Solid/Liquid Phase Change: Recent Studies and Models

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Abstract: Some problems related to solid/liquid phase change are presented. Attention is focused on interface modeling for numerical analysis and one-dimensional directional growing and melting. Microgravity relevance of some situations is emphasized. It is shown, in particular, that in some circumstances melting is not the simple reversal of crystal growth due to some (still poorly known) phenomena (nucleation and growth of liquid droplets in the bulk, solid and liquid dendrites due to a morphological instability of the phase boundary). Relevant mathematical models are discussed and described (to a certain extent) for analysis and/or characterization of these phenomena when they are disjoint or partially combined. Some effort is provided to model phenomena occurring at both the microscopic and macroscopic scale lengths.

1 Introduction

Solid/liquid phase change phenomena are sensitive to gravity. On Earth natural convection induced by thermal or solutal buoyancy forces occurs during solidification, and as a consequence, the interfacial zone is often very complex. Front curvature and solid phase structure depend on several factors. Microgravity can help in the understanding of such factors through removal of buoyancy convection that tends to be a dominating effect in normal gravity conditions.

Experiments under microgravity conditions are usually performed using drop towers, parabolic flight, sounding rockets and satellites. French Research activity on that field particularly developed in the Group: "Transport Phenomena and phase transitions in Microgravity" GDR - P2TPM, (presently GDR-MFA for Fundamental and Applied Microgravity) of Centre National d'Etudes Spatiales (CNES) / Centre National de la Recherche Scientifique (CNRS) [Prud'homme & Zappoli, 2003; Prud'homme et al., 2001; Prud'homme & El Ganaoui, 2004, a, c].

This review runs as follows: Various fundamental considerations are given in section "Basic considerations": - the fluid motion caused by gravity is characterized by relevant dimensionless numbers (Sect. 2.1) - the couplings between the force of gravity and other forces (surface tension, crystalline force) are evoked in Sect. 2.2 - the effects of the convection on the morphology of alloys are presented in relation to classical theories and recent results (Sect. 2.3) - finally, the attention is focused on the significant problem related to the conditions of preparation of the samples to solidify (Sect. 2.4). Section 3 "Interfacial modeling and applications" is devoted to modeling necessary to the analysis of various processes: - modeling of the liquid solid/transition - modeling of the type "phase field ": on a macroscopic scale to reproduce the total shape of the interfaces of phase change, then on a microscopic scale to study the fine structure of the formation of eutectics in directional solidification. The last section is devoted to additional considerations not discussed in the earlier sections.

Of course, the present review is necessarily limited in scope and depth, both because of the page limit and because of the amount of published literature on these subjects that is now enormous. Consequently, the emphasized topics have been selected according to our opinion and experience. Owing to the elaborate nature of some of these studies it has not always been possible to fit an adequate account of them into the framework of the present analysis, but attempts are made to give

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some indication of the most important results, and of the methods employed.

2 Basic Considerations

2.1 Gravitational convection

Convection during phase change can be driven by several forces. For typical solidification systems, unless the process takes place under microgravity conditions on orbiting space platforms, gravity is always present and thermal or compositional non-homogeneities give rise to density differences. According to the mutual orientation of the gravity vector and the density gradient, buoyancy driven convection can arise significantly altering the thermal and compositional state of the system, and affecting the solid/liquid interface shape and motion. This microgravity relevance for fluid flows with large density gradients and negligible forced convection is generally encountered for Grashof number [Landau & Lifschitz, 1971] of orders one or larger:

$$Gr = \Delta \rho \, g \, L^3 / v^2 \qquad (1)$$

Gr permits to compare the effects of the buoyancy force and the viscous drag force [Prud'homme & El Ganaoui, 2004 b]. In this formula, $\Delta \rho$ is the local density deviation v is the kinematical viscosity, L is a characteristic length, and g is the gravity acceleration.

The Rayleigh number is more appropriate than Grashof number when the fluid is a liquid and when coupling of the momentum equation with energy equation [Chandrasekhar, 1961] and sometimes with mass balances of species- happens. Two non-dimensional numbers are then used according to the studied case:

$$Ra_T = Gr_T \, Pr, \quad Ra_S = Gr_S Sc \qquad (2)$$

Where subscripts $(_T)$ and $(_S)$ refer to the thermal and the solutal origin of $\Delta \rho$: $\Delta \rho = \beta_T \Delta T$ or $\Delta \rho = \beta_S \Delta C$ respectively. The classical Rayleigh-Bénard thermal instability occurs in a fluid layer heated from below (see Lappa [2005a], Punjabi et al. [2006]). However, in typical solidification processes, horizontal thermal gradients can also

occur and drive natural convection [Benielli et al., 2001]. Instabilities appear when the appropriate Rayleigh numbers reach critical values depending on the considered processes.

2.2 Flow instabilities and possible control strategies

As explained above, in terrestrial solidification (and melting) problems, flow instabilities occur when the Rayleigh number becomes higher than a critical value, which depends in particular on the geometrical configuration.

If a free surface separating the liquid phase and a surrounding ambient gas is present, an additional type of convection can appear. A surface tension gradient caused by thermal and/or compositional non-homogeneities along the interface can lead, in fact, to Marangoni convection (a typical example is solidification by the floating zone technique, see Lappa, 2005b, Gelfgat et al., 2005; Lan and Yeh, 2005; or other solidification procedures in which the contact between the fluid phase and the wall of the crucible is not ensured, see Scriven and Sternling, 1964; Bennacer et al., 2002, 2004; Amberg and Shiomi, 2005; Tsukada, Kobayashi, Jing and Imaishi, 2005; Li, Peng, Shi and Imaishi, 2006; El-Gamma and Floryan, 2006; Matsunaga and Kawamura, 2006).

When a free surface is present phase change may also interact with other types of hydrodynamic instabilities, such as the Marangoni-Bénard flow [Nield, 1964; El Ganaoui, 2002; Bergeon et al., 1994; Timchenko et al., 2002].

It is known that magnetic fields can be used for compensating gravity effects [Lehmann et al., 1999; Touhiri et al., 1999; Giessler et al., 2005; Ma and Walker, 2006, Kakimoto and Liu, 2006]. Lappa (2005b) investigated the possibility to use magnetic fields to control also the instabilities related to Marangoni flow.

For some materials, the crystalline anisotropy can interact with the phenomena mentioned above. Both the kinetics of growth and surface tension are sensitive to crystal anisotropy, and there are consequences on morphologies resulting from growth or melting [Debierre et al., 2003].

2.3 Morphological instabilities in directional solidification

It has been noted that convection effects strongly influence the development of solidification microstructures. However, most microstructure models are based on purely diffusive transport mechanisms. For this reason, crucial benchmark data required to test fundamental theories of micro structural pattern formation have resulted from solidification experiments conducted in microgravity environment, where convection effects can be substantially reduced, and/or from ground based experiments with restricted samples sizes [Karma, 2001].

The conditions for instabilities to appear in directional solidification of dilute alloys were initially given by Mullins and Sekerka [1964].

In the case of binary lamellar eutectics, relations between the lamellar characteristic spacing, the temperature gradient and the front velocity were found first by Jackson and Hunt [1965], who also gave a first estimate of the stability bounds of the lamellar pattern.
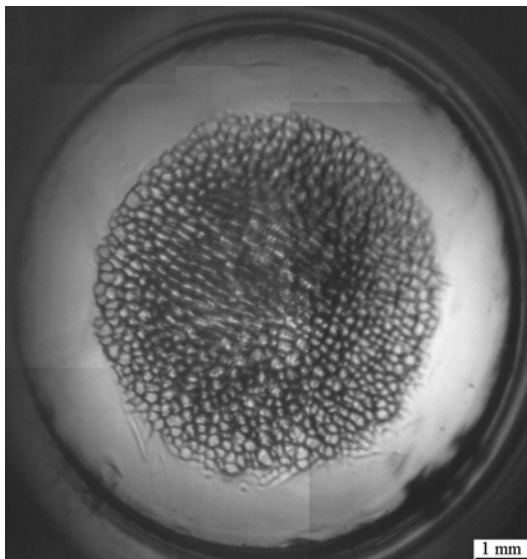

Figure 1: Interface observation from the top in growth direction for a thick solid seed: cells are localised in the middle of the interface. (SCN-0.2%Acetone, V=1.4 μm/s) [Benielli et al., 2001].

These theories apply to highly idealized systems [Langer, 1986]. They cannot explain complex phenomena observed in real systems such as curvature of the front in relation to convective instabilities (figure 1), or eutectic colony formation. The later phenomena have been observed in the thin-sample directional solidification of the transparent eutectic alloy CBr_4-C_2Cl_6 showing the existence of colonies when impurities are present [Akamatsu & Faivre, 2000]. The transition between the planar lamellar eutectic front and the eutectic colonies goes through intermediate structures as traveling waves and a two-phase finger, which have been recently observed and are currently studied by linearized theories or by phase field methods (see Section Phase field model for microstructures). General stability problems in eutectic solidification have been examined thoroughly by Akamatsu et al. (2002; 2004).

2.4 Melting and thermal stabilization

Stages preparatory to directional solidification greatly affect the initial state at which growth is started [Benielli et al., 2002], and therefore the directional solidification itself [Akamatsu & Faivre, 2000].

Nguyen Thi et al. (2001) have stressed the importance of microstructures for the resulting properties of the materials. Spatial experiments *LMS* and *STS-95* on directional solidification of Al-3.5 wt% Ni alloy enabled them to understand the role played by the preparatory phases of melting and stabilization and the initial directional-solidification transient.

In such a context, it has been pointed out that melting is not the simple reverse of crystal growth. Two phenomena have been observed in situ and in real time during the directional melting of transparent succinonitrile-acetone alloys in a cylinder: nucleation and growth of liquid droplets in the bulk solid and liquid dendrites due to morphological instability of the phase boundary (relevant models and additional information are provided in Sect. 3.5). Benielli et al. [Benielli et al, 2001; 2002] analyzed the dynamics of melting morphology at the solid-liquid interface. These authors have emphasized the importance of carry-

ing out experiments in the limit of diffusive transport of solute in the liquid, which imposes to work in the absence of gravity [Prud'homme & Zappoli, 2003].

3   Interfacial Modeling and Applications

3.1   Solid/liquid transition zone

The study of the solid/liquid transition zone is often very complex. In some cases of directional solidification in alloys, the solidification front appears as a smooth (but generally non planar) surface with local thermo-chemical equilibrium between the solid and the liquid. In other cases, for sufficiently high values of pulling velocity, microstructures resulting from an instability process appear. In the case of lamellar eutectic alloys, it is important to determine the size and distribution of microstructures in relation to the external constraints. The interfacial morphology becomes very complicated for dendrites and for dendritic layers.

Local balance equations resulting from the irreversible thermodynamics can be written in each case, both for the interface and for the bulk phases. A critical problem for numerical solution is the presence of an interfacial discontinuity at the boundary between the phases [Ludwig, Gruber-Pretzler, Wu, Kuhn and Riedle (2005); Abhilash, Joseph and Krishna, (2006); Hong, Zhu and Lee, (2006); Narski and Picasso, (2007)]. To avoid this difficulty, some authors replaced the discontinuity by a continuous description of the transition region. This is the concept of phase field. Two classes of phase field methods, which differ by the scale of the description (macroscopic, and microscopic -or mesoscopic, but not molecular) are presented here.

One of the macroscopic methods is applied to dendritic-like porous structures. The main difference with the other cases is that a 'mushy zone' between the fully solid and the liquid phases is considered [Camel and Arquis, 1992; Goyeau et al., 1999]. The Darcy source term $-\left(\mu_1\rho/K\rho_1\right)(\vec{v}-\vec{v}_S)$ appears on the right-hand side of the momentum equation, where K denotes the isotropic permeability and represents a direct link between the microscopic morphology and the macroscopic equations of conservation. Here, K is a scalar for simplicity [Benielli et al., 2001, Akamatsu & Faivre, 2000]. It is expressed as a function of the volume fraction ϕ of the liquid. In more general case it becomes a second order tensor. Closure relations were given by [Goyeau et al., 1999; Favier & Camel, 1986].

3.2   Enthalpy method: a homogeneous macroscopic approach

The enthalpy approach for directional solidification [Benielli et al., 2001; El Ganaoui, 2002; El Ganaoui et al., 2002; Guérin et al., 2001] considers a single set of homogenous balance equations adapted to the problem of phase change in a 2D configuration, as explained in [Prud'homme & El Ganaoui, 2004].

In these equations, ρ is an average density weighted by the liquid and solid mass fractions, $\vec{v}$, $\vec{v}_S$, C and h are averaged local quantities weighted by the liquid and solid volume fractions. For instance one has. $C=\phi C_l+(1-\phi)C_s$.

With this model, including appropriate closure relations and boundary and initial conditions, it is possible to obtain isotherms and streamlines which show in particular, the position of a curved smooth interface. The numerical solution is obtained by the enthalpy model associated to a finite volume approximation. One obtains evolutions of the melt-solid interface, and of velocity and solutal fields. Illustrations of flow evolving from monocellular to multicellular convective cells with thermal Ra are given in figure 2 and coupling between buoyancy and magnetic effects in figure 3.

Other important applications in the field concern the floating zone configuration. Generally this configuration is investigated in a restricted situation without phase change [Bennacer et al, 2002]. The enthalpy formulation permits to access to the fully coupled problem, and to include the shape of the solid/liquid interface and its displacement. It is possible to quantify some external parameters effect and their use as controlling parameters (heat distribution, pulling velocity, etc.) [Bennacer et al, 2004].
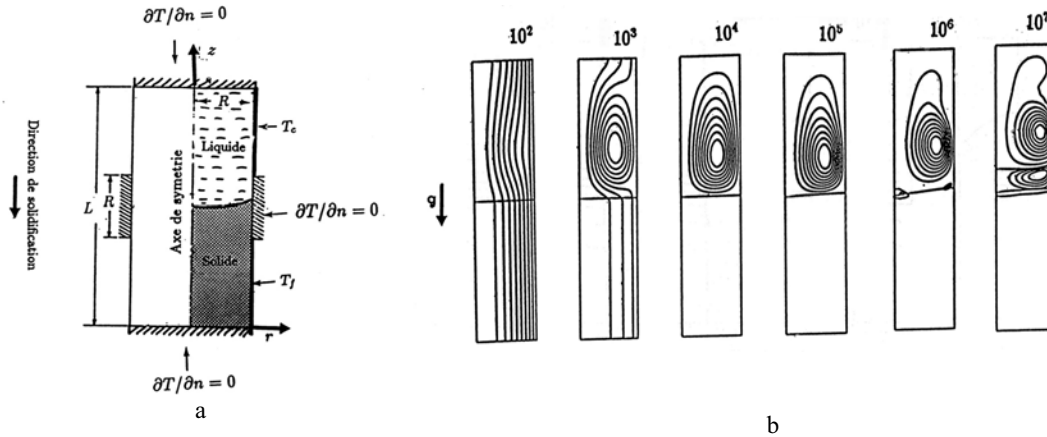
Figure 2: Illustration of the enthalpy-porosity based computations : melt under directional solidification using vertical Bridgman method (a) for $10^2 \leq Ra_T = g\beta(T_h - T_c)L^3/\nu\alpha \leq 10^7$ (i.e gravity effect) showing evolution from mono-cellular to multicellular flow (b). Simulation corresponding to an enthalpy porosity formulation/FiniteVolumes method [El Ganaoui, 2002].
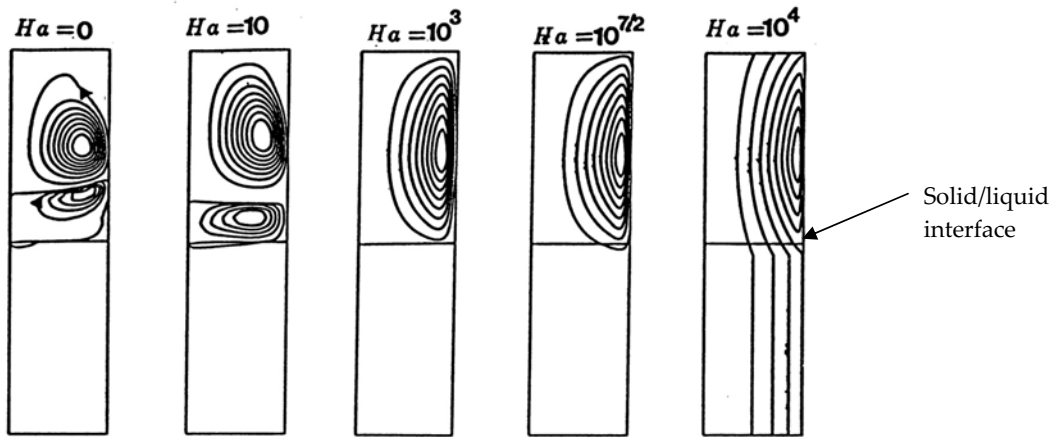

Figure 3: Illustration of the enthalpy-porosity based computations : effect of constant magnetic field ($0 \leq Ha = (\sigma BL^2/\mu)^{1/2} \leq 10^4$) opposite to gravity on multi-cellular flow corresponding to $Ra_T = 10^7$ for vertical Bridgman configuration [El Ganaoui, 2002].

Figure 4 (Benielli et al., 2001) illustrates the thermocapillary flow occurring in a horizontal Bridgman cavity with a non deformable free surface.

3.3 Phase field model for microstructures

To explain microstructures in constrained growth, and recently in eutectic colony formation [Akamatsu & Faivre, 2000], Plapp and Karma studied the problem first analytically [Plapp & Karma, 1999], and then numerically, using a phase field model [Plapp & Karma, 2002]. They studied a ternary alloy close to a binary eutectic, assuming for simplicity a symmetric phase diagram. They introduced a phase field $\phi(x,z,t)$ that distinguishes between solid and liquid, i.e., a scalar field that has a fixed value in the bulk phases and varies continuously across the smooth, diffuse interface. They started with a dimensionless Helmholtz free energy functional and deduced, by minimization, three local equations between three variables, which are valid everywhere under appropriate assumptions.

In the obtained equations, reminded in Prud'homme & El Ganaoui (2004), u is a
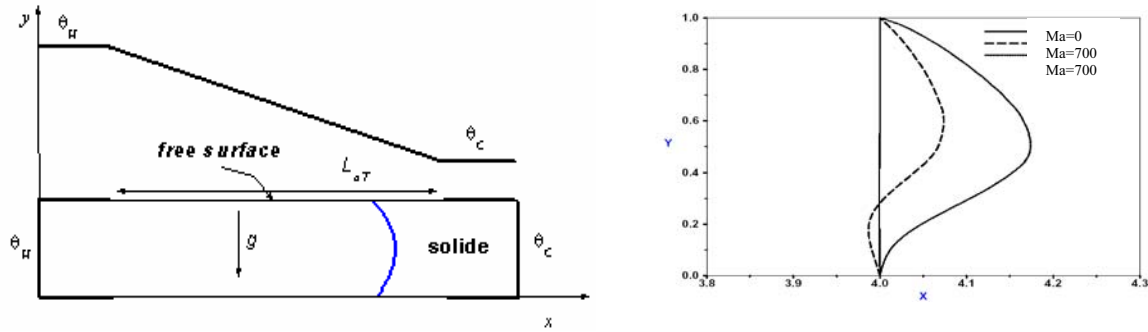
Figure 4: Illustration of the enthalpy-porosity based computations: Schematic of physical system and boundary (right) followed by the solid/liquid interface form under Marangoni effects for Grashof number Gr=1000 ($Ma = |\partial \sigma / \partial T| \Delta TH / \mu$, σ is the surface tension, μ the kinematics viscosity and H the cavity height).

scaled concentration $u(x,z,t) = \frac{C(x,z,t)-C_E}{(C_\beta - C_\alpha)/2}$, where C_E, C_α and C_β are the compositions of the liquid and the two solid phases in the pure binary eutectic at the eutectic temperature T_E, $\tilde{C}(x, z, t)$ is the concentration of impurities, $h(\phi) = \frac{3}{2}\left(\phi - \frac{\phi^3}{3}\right)$ is an interpolation function, $f_l\left(u, \tilde{C}, T\right)$ and $f_s\left(u, \tilde{C}, T\right)$ are the free energy densities for liquid and solid respectively, W_u and W_ϕ are the characteristic thickness of the solid-solid and of the solid-liquid interfaces respectively, and $M\left(\phi, u, \tilde{C}\right)$ and $\tilde{M}\left(\phi, u, \tilde{C}\right)$ are the mobility functions of the eutectic component and the ternary eutectic, respectively.
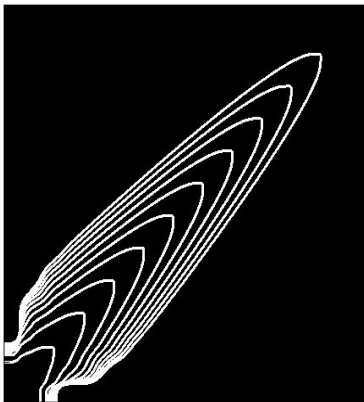

Figure 5: Dendritic growing of a partially facetted crystal from a pure liquid (phase-field numerical simulation) [Debierre et al., 2003].

Numerical simulations performed with this phase field model have enabled the authors to carry out large-scale simulations of colony formation starting with arrays of up to 40 lamella pairs [Plapp & Karma, 2002]. Debierre et al. (2003) extended this method to faceted materials (Figure 5).

The phase field model must be extended to 2D interfaces in 3D configurations [Plapp & Dejmeck, 2004]. Akamatsu et al. (2001) compared results obtained in thin and massive sample experiments. In the first case, one has generally a flat solid-melt interface. In the last case, the 2D simulated solidification front presents a curvature. This curvature is of course influenced by gravity but it exists also in micro-gravity. Thermosolutal convection will also lead to modifications in local dynamics of eutectic fronts and can also cause a macrosegregation.

3.4 Fluid-fluid interface

In the case of directional solidification by the floating-zone technique, we have to study a process involving three phases. Especially, a liquid-gas interface does exist implying thermal and solutal Marangoni effects.

Some fluid-fluid interfaces have been studied by second gradient methods [Casal, 1972; Seppecher, 1987]. In second gradient model, internal energy per unit mass depends not only on the entropy and the density but also on the magnitude of the density gradient [Gatignol & Prud'homme, 2001]. A coefficient λ, called the "capillarity coefficient ", is introduced.

Using average quantities, and introducing the vol-

umetric masses of the species and their gradients, this type of model can be certainly extended to solidification fronts in mixtures and can be considered as a phase field model [Gatignol & Prud'homme, 2001; Gouin, 1998].

A phase-field model has been recently introduced by Jamet & Ruyer (2004) to study liquid-vapor flows with phase-change.

3.5 Directional melting and thermal stabilization

As anticipated in Sect. 2.4, studying directional melting and thermal stabilization stages preparatory to directional solidification of transparent succinonitrile-acetone alloys, Benielli et al. (2002) observed two categories of phenomena: melting morphologies resulting from the instability of the liquid-solid interface and liquid droplets in the superheated solid (Figure 6).

The criterion for the occurrence of *morphological instability* in directional melting is given by:

$$V \; > \; V_{MI} = \; (k D_S + D_L) \; G \; / \; [m C_0 (k-1)]$$
$$\cong \; D_L \; G \; / \; [m C_0 (k-1)] \qquad (3)$$

where V_{MI} is the critical melting velocity, C_0 is the initial solute concentration, D_S, D_L are the diffusion coefficients for the solid and the liquid respectively. The orientation of dendrites with respect to the liquid-solid interface is determined by the anisotropy of solid succinonitrile that favors growth in $< 100 >$ direction, and the orientation of the grain they occupy.

In many circumstances, however, the observed morphological instability structure depends on local experimental conditions: buoyancy-driven fluid flow is always present, and the formula may be applied with C_{loc} instead of C_0.

Benielli et al. (2002) observed and modeled the phenomenon related to *droplet growth in superheated solids* during directional melting. The length L_{SH} of the superheated solid can be derived from the phase diagram under the restrictive conditions

$$L_{SH} = \; m \; C_0 \; (k-1) \big/ G \; k \qquad (4)$$

The global droplet velocity is the difference $V_d = V_P - V_{mig}$ between the pulling velocity and the migration velocity

$$V_{mig} \; = \; -G \; D_L \big/ [m \; C_L \; (k-1)] \qquad (5)$$

The mechanism of migration of the liquid droplets by TGZM (Temperature Gradient Zone Melting) is elucidated and well explained in the cited papers: the hot and cold sides of the droplet having different compositions (local equilibrium holds at solid-liquid interface of the droplet), there is a concentration gradient across the liquid droplet and solute diffuses from the cold side to the hot side. This causes solidification at the cold side and melting at the hot one, so that a droplet migrates up the thermal gradient.

The authors deduce a droplet lifetime τ as a function of non-dimensional nucleation depth $f(f = Zi / L_{SH}$, where Z_i is the nucleation depth, i.e. the distance between the nucleation of the droplet and the solid/bulk liquid interface).

The modeling of the growth of the droplets can be performed assuming a separation between the one-dimensional growth in z direction controlled by TGZM, and the free growth of the 2D cross section at $z = 0$ (at the point of nucleation of the considered droplet). For the latter phenomena, the considered equation is given by

$$\frac{\partial^2 C_0 (r, 0)}{\partial r^2} \; + \; \frac{1}{r} \; \frac{\partial C_0 (r, 0)}{\partial r} \; = \; 0 \qquad (6)$$

Solute conservation equation at the droplet solid/liquid interface is

$$[C_L \, (T) \, - \, C_S \, (T)] \, V_R \; = \; D_S \; \left. \frac{\partial \, C (r, 0)}{\partial \, r} \right|_R \qquad (7)$$

where V_R the growth velocity and T satisfies

$$T = T_0 - \; G \; (Z - Z_0) \, + \, G \, V_P \; (t - t_0) \qquad (8)$$

in a frame attached to the crucible, T_0 is the liquidus temperature for the solute concentration C_0 and Z_0 is the vertical position of the isotherm T_0 at time t_0 where the droplet appears. A concentration boundary layer is assumed in the solid surrounding the liquid droplet. The equations are
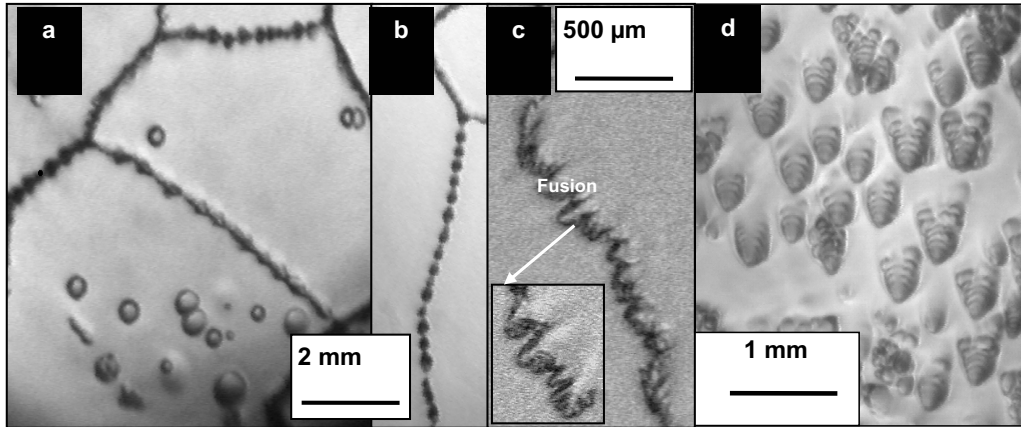
Figure 6: a) Liquid droplets inside grains – b), c) Morphological instability at grain boundaries – d) Arrays of melting dendrites in a grain [Benielli et al., 2002].

solved by a finite difference method. The found time evolution of the droplet is in good agreement with the experimental results, obtained for different pulling rates.

Liquid droplets were also observed in the case of Al-1.5 wt% Ni directional melting followed by the thermal stabilization [Nguyen Thi et al., 2003]. In addition, there are liquid channels separating the solid grains. These structures appear in a mushy zone whose thickness can be evaluated. They migrate during the thermal stabilization period. The mechanism of migration of the Ni rich liquid droplets by TGZM is the same as for the previous example.

A simple numerical model has been derived to describe the directional melting and the thermal stabilization regime. If $\rho(X, t)$) is the density number of droplets at time t and altitude X from the solid base of the mushy zone, neglecting droplet coalescence, we obtain the balance equation for droplet (or horizontal channel) concentration.

$$\frac{\partial \rho}{\partial t} + \frac{\partial (\rho \, V_{mig})}{\partial X} = 0 \qquad (9)$$

where V_{mig} is the migration velocity given by eq. (5).

During the melting phase $t \leq t_0$, a moving boundary condition $\rho(X_B, t)$ is used at the altitude X_B where $T(X_B, t) = T_E$.

Stopping the furnace at time t_0, with the temperature remaining constant during the stabilization,

the boundary condition becomes $\rho(0, t) \approx 0$. Eq. (9) can be solved by a finite difference scheme on a one-dimensional grid.

Comparison between the numerical and the experimental results has shown a good agreement in the case of micro-gravity experiment, but is less satisfactory on Earth.

3.6 *Other Relevant Solidification Aspects*

To complement the previous sections and conclude this review, some solidification problems relevant to microgravity experiments are briefly discussed.

Berne, Pasturel & Vinet (2001) studied crystalline nucleation and phase selection in refractory metals and alloys in the 50-meters high Grenoble drop-tube of CEA. They emphasize the importance of microgravity and ultra-vacuum to ensure absolute no-contact conditions and purification by evaporation of really spherical droplets.

Camel & Dupouy (2001) have studied the influence of natural convection on the formation of dendritic microstructures in the columnar/equiaxed transition range in the specific case of Al-3.5 wt% Ni alloy.

Process control by vibration was studied for stabilizing solidification fronts and obtaining planar fronts. Reduced gravity experiments are to be performed in this field, for a better understanding of the effects of vibrating fields on fluids, interfaces

and multiphase flows [Lyubimov et al., 1997].

Finally it should be kept in mind that, even in microgravity conditions, solidification remains sensitive to the gravity level and orientation as shown by Simpson and Garimella (2000).

Nomenclature

Gr	Grashof number
Pr	Prandtl number
Ra	Rayleigh number
C	solute concentration, average mass fraction
$\tilde{C}$	concentration of impurities
B	Magnetic field intensity
D	mass diffusivity
F	non-dimensional nucleation depth
f_l	volume fraction of the liquid
g	gravitational acceleration
G	thermal gradient
h	enthalpy per unit mass, interpolation function
k	thermal conductivity, segregation coefficient
K	isotropic permeability
L	reference length
m	liquidus slope
M	mobility function of the eutectic
$\tilde{M}$	mobility function of the impurities
r	radial coordinate
t	time
T, T_F	mixture temperature, melting temperature
u	x velocity component
v	z velocity component
V, V_t	melting velocity, pulling velocity
$\vec{v}$	local velocity
W	characteristic thickness
X	altitude from the solid base
x	horizontal coordinate
z	vertical coordinate
Zi	nucleation depth
β_T, β_S	thermal, resp. solutal, expansion coefficient of the liquid
Δ	jump
λ	thermal diffusivity, capillarity coefficient
μ, v	dynamical viscosity, kinematical viscosity
ρ	density, number density of droplets
ϕ	phase field
τ	droplet lifetime

Subscripts

E	eutectic
F	melting
P	pulling
SH	superheated
l, s,sl	liquid, solid, transition zone
mig	migration
α, β	solid phases in a binary eutectic

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