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Rapid solidification: in situ diagnostics and theoretical modelling

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Abstract

We report on progress in direct experimental investigations of non-equilibrium crystallization in undercooled melts. Containerless processing is applied for undercooling and is combined with diagnostic means. The results of such experiments are utilized to critically assess physical models for rapid solidification. Existing models of rapid growth of solid into undercooled melts are reviewed. The sharp interface model is applied to quantitatively describe rapid dendrite growth of metals and alloys. It is extended to include effects of fluid flow in liquids induced by forced convection in electromagnetically levitated drops. Morphological transitions resulting in grain refinement via instabilities of growing dendrites are analyzed and used to evaluate the effect of fluid flow on grain refinement. Theoretical predictions given by sharp-interface model are compared with results of phase-field modeling and with new data of crystal growth dynamics measured with high accuracy. Depending on the undercooling prior to solidification the effects of solute diffusion, convective flow, solute trapping and microstructure evolution are demonstrated.
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Keywords: Containerless processing; Undercooling; Non-equilibrium solidification; Dendrite growth; Grain refinement; Convection

1. Introduction

By far most of materials of daily human life are solidified from the liquid state. Solidification is therefore, one of the most important phase transformations in industrial production routes. It plays an important role in casting and foundry industry. In these cases, solidification takes place under near equilibrium conditions. Solidification under near equilibrium means that there is a solidification pathway from the liquid to the stable solid state and the system is cooled slowly with rates ranging between 10^{-3} and 10^3 K/s.

It is well known that the spectrum of materials with different properties is greatly enhanced by rapid quenching the liquid. Techniques of rapid quenching realize cooling rates up to 10^7 or even 10^8 K/s. Melt spinning, splat cooling, laser or electron beam surface re-solidification, atomization, spray deposition have demonstrated very successfully that metastable solids can be produced by such techniques. Supersaturated solutions, disordered superlattice structures of intermetallics, grain refined alloys and even amorphous metallic alloys have been rapidly solidified. From a thermodynamic view, undercooling is mandatory to form a metastable solid from liquid state. The major rapid crystal growth mode in undercooled melt is dendrite growth that controls the microstructure evolution [1]. Present attempts are directed towards quantitative observation of dynamics and morphology of dendrite growth in order to develop experimentally verified theoretical models. In the present work, modern concepts of experimental diagnostics during rapid solidification on containerlessly undercooled drops are presented. The experimental results will serve to verify theoretical concepts to describe rapid dendrite growth taking into account heat and mass transport by forced convection in electromagnetically processed melts. Their impact on microstructure evolution will be demonstrated.

2. Experimental

Electromagnetic levitation technique is used to undercool samples in diameter of 5–8 mm in containerless state. Fig. 1 shows the experimental set up. The temperature of the levitated melt is measured contactless by a pyrometer with an accuracy of ±5 K. To measure the dendrite growth velocity with high reliability and reproducibility, a capacitance proximity sensor (CPS) is used [2]. The set-up consists of a nucleation trigger needle made of the same material as the sample. The needle is part of a resistance–capacitance (RC) electrical circuit whose resonance frequency is measured. If the needle is touching the sample

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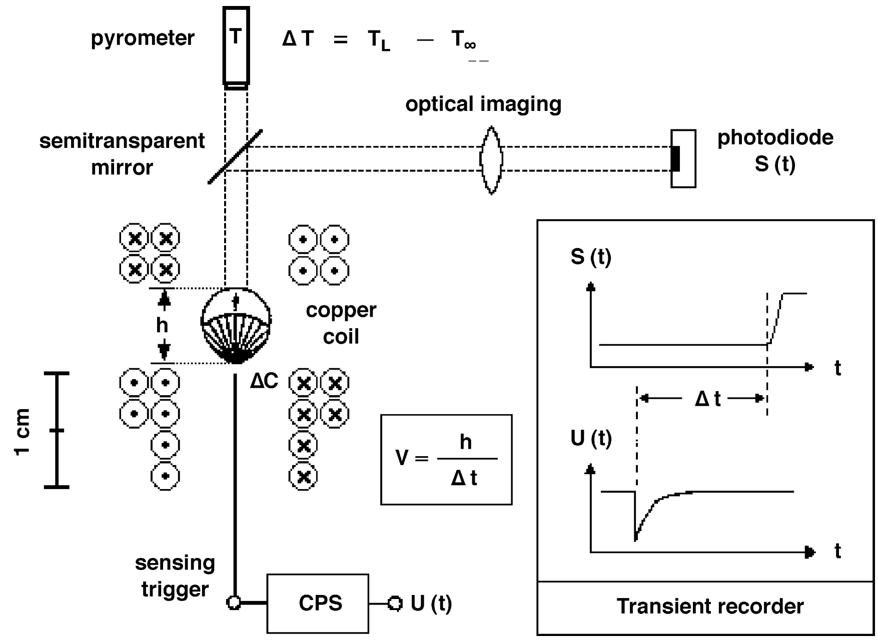

Fig. 1. Experimental technique to measure the velocity of the solidification front in levitation undercooled samples by using the capacitance proximity sensor (CPS) technique [2].

and initiates solidification, the capacitance of the RC—circuit changes abruptly. The time, t_1, of initiating crystallization is measured by a sudden change of the output signal of the RC circuit with a time resolution of 1 μs. The counterpart of the triggering point at the opposite side of the sample is focused by an optical system on the sensitive area of a photodiode. As soon as the central dendrite is arriving at the top of the sample at time, t_2, the signal of the photodiode rapidly increases. The growth velocity, V, is then obtained by dividing the height, D_o, of the as-solidified sample by the time difference $\Delta t = t_2 - t_1$: $V = D_o/\Delta t$. In addition to CPS, we also apply a high-speed digital camera (frame rates up to 120,000 frames per second (fps) at a resolution of 128 × 16 pixel) to observe the propagation of the solidification front. Fig. 2 gives a sequence of images taken during solidification of a Ni sample undercooled by $\Delta T = 90$ K (upper part) and undercooled by $\Delta T = 140$ K (lower part), respectively [3]. It can be seen that the solidification front at the sample surface is of an irregular shape for $\Delta T = 90$ K, while at $\Delta T = 140$ K the front appears very smooth. The high-speed camera allows for investigations of both the macroscopic morphology and the velocity of the advancing solid–liquid interface.

3. Results and discussion

3.1. Pure nickel

Fig. 3 shows the dendrite growth velocity as a function of undercooling measured on nominally pure Ni (4N) in the undercooling range 35 K < ΔT < 305 K. A monotonous increase of V is observed with undercooling. Compared with previous measurements the CPS sensor technique leads to an essential improvement of the accuracy [3]. In order to analyze the results sharp interface theory is applied. Since we are dealing with solidification of electromagnetically levitated drops forced convection induced by the strong alternating electromagnetic fields has to be taken into account. We extend the LGT/LKT model [4,5] by including changes of heat transport by convection in the solution of the heat transport equation [6]. Accordingly, the thermal

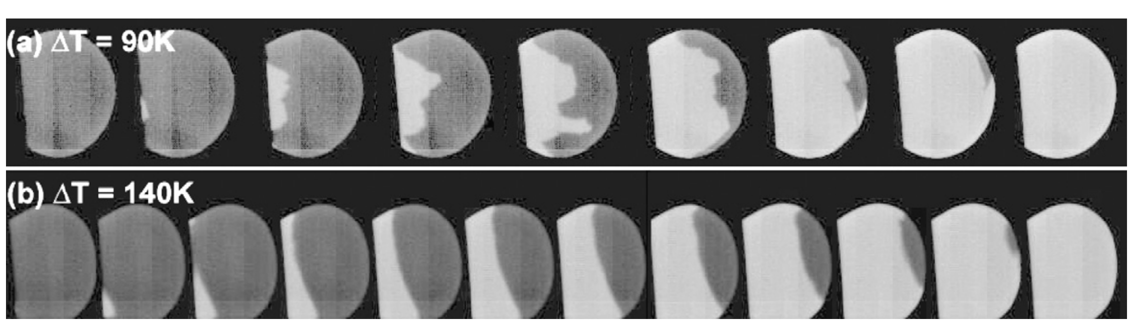

Fig. 2. Propagation of the solidification front (light gray) through an undercooled Ni melt (dark gray) as observed by a high speed camera for undercoolings of $\Delta T = 90$ K (a) and 140 K (b). Both sequences were recorded at a frame rate of 30,000 fps, for 90 K only each fifth image is shown. Triggering occurred at the bottom of the sample, which is to the left in this display.

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