In-Situ Observation of Rapid Solidification during "Strip Casting" of Transparent Alloys

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ABSTRACT

In this article a technique is presented which extrapolates the solidification conditions governing the strip casting of metals to the rapid solidification of transparent alloys. Fine capillary tubes filled with different transparent alloys were pulled rapidly from a hot into a cold zone. Solidification within the tube was observed with an optical microscope. Under the conditions applied a flat dendritic microstructure covered the tube from inside, growing in a highly unstable manner with the pulling velocity. On this solid layer solidification proceeded with different morphologies depending on the conditions present and the alloy concentration in use. Five different morphologies are presented.

KEYWORDS

rapid solidification, cellular growth, surface dendrites, melt spinning

1. INTRODUCTION

Materials with metastable microstructures and superior properties produced by rapid solidification have been studied in the last few years [1-3] and continue to be the subject of intensive research [4, 5]. Strip casting of metals, in particular, gained more and more attention because of its ability to produce thin foils or sheets directly from the melt. Among a great number of different techniques chill block melt spinning, planar flow casting, melt extraction and double roller casting are most often used [6-8]. The solidification conditions governing these processes are determined by the contact of a flat liquid layer with a cooled substrate. Hence the heat is extracted very rapidly producing, together with the high substrate velocity, a directional solidification almost perpendicular to the substrate [9].

In this work a similar configuration had been used for the rapid solidification of transparent organic materials. These materials are known to solidify like metals. Thus the evolution of interface morphologies can be observed in-situ under solidification conditions comparable to strip casting processes. Figure 1 shows the ribbon formation during planar flow casting and the analogue situation which were used to solidify the organic material.

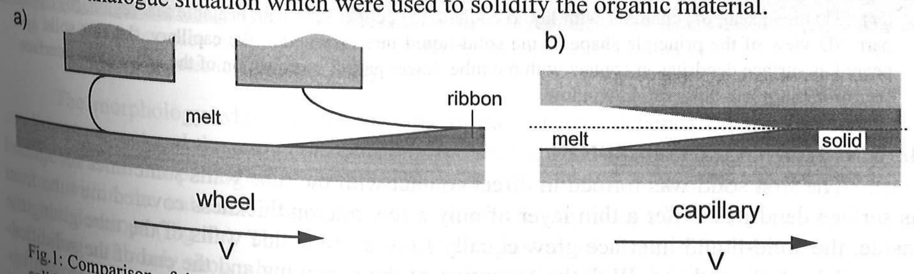

Fig.1: Comparison of the ribbon formation in planar flow casting (a) and the analogue situation for rapid solidification of transparent alloys in capillary tubes (b).

2. EXPERIMENTAL PROCEDURE

In situ observations of the interface morphologies were performed by rapidly pulling long, fine capillary tubes from a furnace into a liquid coolant. The capillaries used in this work had an inner cross section of 200 x 200 µm^2 with a wall thickness of 100 µm. They were 900 mm long and made of borosilicate glass.

The apparatus used in the experiments consisted of a long furnace, a cooling chamber through which the liquid coolant was pumped, and a pulling device, which permitted pulling velocities between 1 and 30 mm/s. For a solidification experiment the capillary tube was first threaded through the seals of the cooling system and then aligned parallel/perpendicular to the optical axis of the microscope. While pulling the tube with a constant withdrawal velocity from the furnace into the coolant, solidification inside the tube was studied with the microscope and the observations monitored by a VCR.

A keypoint in the experimental setup is the heatable sealing between the furnace and the liquid coolant. This sealing allows the tube to be kept at a constant temperature until it reaches the coolant. Thus the solid-liquid interface can be directly observed from the beginning of the solidification process at the substrate/melt interface to the end in the middle of the tube. A schematic drawing of the heatable sealing and resulting pyramidal solidification front including the typical observation window is shown in figure 2.

Due to this pyramidal interface the solidification front was observed perpendicular to the solid-liquid interface, and simultaneously, laterally. The actual growth rate could thus be obtained from the angle α between the interface normal and the pulling direction ($V = V_0$ cosα, with V_0: pulling velocity). Local solidification velocities V (normal to the solid-liquid interface) between 0.1 mm/s and almost 2 mm/s were achieved.

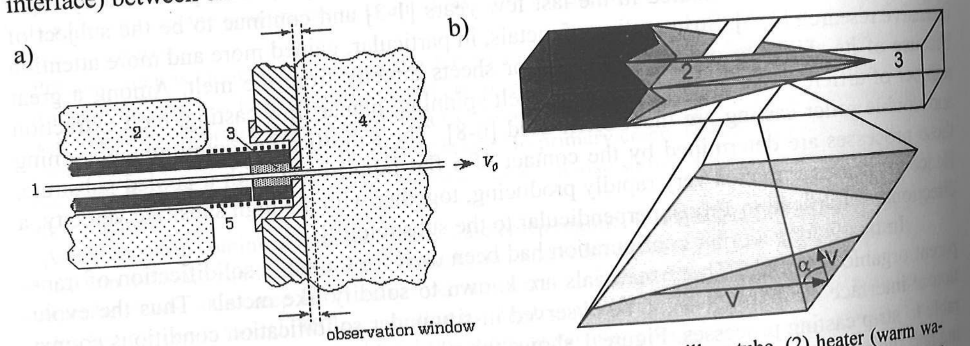

Fig.2: Details of the experimental arrangement (schematic): (a) (1) capillary tube, (2) heater (warm water), (3) silicon seal, (4) chamber with liquid coolant, (5) copper tube with heatable seal region, (b) Upper part: 3D-view of the principle shape of the solid-liquid interface within the capillary: the first solid appeared as surface dendrites in contact with the tube. lower part: 2D-projection of the solid-liquid interface seen from a typical observation window.

3. RESULTS AND DISCUSSION

The first solid was formed in direct contact with the tube walls sometimes recognised as surface dendrites. After a thin layer of only a few micron thickness covered the tube from inside, the solid-liquid interface grew equally from all four side walls of the tube giving the pyramidal interface shape. With the exception of the beginning and the end of the solidification region, the solid-liquid interface was macroscopically flat and therefore the growth velocity was constant over most of the half-width of the capillary. The diffusion boundary layers

from opposite side planes did not overlap except in the region where almost the whole liquid was solidified [10].

Figure 3 shows a solidification front, observed in a hypoeutectic CBr_4-C_2Cl_6 alloy with a solidification velocity of $V = 0.2$ mm/s. The dendritic front in contact with the tube wall grew in a highly unstable manner with the pulling velocity. Another example of the dynamic growth of these surface dendrites is shown in Figure 4a. Permanent competition between different growth directions including overgrowth and even curved surface dendrites resulted in a irregular structured solid layer, on which solidification proceeds.

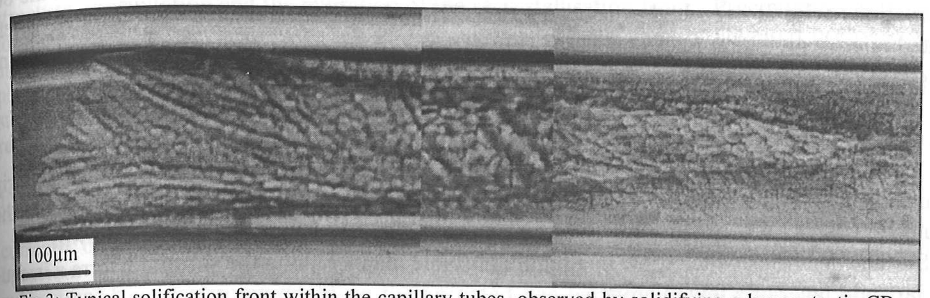

Fig.3: Typical solification front within the capillary tubes, observed by solidifying a hypoeutectic CBr_4-C_2Cl_6 alloy. To give an overall impression of the solidifying region, three photos from different locations were mounted together.

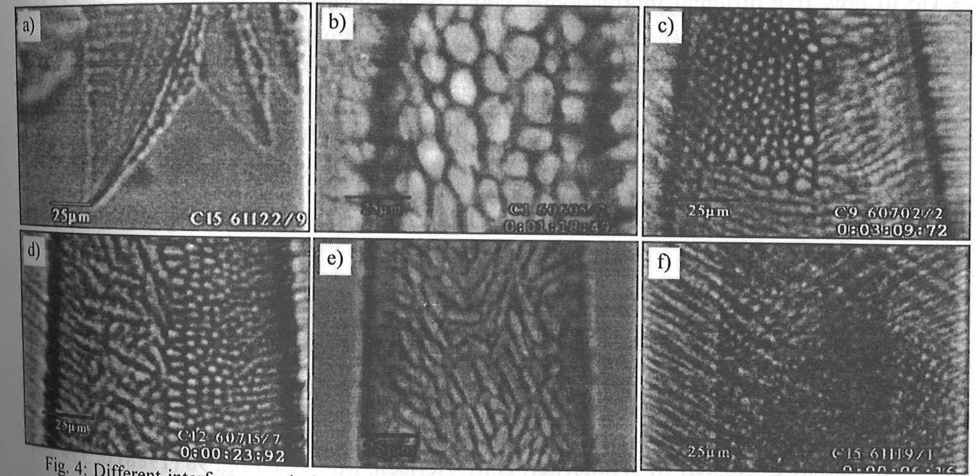

Fig. 4: Different interface morphologies observed during the rapid solidification of alloys with different concentrations in the eutectic CBr_4-C_2Cl_6 alloy system.

The morphology which established by further growth depends on the solidification conditions present and the alloy concentration in use. Figures 4b-f present an overview of solidification front morphologies observed under various conditions for different alloy concentrations within the eutectic CBr_4-C_2Cl_6 system. In slightly alloyed CBr_4 a coarse cellular structure develops (Fig 4b). This morphology grows just above the limit of constitutional undercooling as already shown by one of the authors in ref. [10]. With increasing concentration cylindrical and elongated cells separated by a grain boundary were observed (Fig. 4c). This boundary often revealed a small inclination of some degree with respect to pulling direction or even showed a sharp turn from normal to perpendicular to the pulling direction [11]. For hypoeutectic alloys close to the eutectic concentration a transition from cellular morphologies t elongated structures with small shape instabilities was observed (Fig 4d). Theoretical consi erations suggest that this struture could be quasi-dendritic. In hypereutectic alloys near th eutectic composition a morphology with cone-shaped structures prevailed (Fig. 4e). Note th this morphology does not show microsegregation tracks on the side view. Because the solid fication velocity is too low to cause any solute trapping effects, the origin of this observatio must be elsewhere. It is conceivable that a eutectic with spacings too small to be resolved by microscope grew with a irregular interface. Similar differences in the interpretation of the o servations arise at even higher concentrations. Here irregular structures with different orie tated microsegregation tracks appeared (Fig 4f). The observed mophologies and the appearin morphological transitions are the subject of further investigations.

4. CONCLUSIONS

A configuration for rapid solidification of transparent organic alloys similar to stri casting processes is presented. The morphology of alloys with different compositions in th eutectic CBr_4-C_2Cl_6 system was investigated. The following conclusions can be drawn:

- Surface dendrites which grew in a highly unstable manner along the substrate, form a irregu lar solid layer of a few microns.
- Solidification proceeds on this irregular solid layer. The resulting morphologies depend o the solidification conditions present and the alloy composition in use.
- Up to now five different morphologies could be distinguished: (i) a cellular morphology with cylindrical cells; (ii) a cellular morphology with elongated cells; (iii) a morphology with elongated structures with small shape instabilities, probably of dendritic nature; (iv) a morphology with cone-shaped structures; (v) a morphology of irregular structure.
- The morphology (i) and (ii) as well as (i) and (iii) can coexist, separated by boundaries.

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