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Solidification of an alloy in a cavity cooled through its top surface

W.-Z. CAO and D. POULIKAKOS
Mechanical Engineering Department, University of Illinois at Chicago,
P.O. Box 4348, Chicago, IL 60680, U.S.A.

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Abstract—An experimental study on the phenomenon of solidification of a binary alloy (water and ammonium chloride) in a cavity cooled through its top wall is reported. Three distinct regions exist in the cavity during the solidification process. A region occupied by the solid, a region occupied by the liquid and a mixed phase region (mushy zone) consisting of a complex combination of dendrites and liquid. The mixed phase region is of considerable extent, often larger than that of the solid. The growth of the solid/mush interface and the mush/liquid interface with time as well as the temperature distribution in the cavity are determined in the course of the study for a host of initial concentrations. The temperature field in the mixed phase and the solid regions is conduction dominated. This, despite the fact that the mushy region is permeable and allows for flow within it. Apparently, in the experiments of this study the flow in the mush is not sufficiently strong to render convection the main heat transfer mode in the mush. In the liquid phase a bicellular flow field exists. The flow has an effect on the shape of the mush/liquid interface. This effect, however, is weak and the interface remains predominantly flat especially for higher concentrations. Double diffusive convection takes place in the liquid region because ammonium chloride is constantly released into the liquid as the solidification progresses downwards, after it started in the vicinity of the top wall. Salt fingers are clearly visible penetrating into the liquid region. The thickness of the mushy zone generally increases with time. Increasing the initial concentration of the alloy reduces the thickness of the mixed phase region.

1. INTRODUCTION

SOLIDIFICATION of alloys has numerous engineering applications some of which are important to the manufacturing of products widely used in everyday life. To exemplify, casting of metals or other composites, coating processes, crystallization processes and freezing of mixtures in the food industry are all directly related to solidification of alloys.

In addition to its unquestioned significance and engineering relevance, alloy solidification constitutes a challenging research area because of the numerous intricacies involved in this process. From the heat and mass transport standpoint, the interest in solidification of alloys is threefold.

(a) In the liquid phase, convection affected by the simultaneous action of temperature and concentration gradients often occurs.

(b) In the solid phase combined heat and mass diffusion arises.

(c) Most importantly, the liquid region and the solid region are not separated by a sharp interface. Instead, a mixed phase region (often termed 'the mushy zone') consisting of a complex mixture of solid freckles and liquid exists between the solid and the liquid regions. In the mushy zone, heat and mass transfer occur in both phases and liquid motion is possible because of the permeable nature of the solid matrix composed by the freckles. The porosity of the mushy zone varies both spatially and temporally and it is not known a priori.

Several investigations have been reported in the literature pertaining to the freezing of alloys. For example, O'Callaghan *et al.* [1] used a model which accounts for the mushy zone to study theoretically heat and mass transfer during solidification of a eutectic binary solution. The speed of propagation of the liquid/mush interface was taken to be identical to the speed of propagation of the solid/liquid interface. Fang *et al.* [2] reported an experimental and theoretical study of the selective freezing of a dilute solution on a cold ice surface. The comparison between theory and experiment showed good agreement. Worster [3] investigated theoretically the effect of the mushy zone on the solidification of an alloy cooled from below. His model accounted for the simultaneous action of heat and mass diffusion. Theoretical work on the heat and mass transfer processes during the growth of a solid from a thin pipe or wire immersed on a binary alloy is given in refs. [4, 5]. The mixed phase region proved to be of paramount importance on the resulting solid growth rate. Bennon and Incropera [6] derived a set of continuum conservation equations for binary solid–liquid phase change systems by integrating semi-empirical laws and microscopic descriptions of transport behavior with principles of classical mixture theory. The same authors [7] used these con-

NOMENCLATURE

C	concentration, weight fraction
H	height of the cavity [mm]
Q	heat transfer rate through the bottom wall [W]
s	distance from the top wall [mm]
t	time [s]
T	temperature [°C]
y	distance from the bottom wall [mm].

servation equations to model and solve numerically the problem of solidification of a binary alloy in a cavity cooled through one of its side walls. Additional work pertinent to the study of the mushy zone that has been reported in the literature is exemplified by refs. [8–10].

In the present paper, the freezing of a mixture of water and NH_4Cl is considered. The mixture occupies a rectangular cavity which is cooled through its top boundary. The water and NH_4Cl alloy is eutectic and follows trends observed in solidification of metals. In this respect, the results of this paper will elucidate some aspects related to the importance of the mixed phase region in casting processes. In addition, the work reported in this paper is directly related to freezing of mixtures in the chemical and food industries.

2. APPARATUS AND PROCEDURE

The setup for the experiment consisted of the test apparatus and three supporting devices. The three supporting devices were a data acquisition system, a power supply, and a bath refrigerator circulator. The data acquisition system consisted of a Hewlett–Packard 150 Touchscreen II PC, two Hewlett–Packard 3421A data acquisition/control units, a printer, and a Hewlett–Packard data acquisition software package. The power supply for the heater element was a Hewlett–Packard 6012B 1000 W d.c. power supply. A Brinkman RC20 refrigerator unit was used to cool the top plate of the apparatus.

The test apparatus was a rectangular cavity. The internal dimensions of the test apparatus measured 48.3 cm long by 25.4 cm tall by 12.7 cm deep. Plexiglas (low thermal conductivity) 1.27 cm thick was used to construct the side walls of the apparatus so that these walls did not alter the vertical heat transport. The top and the bottom walls were constructed out of aluminum of thickness 2.54 and 1.27 cm, respectively. The bottom plate had 10 (T-type) thermocouples embedded 1.6 mm from the inside surface. Machined into the bottom plate was a cavity for a 3.2 mm thick, high-density (10 W in.$^{-2}$) flexible rubber electric heater. Highly conductive silicon paste was used to ensure good conductivity between the heater and the aluminum plate. The heater was insulated from below with two layers of 8 mm thick asbestos gasket material and a layer of 16 mm thick rubber insulation. The presence of the heater allowed for heating from below (whenever desirable) simultaneously with cooling from above. This heating effect enabled us to enhance the buoyancy driven flow in the liquid phase and to contrast its effect to experiments in which no heating from below existed.

The top plate was machined to allow for eight thermocouples and a counterflow heat exchanger. The counterflow heat exchanger was constructed by milling four channels into the top plate lengthwise. A 50% ethylene glycol/water solution precooled by the bath refrigerator was circulated through the heat exchanger. The direction of flow of the coolant was alternated in adjacent channels to establish isothermal conditions (within 0.5°C) along the top plate. The channels were sealed with a 1.27 cm thick sheet of Plexiglas which covered the entire top plate.

Placed within the cavity were two vertical plastic rods on each of which 25 thermocouples were mounted. The so constructed two thermocouple columns allowed for the determination of transient temperature distributions in the vertical direction. One thermocouple column was placed at the centerline of the cavity. The other in the middle of the left half of the cavity.

3. RESULTS AND DISCUSSION

The main results of this study document the effects of the initial concentration of the alloy and of the bottom wall heating on the solidification process (the growth of the solid, the liquid and the mixed phase regions). In Fig. 1 the phase equilibrium diagram of a water–ammonium chloride mixture is shown [11]. According to this diagram, when a liquid mixture of

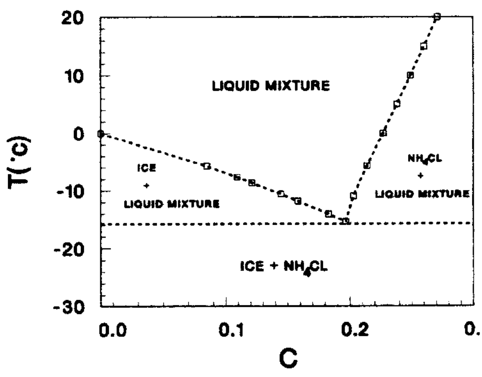


FIG. 1. Equilibrium phase diagram for a water–NH_4Cl eutectic binary alloy.

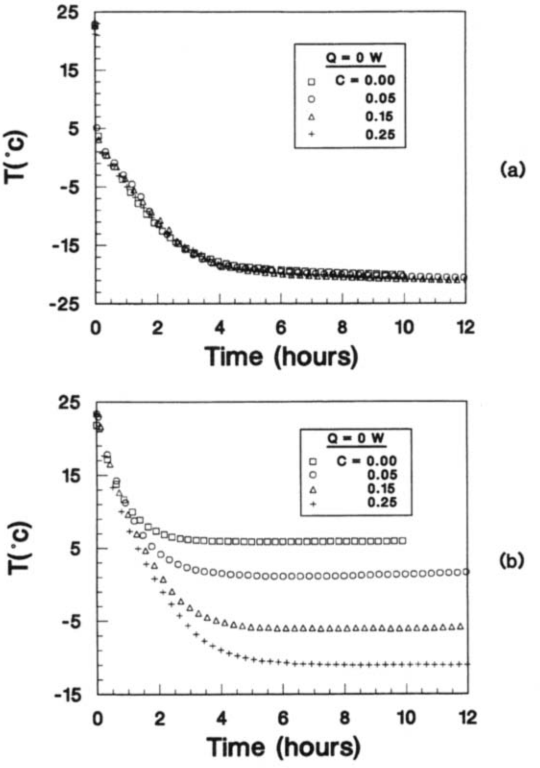


FIG. 2. (a) Top wall temperature variation with time for $Q = 0$ W. (b) Bottom wall temperature variation with time for $Q = 0$ W.

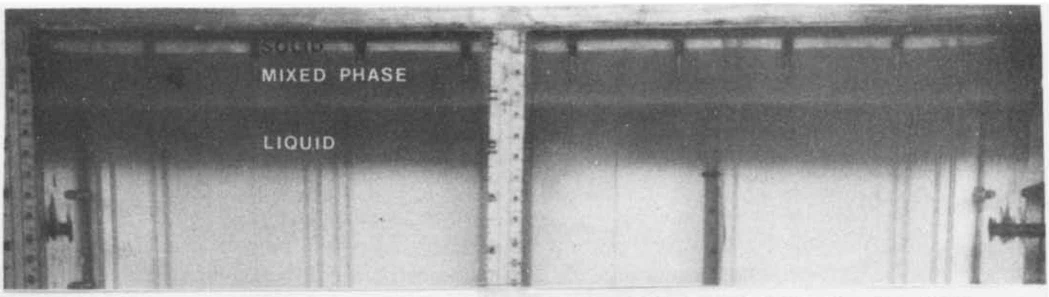
concentration less than about $C = 0.19$ is cooled at static conditions below the temperature of the liquidus curve, ice grows from the liquid mixture creating a mixed phase region (ice + liquid mixture). The last liquid to solidify forms a eutectic solid mixture of ice and ammonium chloride. On the other hand, if the temperature of a mixture of concentration greater than approximately $C = 0.19$ is lowered below the liquidus temperature, ammonium chloride grows from the liquid.

In the first series of experiments the top wall of the cavity was cooled with the help of the heat exchanger described earlier. Before it was circulated through the top wall heat exchanger, the coolant temperature was lowered down to −25°C in the refrigerator circulator. The temperature of the coolant in the refrigerator circulator remained practically unchanged for the duration of the experiments. Figure 2 shows the dependence of the top (Fig. 2(a)) and bottom (Fig. 2(b)) wall temperature on time for different initial alloy concentrations in the cavity. In all cases, the temperatures of both walls decrease rapidly until a plateau is reached. The initial concentration effect on the top wall temperature is practically negligible. However, the temperature of the bottom wall decreases noticeably as the concentration increases. This result indicates that the liquid mixture needs to be brought down to considerably lower temperatures for the solidification to take place as the concentration increases.

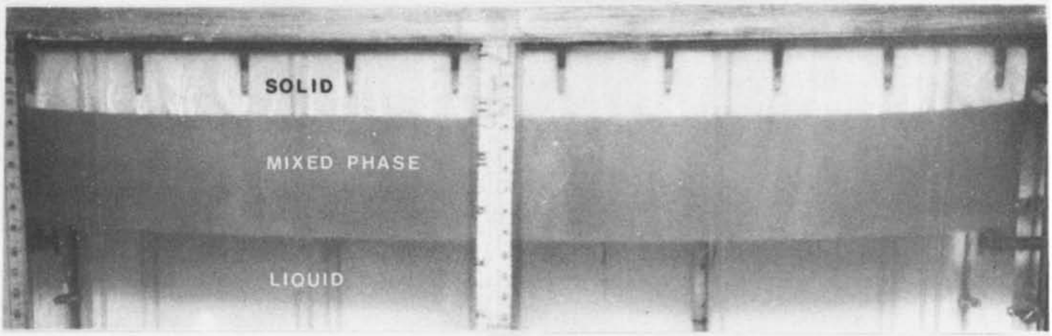
Figure 3 offers photographic evidence for the existence and the extent of the different regions in the cavity. The first two photographs in the sequence (Figs. 3(a) and (b)) correspond to the same initial concentration ($C = 0.05$). At early times ($t = 4.5$ h) a thin layer of solid ice (+NH_4Cl) is attached to the top wall. A sizable mushy zone separates this solid from the liquid region. The mushy zone consists of ice and liquid mixture. Both the solid/mush and the mush/liquid interfaces are rather smooth indicating that convection within the mushy zone is not dominant. Indeed, the interweaving ice dendrites in the mixed phase region create a solid matrix permeable to liquid. According to published results on solidification of ammonium chloride mixtures cooled from below with the help of a chill, liquid jets in the mush were observed moving upward [12]. These jets are known to leave a distinct trail (hole) in the mush and, as they break through the mush/liquid interface, they create sites of advanced dendrite growth. The randomness of these sites is responsible for a coarse mush/liquid interface, infested by tips of dendrites growing at different rates [11]. In our experiments, the cooling took place through the top wall in the manner defined in Fig. 2(a). No liquid jets were observed and the mushy zone appeared to be well structured and homogeneous. As the time progressed (Fig. 3(b)) both the solid region and the mush grew significantly. The thickness of the mushy zone appears to be about twice the thickness of the solid region. This is an indication of the importance of the mushy zone on the solidification process.

Observations indicated that buoyancy driven flow in the liquid region was bicellular in all the experiments reported in this study. The flatness of this interface, however, implies that buoyancy driven flow is weak.

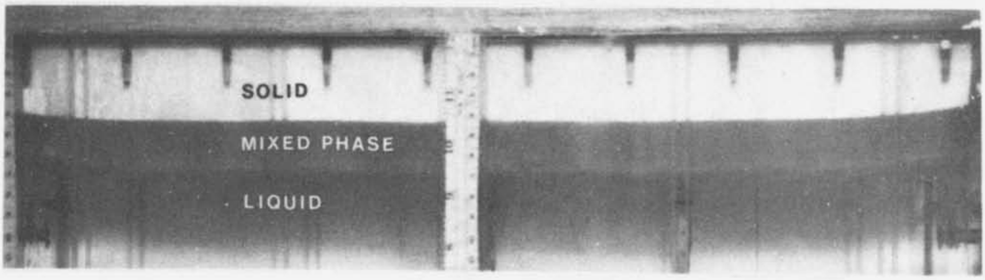
Increasing the initial concentration of the mixture to $C = 0.15$ significantly decreases the thickness of the mushy zone as a comparison of Figs. 3(b) and (c) corresponding to the same time indicates. A further increase of the initial NH_4Cl concentration to $C = 0.25$ (Fig. 3(d)) has a remarkable effect: no mushy zone exists at $t = 15$ h. The solid and liquid regions are separated by a sharp interface. Note that the dark region in the photograph at the solid/liquid interface is not a mushy zone. It is simply a shade created by the bottom surface of the solid/liquid interface. According to the phase diagram of Fig. 1, a mushy zone should exist for $C = 0.25$ at *static* conditions, i.e. in the absence of fluid flow. Indeed, as will be discussed next in connection with Fig. 4(a) a mushy zone appeared at early times. However, at later times it disappeared. The physical reasoning for this fact is that the presence of the flow in the liquid region inhibits the creation of a permeable solid matrix consisting of crystallized NH_4Cl. Ammonium chloride precipitates through this interface into the liquid in the form of 'salt fingers' penetrating downward into the liquid (Fig. 3(d)). The buoyancy-driven convection in the liquid region is of the double diffusion



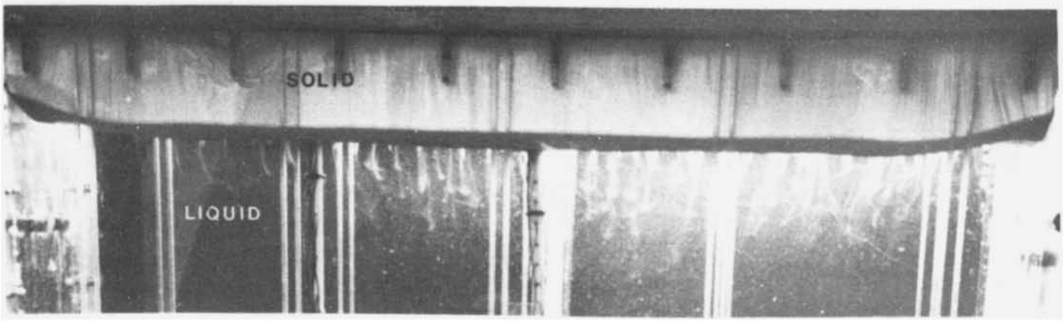
(a) C = 0.05, Q = 0.0 Watts, t = 4.5 hours



(b) C = 0.05, Q = 0.0 Watts, t = 15 hours



(c) C = 0.15, Q = 0.0 Watts, t = 15 hours



(d) C = 0.25, Q = 0.0 Watts, t = 15 hours

FIG. 3. Sequence of photographs documenting the solidification process for $Q = 0$ W: (a) $C = 0.05$, $t = 4.5$ h; (b) $C = 0.05$, $t = 15$ h; (c) $C = 0.15$, $t = 15$ h; (d) $C = 0.25$, $t = 15$ h.

type with unstable concentration gradients (high concentrations at the top of the liquid layer).

Figure 4(a) shows the growth of the mixed phase region at the centerline of the cavity for characteristic initial concentrations of the alloy. The dark symbols refer to the solid/mush interface and the open symbols to the mush/liquid interface. Clearly, as time progresses the thickness of the mushy zone increases in all cases. No mush exists in the case of solidification of pure water ($C = 0$). Increasing the initial concentration of the alloy from $C = 0.05$ to 0.15 decreases the distance of the mush/liquid interface from the cold boundary at any given time and, more importantly, it increases the thickness of the solid. A further increase

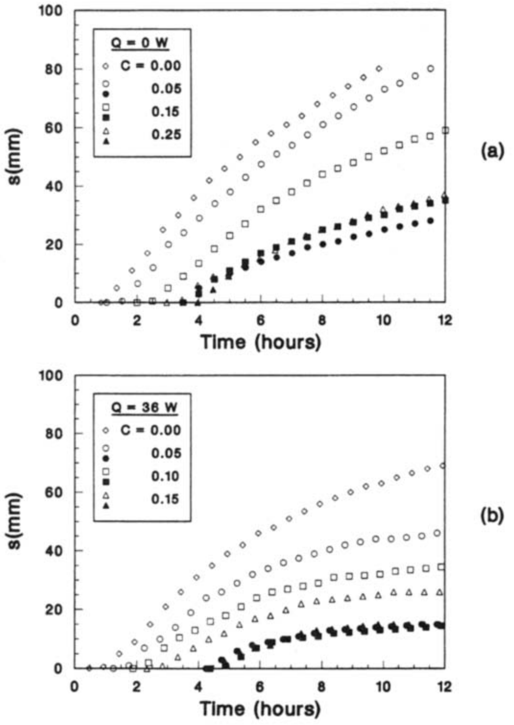


FIG. 4. The growth of the solid/mush (dark symbols) and mush/liquid (open symbols) interfaces at the centerline of the cavity: (a) $Q = 0$ W; (b) $Q = 36$ W.

of the initial concentration to $C = 0.25$ still results in the early appearance of a mushy zone. However, after $t = 5.5$ h the mushy zone disappears and the solid and the liquid are separated by a sharp interface.

In all cases, it is observed that a mushy region appears first in the close vicinity of the cooled top boundary (Fig. 4(a)). The solid region appears after the mixed phase region is well into existence. Increasing the initial concentration delays the solidification phenomenon and the appearance of both the mixed phase region and the solid region.

An example of the temperature distribution in the cavity is shown in Fig. 5(a) with the help of centerline temperature profiles at $t = 12$ h ($t = 10$ h for $C = 0$). In each case, the position of the mush/liquid interface is indicated by a small horizontal line. Clearly, at $t = 12$ h the temperature distribution in the solid and the mixed phase region for all concentrations investigated is linear. This is an important fact for it indicates that the main heat transfer mechanism in the mushy zone is conduction. Even though the mushy zone is permeable, the flow within it is not strong enough to seriously affect the temperature field. Figure 5(a) shows that the temperature distribution in the liquid phase is practically uniform. Increasing the initial concentration decreases the temperature throughout the cavity.

To explore further the effect of the buoyancy-driven flow on the solidification phenomenon, we conducted a series of experiments in which the bottom wall was heated with a constant heat flux with the help of the heater discussed in the previous section. More specially, at $t = 0$ the coolant was circulated through the top wall heat exchanger and simultaneously a constant heating rate $Q = 36$ W equally distributed was applied to the bottom wall. This heating condition induced larger temperature gradients in the cavity and enhanced the buoyancy-driven flow.

Figure 6 shows the temperature history of the top and bottom walls at characteristic initial concentrations. These temperature distributions exhibit the same features with Fig. 2 for $Q = 0$ W. The most noticeable difference is that the bottom wall temperature reaches a plateau at considerably higher values (Figs. 2(b) and 6(b)). For example, at $t = 6$ h and for $C = 0.15$ the bottom wall temperature in Fig. 2(b) ($Q = 0$ W) is $T = -6$°C. For the same case, Fig. 6(b) ($Q = 36$ W) yields $T = 0$°C.

The photographic evidence presented in Fig. 7 proves that the effect of heating the bottom wall is indeed significant. In the limit of solidification of pure water (Fig. 7(a)) the solid and the liquid are separated by a sharp interface. This interface is 'carved' by the buoyancy-driven flow in the liquid which consists of two counterrotating cells. Figures 7(b) and (c) indicate that the solidification process starts ($t = 3.75$ h) with the appearance of a mushy region in the vicinity of the cold boundary. The solid region appears later (Fig. 7(c)). The mush/liquid interface is again rather flat (not carved) indicating that the presence of the species concentration acts as to weaken the buoyancy-driven flow. Increasing the concentration to $C = 0.15$ reduces visibly the thickness of the mushy region (Figs. 7(d) and (c)). Comparing Figs. 3(c) and 7(b) shows clearly that the effect of the bottom wall heating is to retard drastically the solidification phenomenon and reduce the thickness of the solid and the mushy zone. On the other hand, the shape of the solid/mush and the mush/liquid interfaces is not significantly altered by the bottom wall heating.

The thickness of the mixed phase region and the position of the solid/mush and the mush/liquid interface at the centerline of the cavity for $Q = 36$ W is shown in Fig. 4(b). Overall, the mushy zone thickens as time progresses, however, this thickening effect is considerably weaker than what was observed in Fig. 4(a) ($Q = 0$ W). As the concentration increases to $C = 0.1$ the thickness of the mushy zone becomes practically independent of time. Another interesting observation in Fig. 4(b) is that while the thickness of the mush/liquid interface noticeably decreases as the initial concentration increases, the thickness of the solid/mush interface remains practically unchanged. This finding is unlike Fig. 4(a) where increasing the initial concentration increased the thickness of the solid. It appears that the flow in the mushy region in Fig. 4(b) is strong enough (because of the bottom wall heating) to retard the growth of the solid.

The temperature variations in Fig. 5(b) indicate that the heat transfer in the solid and the mixed phase

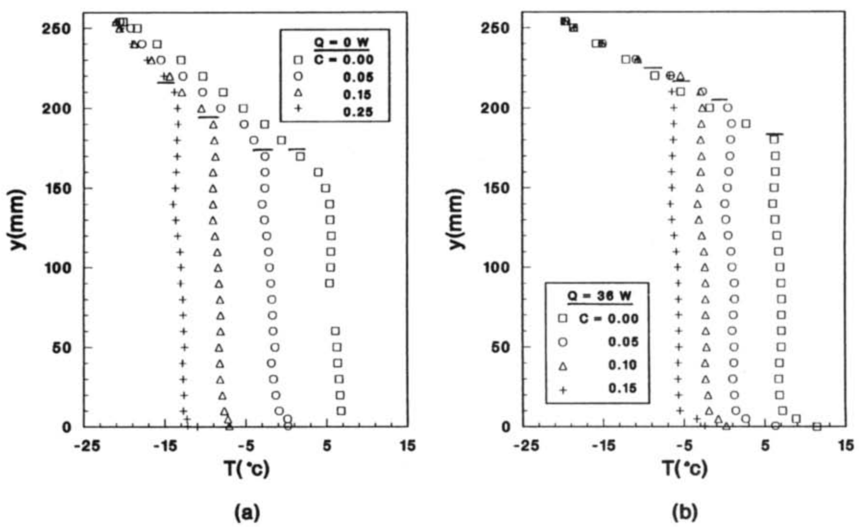


FIG. 5. Temperature variation in the vertical direction at the centerline of the cavity: (a) $Q = 0$ W; (b) $Q = 36$ W.

regions is still dominated by conduction. However, a sharp thermal boundary layer exists along the bottom heated wall. In the core region between the thermal boundary layer and the mush/liquid interface the fluid is practically isothermal.

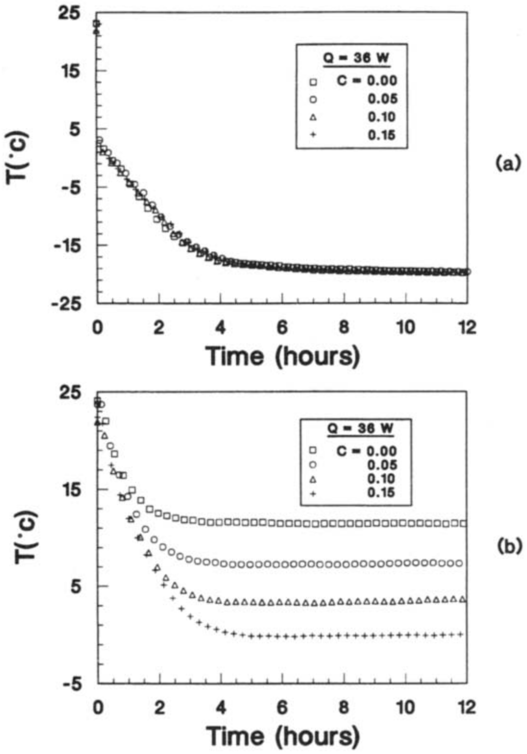


FIG. 6. (a) Top wall temperature variation with time for $Q = 36$ W. (b) Bottom wall temperature variation with time for $Q = 36$ W.

4. CONCLUSIONS

In this paper, an experimental investigation on the solidification of an alloy (water and ammonium chloride) in a rectangular cavity cooled through its top wall was presented. It was found that a mixed phase region of significant extent exists separating the solid phase from the liquid phase. The thickness of this region increases with time. The temperature distribution in the mixed phase region is linear indicating that conduction is the dominant heat transfer mechanism there, despite the fact that liquid motion takes place between the interweaving dendrites. Apparently, this motion is not strong enough to affect the temperature field.

In all cases, the solidification process started with the appearance of a mushy zone adjacent to the cooled top wall. The flow in the liquid region is characteristic of double diffusion with unstable concentration gradients. Salt fingers were clearly visible in the solid/liquid interface vicinity at high concentrations, caused by the precipitation of NH_4Cl. Increasing the initial concentration decreased the thickness of the mushy zone. The flow in the liquid phase was mainly bicellular but it did not affect the shape of the solid/mush or mush/liquid interface significantly. However, in the limit $C = 0$ (freezing of pure water) the bicellular flow noticeably altered the shape of the solid/liquid interface.

In summary, this study reported observations and measurements relevant to the complex flow, heat and mass transfer phenomena that occur during the freezing of alloys. These phenomena drastically affect the solidification phenomenon and, therefore, the growth rate and structure of the resulting solid.

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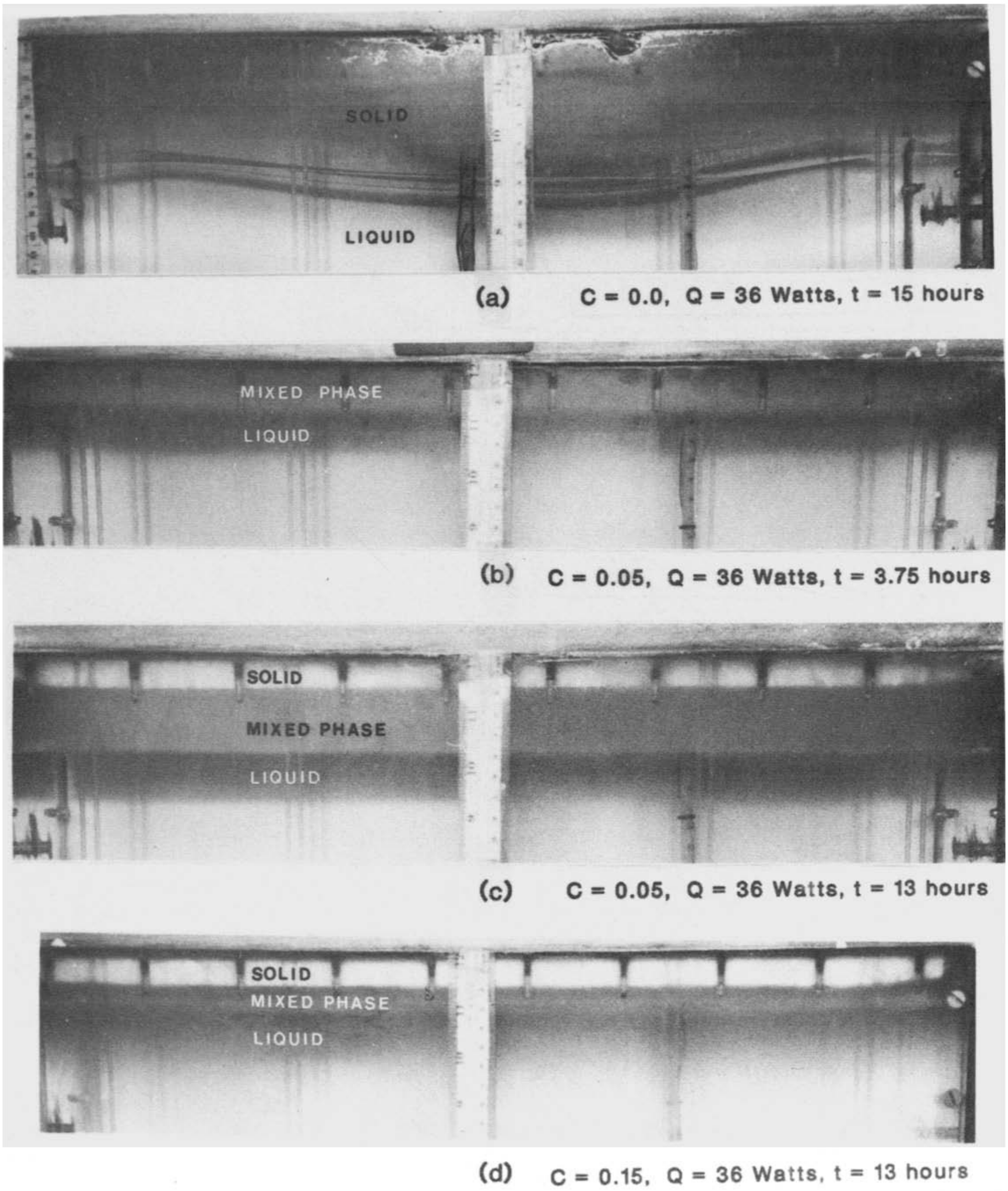

FIG. 7. Sequence of photographs documenting the solidification process for $Q = 36$ W: (a) $C = 0$, $t = 15$ h; (b) $C = 0.05$, $t = 3.75$ h; (c) $C = 0.05$, $t = 13$ h; (d) $C = 0.15$, $t = 13$ h.

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SOLIDIFICATION D'UN ALLIAGE DANS UNE CAVITE REFROIDIE A TRAVERS SA SURFACE SUPERIEURE

Résumé—On décrit une étude expérimentale du phénomène de solidification d'un alliage binaire (eau et chlorure d'ammonium) dans une cavité refroidie à sa surface supérieure. Il existe trois régions distinctes dans la cavité pendant le mécanisme de solidification : une région occupée par le solide, une région occupée par le liquide et une phase mixte (zone de bouillie) avec une combinaison complexe de dendrites et de liquide. La région de phase mixte est d'une grande étendue, souvent plus grande que celle du solide. La croissance dans le temps de l'interface solide/bouillie et de l'interface bouillie/liquide ainsi que le champ de température dans la cavité sont déterminés par un ensemble de concentrations initiales. Le champ de température dans la phase mixte et la région solide est dominé par la conduction. Apparemment, dans les expériences, l'écoulement dans la bouillie n'est pas suffisamment important pour rendre sensible la convection thermique. L'écoulement a un effet sur la forme de l'interface bouillie/liquide. Cet effet est néanmoins faible et l'interface reste plutôt plan pour les fortes concentrations. La convection doublement diffusive prend place dans la région liquide parce que le chlorure d'ammonium est constamment déchargé dans le liquide quand la solidification progresse vers le bas à partir de la paroi supérieure. On observe une digitation salée qui pénètre dans la région liquide. L'épaisseur de la zone de bouillie croît généralement dans le temps. Un accroissement de la concentration initiale de l'alliance réduit l'épaisseur de la région de phase mixte.

ERSTARRUNG EINER MISCHUNG IN EINEM VON OBEN GEKÜHLTEN BEHÄLTER

Zusammenfassung—Die Erstarrung einer binären Mischung (Wasser und Ammoniumchlorid) in einem von oben gekühlten Behälter wird experimentell untersucht. Während des Erstarrungsvorgangs gibt es drei abgegrenzte Gebiete im Hohlraum : ein Gebiet besteht aus Feststoff, ein weiteres aus Flüssigkeit, dazwischen gibt es ein Mischphasengebiet (Erweichungszone) aus einer komplexen Kombination von Dendriten und Flüssigkeit. Das Mischphasengebiet hat eine beträchtliche Ausdehnung und ist oft größer als das des Feststoffs. Das Wachstum der Grenzflächen zwischen den Gebieten und die Temperaturverteilung im Behälter wird für eine große Zahl von Anfangskonzentrationen bestimmt. Das Temperaturfeld im Mischphasengebiet und in den festen Gebieten wird von Wärmeleitung bestimmt, obwohl dieses Gebiet durchlässig ist und Strömungen erlaubt. Anscheinend war bei den Versuchen die Strömungsgeschwindigkeit im Mischphasengebiet nicht groß genug, um die Konvektion zum bestimmenden Wärmetransportvorgang zu machen. Im Flüssigkeitsgebiet herrscht ein bizellulares Strömungsfeld. Die Strömung beeinflußt die Form der Grenzfläche zum Mischphasengebiet hin. Dieser Einfluß ist jedoch gering, und die Grenzfläche bleibt speziell bei hohen Konzentrationen überwiegend flach. Im Flüssigkeitsgebiet findet doppelt-diffusive Konvektion statt, da Ammoniumchlorid ständig in die Flüssigkeit entweicht, solange die Erstarrung nach unten fortschreitet, nachdem sie in der Nähe der Deckfläche begonnen hat. Es ist klar sichtbar, daß Salzfinger in das Flüssigkeitsgebiet eindringen. Die Dicke des Mischphasengebiets vergrößert sich im allgemeinen mit der Zeit. Eine Erhöhung der Anfangskonzentration der Mischung verringert die Dicke des Mischphasengebiets.

ЗАТВЕРДЕВАНИЕ БИНАРНОГО РАСТВОРА В ПОЛОСТИ С ОХЛАЖДАЕМОЙ ВЕРХНЕЙ ПОВЕРХНОСТЬЮ

Аннотация—Экспериментально исследуется затвердевание бинарного раствора (воды и хлористого аммония) в полости с охлаждаемой верхней стенкой. При этом наблюдаются четко различаемые области твердой и жидкой фаз, а также пористая зона, состоящая из сложных образований дендритов и жидкости. Пористая зона имеет значительную протяженность, зачастую больше, чем область твердой фазы. Определена эволюция границ раздела между твердой и пористой, пористой и жидкой фазами, а также распределение температур в полости для самых различных значений начальной концентрации. Несмотря на то, что пористая область является проницаемой для течения, доминирующим фактором в эволюции температурного поля в областях смешанных и твердой фаз является теплопроводность. Очевидно, что в данных экспериментах течение в пористой фазе не является достаточно сильным для того, чтобы конвекция стала преобладающим процессом в теплопереносе в пористой фазе. В жидкой зоне существует поле двухъячеистого течения. Течение оказывает влияние на форму границы раздела пористой и жидкой фаз. Однако это влияние слабое, и межфазная граница остается преимущественно плоской, особенно при больших концентрация. В жидкой области происходит двойная диффузия, обусловленная непрерывным поступлением хлористого аммония в жидкость по мере продвижения границы затвердевания вниз после его начала вблизи верхней стенки. Отчетливо прослеживается проникновение солевых дендритов в жидкую область. Как правило, толщина пористой зоны увеличивается во времени. С ростом начальной концентрации раствора толщина области смешанных фаз уменьшается.