temperature, denoted T_C , indicated in figure 3 and 4. For internal temperature measurements, an 80 μm diameter T-type thermocouple with Teflon sheath and exposed junction is optionally traversed into the crucible from the top of the rig through an O-ring seal. The internal thermocouple is housed in a 3mm outer diameter stainless steel tube which is used for positioning and extends 30 mm from the end of the tube into the baffle zone. A clamp fixture, shown in figure 3, holds the thermocouple in place and allows for accurate positioning of the probe in the baffle zone.

2.2 Experimental Control

Control of the temperatures of the heater and chiller, T_H and T_C respectively, facilitate the application of nominal temperature gradient, G , and cooling rate $\dot{T}$ in the baffle zone, and hence isotherm speed can be calculated via Eq. 2. When the heater and chiller temperatures are decreased at the same rate, the cooling rates $\dot{T}_{A1}$ and $\dot{T}_{A2}$ in the baffle zone reach steady-state after which averaged values for cooling rate, isotherm speed, and temperature gradient are recorded.

Figure 4 shows a schematic of the control setup for the experiment. For each experiment, T_H and T_C were initially kept constant at a pre-set value. T_C was kept constant by controlling the coolant temperature of the chiller and subsequently ramped down at a given cooling rate between 0.2 and 1 K/min for the duration of the experiment using the controls on the precision chiller. The power supplied to the electrical heater was modulated with a Proportional and Integral (PI) feedback control loop using the heater temperature, T_H , as the process variable. The feedback control was run with LabView® triggering the power supply using an Automatik EA-IF-U1 interface card to signal the electrical power supply. In this way, T_H could then be ramped down at the same cooling rate as $\dot{T}_C = dT_C/dt$ in order to maintain a constant temperature gradient $G = (T_H - T_C)/z_{HC}$, with z_{HC} being the axial distance between thermocouples T_H and T_C . Thus, for a fixed G specified by the user, the setpoint T_H for the controller is calculated as:

$$T_H = T_C + Gz_{HC} \quad (3)$$

2.3 Experimental Procedure

This work investigated directional solidification occurring vertically upward from the cooled solid zone following initiation of a controlled ramp down change of T_C and T_H .

Figure 4. Schematic diagram of experimental facility

To investigate conditions leading to a columnar to equiaxed transition, the cooling rate $\dot{T}$ and hence liquidus isotherm speed, v_l , were varied, and the temperature gradient, G was kept constant for all experiments. In a given test the morphologies were characterised as being either fully columnar or fully equiaxed after review of the optical camera footage. Subsequent tests were then performed for different values of applied cooling rate $\dot{T}$, and hence isotherm speeds.

For the present experiments, the test material above the cooled heat sink region was initially heated such that the material in the heated section and the baffle zone region was in the liquid phase. T_H and T_C were then adjusted such that the solidification front was just below the camera's Field of View (FoV) and was maintained constant for a period of at least 40 minutes to allow the solidification front to reach the liquidus isotherm. At this point in the experiments, ramp down cooling was initiated, and optical imaging was commenced. Image

processing techniques to track the solidification front, described in section 2.4, confirmed if the growth rate had achieved steady state.

As mentioned, T_C was set by controlling the precision chiller. Simultaneously, T_H was computer-controlled to decrease at the identical rate so as to maintain a constant axial temperature gradient, G , in the baffle zone (see Eq. 3). The camera clock, being synchronised with the computer clock, facilitated direct visual correlation of the observed solidification morphology with the instantaneous thermal environment. As this was a relatively slow process, the frame rate of the camera was set to capture images continuously at 0.5 fps. Table 1 lists the imposed process parameters implemented for T_H and T_C while tabulated results for temperature gradient, cooling rate and isotherm speed measurement have been determined using thermocouples seated in the wall of the sapphire tube adjacent to the solidification front and in the baffle zone (T_{A1} and T_{A2}). Eq. 3 was used to estimate that the isotherm speeds were between 1.9 and 9.3 $\mu\text{m/s}$.

Table 1 A list of the thermal processing parameters imposed during experiments

Test	Cooling Rates (K/min)	Temperature Gradient (K/cm)	Isotherm Speed ($\mu\text{m/s}$)
I	0.2	18	1.9
II	0.4	18	3.7
III	0.6	18	5.6
IV	1.0	18	9.3
V	1.0	18	9.3

2.4 Automatic Front Tracking Algorithm

An image processing technique has been developed for automatically tracking position and growth rate of the advancing solidification front using the photographic image sequences captured during the experiments. The technique employs background subtraction where the grayscale pixel intensities of a frame under evaluation deviating significantly

from those of the background model are selected for further processing steps. Several background models have been established in the literature for computer vision applications [20]–[23]. In the current application, the scene is controlled under laboratory conditions, and the camera is static relative to the crucible with limited background motion. Frame differencing background subtraction is used to threshold for any newly formed solid between two input frames. Figure 1 shows a schematic of the baffle zone with the solidification front (SF) and reference coordinates. The automatically tracked position and speed at time t_1 are denoted, z_{t1} and $v_{f,t1}$ respectively. The SF and tracked position z_{t1} is located at the top of the coherent mushy zone, also referred to as the coherency point.

The flow chart in figure 6 summarises the algorithm used by the tracker to determine the position of the SF. Firstly, high-resolution images are cropped to the width of the sapphire crucible and downsampled by 80% resulting in 6000 x 4000 rgb images being converted to 725 x 800 grayscale images. An anti-shake algorithm is used to preclude any vibrations arising in the microscopic video sequence due to vibrating lab equipment or general lab activity. This is an important step to ensure that the measured growth rate is independent of camera disturbances. To that end, all images in a given sequence are registered to the first image of the sequence. Feature points are detected using the minimum eigenvalue algorithm developed by Shi and Tomasi [24] and matched with the first image in the video sequence to determine any translation or rotation that needs to be applied to the image. Following these pre-processing steps, the tracker algorithm is implemented. First, two sequential grayscale images (725 x 800 pixels) corresponding to times t and $t + \Delta t$ are loaded and denoted I_t and $I_{t+\Delta t}$, respectively.

Figure 6. Flow chart of auto tracker algorithm

Figure 5. Images illustrating steps in the tracker algorithm; (a) 725 x 800 binary image $I_{s,i}$ from image subtraction and thresholding, (b) vertical pixel distribution of binary image (after 11 point moving average), and (c) tracker position overlaid onto image in between original input images.

Frame differencing background subtraction is then applied according to Eq. 4 as follows:

$$|I_{t+\Delta t} - I_t| > b \quad (4)$$

The inequality holds true if, at a given pixel, the absolute value of the difference between the two images is greater than the threshold value, b . Figure 5 (a) shows an example of the binary image, B , resulting from the inequality in Eq. 4. Summation of pixels in each row of the binary image is then calculated and smoothed with an unweighted 11 point moving average to provide a vertical distribution of change between the original input images. An example of the resulting smoothed distribution, d_k , is shown in figure 5 (b). The index k , where d_k is a maximum, selects the exact vertical pixel location of the solidification front for the image between the two original input images corresponding to time $t_1 = t + \Delta t/2$. Figure 5 (c) illustrates the SF position superimposed on the image in between, i.e. I_{t_1} .

The position corresponds to the middle row of the block of 725×11 pixels with the highest number of pixels passing the threshold. The pixel resolutions for each experiment are measured using ImageJ software and, knowing the diameter of the crucible, original images give a resolution of approximately $0.36 \text{ px}/\mu\text{m}$ and correspondingly $\sim 0.072 \text{ px}/\mu\text{m}$ for downsampled images.

Depending on the value of the threshold, b , unwanted pixels may appear in the foreground if the threshold is too low. Contrastingly, an insufficient amount of pixels may pass the threshold if the value, b , is too high. As detailed in figure 6, the algorithm implements two simple criteria to select the threshold at a given time: (i) if the minimum value of the distribution, d , is not 0 the threshold, b , increments by 0.01 and the algorithm returns to the image subtraction step with an increased threshold. This ensures that a region of at least 11 rows in the foreground mask consists entirely of pixels failing the threshold b ; (ii) the maximum value of the smoothed distribution d_k must be greater than 100 to ensure that a prominent peak has been obtained. If the second condition is

also satisfied, the location of the maximum value of the smoothed distribution then passes as the position of the solidification front. In the scenario where the second condition is false, the algorithm assigns a placeholder value to the position for that iteration and continues to the next timestep of the video sequence.

The position determined directly from the image sequence, z_{t_1} , is subsequently smoothed using a Savitsky-Golay filter with a window of length $N_k/4$. The resulting filtered data denoted z_{f,t_1} , is then used to determine the tracker growth rate, v_T , using Eq. 5.

$$v_T = \frac{z_{f,t_1+1} - z_{f,t_1-1}}{t_{t_1+1} - t_{t_1-1}} \quad (5)$$

The tracker algorithm has been implemented in the MATLAB® 2020a environment. Additionally, the anti-shake algorithm has been employed using the MATLAB® implementation of the minimum eigenvalue algorithm and feature matching algorithm.

2.5 Reference Position and Growth Rate Measurements

Manual position and growth rate measurements were used as a reference to assess the accuracy of the tracker as well as isotherm speed. A total of 40 images, equally spaced in time throughout each experiment, were selected. For each of the 40 images in a given experiment, the boundary of the top of the coherent mushy zone or the top of the columnar mushy zone was drawn manually. Figure 7 shows a close-up example of the manually drawn boundary for (a) fully columnar growth and (b) fully equiaxed growth. The boundary was first defined by a series of manually placed vertices connected by straight lines. A second set of vertices were generated with equal spacing along the length of the boundary and the mean height of the second set of vertices was used for determining the average height. The manual growth rate measurements were then determined by central differencing of the manual position and time data.

Figure 7. Close up of manual trace and tracker position; (a) fully columnar growth, (b) full equiaxed growth.

2.6. Thermal Calibration

The propagation of uncertainty in measurement is detailed in this section. Expanded measurement uncertainties have been established with a normal distribution and a coverage factor $k = 2$. All T-type thermocouples have been calibrated using a reference Platinum RTD which has a standard uncertainty traceable to national standards of 0.025°C . For convenience, the maximum temperature measurement uncertainty established during calibration of T-type thermocouples has been applied as a suitable uncertainty for all thermocouple measurements. Summation in quadrature of the independent sources of uncertainty has provided a standard uncertainty of $u_T = 0.065^\circ\text{C}$ with expanded uncertainty $U_T = 0.13^\circ\text{C}$.

The propagation of measurement uncertainty has been determined as standard using Eq. 7 as follows:

$$u_c(y) = \sqrt{\sum_{i=1}^N \left(\frac{\partial f}{\partial x_i} \right)^2 u^2(x_i)} \quad (7)$$

where there are N input measurements x_i , each having a standard uncertainty $u(x_i)$ and sensitivity coefficient $\partial f / \partial x_i$. The output measurement is denoted y with combined uncertainty $u_c(y)$. Table 2 summarises the sensitivity coefficients and standard variances for each measurement. This includes standard variance for mean temperature measurements by two opposing thermocouples at a given height along the crucible wall (see figure 3); the uncertainty for a temperature difference ΔT ; the uncertainty of the temperature gradient measurements from thermocouples seated on the wall of the sapphire tube; and uncertainty of liquidus isotherm speed.

The thermal environment of the adiabatic baffle zone has been further characterised using internal temperature measurements. The apparatus in figure 3 shows an internal thermocouple which is optionally inserted into the crucible via an O-ring seal (not shown) on the upper end-cap. Axial temperature profiles have been measured by traversing the internal thermocouple along the centerline axis of the

adiabatic baffle zone providing temperature and position data. Before taking a temperature profile, the setpoints of the heater and heat exchanger, T_H and T_C , were adjusted to achieve a given temperature gradient and maintained for 40 minutes such that the SF was just below the camera FoV. For each externally applied temperature gradient, the internal thermocouple was traversed to 5 discrete heights in the adiabatic baffle zone, and corresponding positions for the thermocouple bead were determined in ImageJ.

Additionally, a range of internal temperature gradients, G_i , were compared with the temperature gradient measurement on the crucible wall, G_e , for providing calibration data in the form of $G_i = b_0 + b_1(G_e)$, where the intercept and slope are denoted b_0 and b_1 , respectively. The linear relationship provides a non-invasive measurement of the temperature gradient in the liquid melt with associated measurement uncertainty during tests.

The average cooling rate was determined by a linear regression curve fit of time-temperature data acquired during experiments on the wall of the sapphire tube and is denoted $\dot{T}$. The standard error of the slope of the curve fit yielded a standard uncertainty $u_{\dot{T}}$. Finally, the standard uncertainty of isotherm speed, u_{v_I} , is determined using Eq. 8.

$$u_{v_I} = \sqrt{\left(\frac{\partial v}{\partial G} u_{G_i} \right)^2 + \left(\frac{\partial v}{\partial \dot{T}} u_{\dot{T}} \right)^2} \quad (8)$$

3. Results and Discussion

Figure 8 shows the internal temperature profiles acquired for thermal calibration. Linear regression fitting shows the temperature profiles in the adiabatic baffle zone approximate well to a linear model with coefficients of determination in the range of 0.977 to 0.999. A range of internal temperature gradients, G_i , and corresponding temperature gradient measurement on the crucible wall, G_e , provide output and input variables to the calibration equation, respectively. Figure 9 shows the results of the linear regression fit for the internal temperature gradient as a function of external temperature gradient measurement. The calibration curve provided corrected internal temperature gradient measurements with a standard uncertainty, denoted u_{G_i} , of 0.64 K/cm .

Table 2. Summary of sensitivity coefficients and standard variances for propagation of uncertainty in thermal measurements.

Measurement	Input x_i	Functional Relationship $f(x_i)$	Sensitivity Coefficients $\frac{\partial f}{\partial x_i}$	Standard Variance $u_c^2(y)$
Mean Temp.	T_{z1}, T_{z2}	$T_z = \frac{T_{z1} + T_{z2}}{2}$	$\frac{1}{2}, \frac{1}{2}$	$\frac{1}{N} u_T^2$
Temp. Difference	T_{a1}, T_{a2}	$\Delta T = T_{a2} - T_{a1}$	1, -1	$u_{T_{a1}}^2 + u_{T_{a2}}^2$
Ext. Temp. Gradient	$\Delta T, \Delta z$	$G = \frac{\Delta T}{\Delta z}$	$\frac{1}{\Delta z}, -\frac{\Delta T}{\Delta z^2}$	$\left(\frac{1}{\Delta z} \right)^2 u_{\Delta T}^2 + \left(-\frac{\Delta T}{\Delta z^2} \right)^2 u_{\Delta z}^2$
Isotherm Speed	$G_i, \dot{T}$	$V_I = \frac{\dot{T}}{G}$	$\frac{1}{G}, -\frac{\dot{T}}{G^2}$	$\left(\frac{1}{G_i} \right)^2 u_{G_i}^2 + \left(-\frac{\dot{T}}{G_i^2} \right)^2 u_{\dot{T}}^2$

Figure 8 Plot of internal temperature vs position for cases of external temperature gradient.

Figure 9 Plot of internal vs. external temperature gradient measurements, G_i and G_e , respectively and associated line of best fit by linear regression.

Figure 10 shows the results of the growth rate and position measurements for two tests using the tracker and manual measurements. The corresponding experimental conditions; cooling rates, thermal gradients and average isotherm speed, are presented in table 3. The first row in figure 10 shows results for fully columnar growth while the second row show results for a case of fully equiaxed growth. In distinguishing the different growth morphologies, an initially equiaxed crystal is deemed columnar if the length of a primary arm has grown to 4 times that of the other arms. The results show good agreement between the automatic tracking position/speed and the manual reference measurements.

Microscopic videography allowed for the observation of growth morphology and, combined with automatic tracking, temporal measurements of the vertical growth rate. In order to assess the efficacy of different growth rate measurement techniques, the new automatic tracking technique developed here and the traditional isotherm speed measurement technique have been compared with manually measured position/growth rate. The advantages and disadvantages are discussed.

Isotherm speed measurement is useful particularly for ex-situ experiments where liquid melt and the mould walls are opaque. In taking the v_I measurement, the following is assumed: (i) temperature profile between the thermocouples is linear (ii) cooling rate is uniform in the baffle zone (iii) growth rate is steady. As table 3 shows, the isotherm method does not provide accurate temporal measurements and averaged results of the method were between 15.3 to 18.9% different from the manual reference measurement of growth rate, v_M . In contrast, the averaged tracker speed measurements deviated between 0.6 to 2.7 % from the reference. Importantly, temporal speed measurements with the automated tracker compared well for cases of fully columnar and fully equiaxed growth (see figure 10 (b, e)), showing robustness of the image processing technique for different morphologies. A key advantage of automatically tracking the growth rate of the solidification front with this technique is it being repeatable and easily

Table 3. Experimental results of growth rate measurements (manual reference, v_M , tracker, v_T , and isotherm speed v_I), % difference with respect to reference manual measurement, measured processing conditions and observed macrostructures, denoted S being either, (C) fully columnar or (E) fully equiaxed.

Test	v_M ($\mu\text{m/s}$)	v_T ($\mu\text{m/s}$)	%diff	v_I ($\mu\text{m/s}$)	%diff	$\dot{T}$ (K/min)	G (K/cm)	S
I	1.16 ± 0.12	1.14 ± 0.15	0.80%	0.96 ± 0.11	16.70%	0.14 ± 0.01	23.46 ± 1.75	C
II	2.31 ± 0.26	2.26 ± 0.27	1.80%	1.96 ± 0.24	15.30%	0.28 ± 0.01	23.53 ± 1.80	C
III	3.47 ± 0.32	3.45 ± 0.26	0.60%	2.88 ± 0.33	16.90%	0.42 ± 0.01	23.57 ± 1.71	C
IV	6.03 ± 1.39	5.88 ± 0.84	2.70%	4.91 ± 0.62	18.70%	0.71 ± 0.01	23.34 ± 1.84	E
V	5.78 ± 1.83	5.75 ± 1.43	0.60%	4.68 ± 0.54	18.90%	0.69 ± 0.02	23.60 ± 1.69	E

Figure 10. Results of tracker and manual measurements for test number I (a - c) and IV (d - f). (a, d) graphs of position measurements, z_T and z_M against time (b, e) graphs of growth rate measurements, v_T and v_M , against time, (c, f) close up images of the solidification front with manual trace and tracker position overlaid

implemented over all frames, whereas manual position and hence growth rate measurements for each of the thousands of frames captured during experiments would be impractical, not to mention susceptible to human error.

6. Conclusions

An experimental facility and methodology have been developed for investigating in-situ directional solidification of transparent alloys of neopentyl-glycol and (d)camphor. The apparatus, consisting of a transparent sapphire crucible, facilitates optical visualisation of the solidification process in transparent alloys with controlled cooling rate, temperature gradient and isotherm speed, is described in detail and quantifiable experimental uncertainties are provided.

A new image processing technique for in-situ tracking of the position and speed of the solidification front was developed in MATLAB. Growth rates have been measured with the new automatic tracking technique and the traditional isotherm speed technique, and have been compared against manual measurements for five directional solidification experiments. In all cases, the tracker speed was significantly more reliable than isotherm speed and showed good temporal agreement with manual measurements for cases of fully columnar and fully equiaxed growth. Historically, the isotherm speed measurement technique is accepted as a reasonable approximation for growth rates and is necessarily used for ex-situ experiments, where visual access to the solidification front is not possible. This work, which is capable of assessing the accuracy of the isotherm technique, shows that it can be in error up to 19%, which is significant. In

contrast, the new measurement technology developed here compares better than 3% error from manual measurements.

Two growth morphologies were observed for transparent model alloy NPG-35wt%-DC, namely fully columnar, and fully equiaxed. The auto tracked growth rate not only compared well for cases of fully columnar and fully equiaxed growth morphologies but also showed the robustness of the tracker for variation in growth patterns.

The model alloy used in this manuscript has been studied in microgravity conditions on sounding rockets missions [16], and in hypergravity conditions [25] and future work is planned onboard the International Space Station (ISS) with the NPG DC system where buoyancy-driven flow and sedimentation of equiaxed crystals is precluded. Experiments conducted in this manuscript and future work with this experimental facility will serve to provide complementary terrestrial data for future investigations on the ISS. Additionally, by inverting the experimental apparatus, the effect of solidification in the direction of and in opposition to the gravity vector has been investigated [26].

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