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PERTINENCE OF MICRO-GRAVITY STUDIES FOR SOLID/LIQUID PHASE CHANGE

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ABSTRACT

Some problems of solid/liquid phase changes are presented. Attention is focused on interface modeling for numerical analysis, and one-directional growing. Microgravity relevance of some situations is emphasized. Some activities of the French Research Group : "Transport Phenomena and Phase Transitions in Microgravity" (GDR-P2TPM) of Centre National d'Etudes Spatiales (CNES) / Centre National de la Recherche Scientifique (CNRS), are summarized.

INTRODUCTION

Solid/liquid phase changes are sensitive to gravity. Natural convection occurs coupled to solidification front motion. The interfacial zone is often very complex to describe.

Indeed, front curvature and solid phase structure depend on several factors and it will be interesting to eliminate some of them due to gravity for a best understanding of solidification process.

Experiments in microgravity conditions are necessary, in drop towers, parabolic flight, sounding rockets and satellites. For instance, an experimental facility, named DECLIC (Dispositif d'Etude de la Croissance et des Liquides Critiques), will take place in the ISS (International Space Station). Experiments on transparent material solidification will be performed in DECLIC.

At the same time it is very useful to perform calculations with and without gravity effects.

Process control by vibration or magnetic field is sometimes required to stabilize solidification fronts.

Several teams work on these topics in the French Research Group : "Transport Phenomena and phase transitions in Microgravity" (GDR - P2TPM - CNES/CNRS). The research topics of this team are the following [1]:

- Solidification of alloys (Bernard Billia, Laboratoire Matériaux et Microélectronique de Provence Marseille)
- Solidification fronts (Gabriel Faivre, Groupe de Physique des Solides Paris)
- Instabilities, crystal growth and microgravity (Daniel Henry, Hamda Benhadid, Laboratoire de Mécanique des Fluides et d'Acoustique de Lyon)
- Measurements of diffusion coefficients in liquid metals (Peter Lehmann, René Moreau, Elaboration par Procédés Magnétiques Grenoble)
- Elasticity and growth (Alain Pocheau, Institut

de Recherche sur les Phénomènes Hors d'Equilibre)

- Influence of external fields on segregation and structuration in solidification (Thierry Dufar, Elaboration par Procédés Magnétiques Grenoble)

- Numerical modeling (Patrick Bontoux, Pierre Haldenwang, Laboratoire de Modélisation et Simulation Numérique en Mécanique Marseille)

- Thermal, thermosolutal and thermovibrational convection in microgravity (Abdelkader Mojtabi, Institut de Mécanique des Fluides de Toulouse)

- High performance calculations in microgravity/vibrational convection (Bernard Roux, Laboratoire de Modélisation et Simulation Numérique en Mécanique Marseille)

- Elaboration, solidification, welding (Denis Camel, CEA Grenoble)

BASIC CONSIDERATIONS.

Natural convection.

Microgravity is relevant for fluid flows when density gradients are large and forced convection not too important. It is generally the case when Grashov number [2] is of order one or larger. Grashov number

$$Gr = \Delta \rho g L^3 / \nu^2 \quad (1)$$

permits to compare effects of buoyancy force to viscous drag force. It can be introduced writing momentum equation as follows:

$$\rho d\vec{U}/dt + \vec{\nabla} p' = \vec{g} \Delta \rho + \mu \vec{\nabla}^2 \vec{U} \quad (2)$$

In these formula, $\Delta \rho$ is the local density deviation

(one has $\Delta \rho = \rho - \rho_\infty$ where ρ_∞ corresponds to the liquid at rest at the same altitude), $\nu = \mu / \rho$ is the kinematical viscosity, p' is the pressure deviation from the hydrostatic equilibrium, \vec{U} and $d\vec{U}/dt$ are local fluid velocity and acceleration. Fluid velocity resulting from buoyancy can be approximated

$$\rho U \partial U / \partial x \approx g \Delta \rho \Rightarrow \rho U^2 / L \approx g \Delta \rho \Rightarrow U \approx \sqrt{L g \Delta \rho / \rho} \quad (3)$$

The resulting Reynolds number is then

$$Re = UL / \nu = \sqrt{gL^3 \Delta \rho / \rho \nu^2} = \sqrt{Gr} \quad (4)$$

Rayleigh number is more appropriate than Grashov number in the case of liquid phase, when coupling with energy equation [3] happens and, sometimes, with mass balances of species. Two non-dimensional numbers are then used according to the studied case

$$Ra_T = Gr_T Pr = g \beta_T \Delta T L^3 / \nu \lambda, Ra_C = Gr_C Sc = g \beta_C \Delta C L^3 / \nu D \quad (5)$$

where subscripts (T) and (C) refer to thermal and solutal origin of $\Delta \rho$: $\Delta \rho = \beta_T \Delta T$ or $\Delta \rho = \beta_C \Delta C$.

Rayleigh numbers take into account the experimental configuration. The classical Rayleigh-Bénard thermal instability occurs in a fluid layer heated from below. Solidification process is generally more complex; for instance, in directional solidification of alloys inside a cylindrical vertical crucible, the solidification layer is moving and the solidification front is not planar, upward temperature gradient is generally positive, there is a coupling between temperature and concentrations. Radial thermal gradients drive natural convection and modify concentration fields [4]. Instability occurs when the appropriate Rayleigh numbers reach critical values corresponding to the considered processes.

Instabilities and coupling.

In solidification problems, many sources of motion are present. Natural convection occurs when the Rayleigh number becomes higher than a critical value which depends in particular on the geometrical configuration. Increasing temperature (or concentration) gradient, higher order critical Rayleigh numbers appear, and resulting in very complicated situations. An other example of motion is produced by Marangoni effect and Marangoni instability [5] that can occur in floating zone solidification (or in other solidification procedure, when the contact between the fluid phase and the wall of the crucible is not ensured, as it is often the case in microgravity), where there is a liquid-gas or liquid-liquid (monotectic alloys) interface.

On the ground, this other effect is coupled with Rayleigh-Bénard instability [6]. Marangoni-Bénard instability can be coupled to Soret effect, possibly in microgravity [7]. Magnetic field is used for measuring diffusion coefficients in liquids [8], or for compensating for gravity, and numerical studies have been performed about coupling between buoyancy and magnetic effects [9].

MORPHOLOGICAL INSTABILITIES IN DIRECTIONAL SOLIDIFICATION.

"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 microstructural 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 where convection effects can be eliminated" [10].

The conditions for instabilities to appear in directional solidification of dilute alloys were given by Mullins and Sekerka [11].

In the case of binary lamellar eutectics, relations between lamellar characteristic spacing, temperature gradient and front velocity has been found first by Hunt and Jackson, who also gave a first estimate of the stability bounds of the lamellar pattern [12].

These theories apply to highly idealized systems. They can not explain complex phenomena observed in real systems such as curvature of the front in relation to convective instabilities, or eutectic colony formation. These last phenomena have been observed in thin-sample directional solidification of the transparent eutectic alloy $CBr_4-C_2Cl_6$. Fig. 1 shows existence of colonies when impurities are present. The transition between planar lamellar eutectic front and eutectic colonies goes through intermediate structures as traveling waves and two-phase fingers which has been recently observed [13] and are now studied by linearized theory or by phase field method (see Section Phase field model for microstructures).

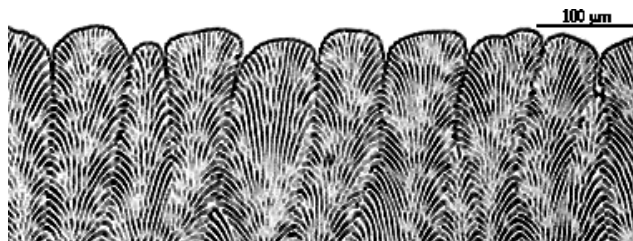


Fig. 1. Eutectic colonies in $CBr_4-C_2Cl_6$ -Naphtalene,

$$V = 31 \mu m s^{-1} [13].$$

INTERFACIAL MODELING AND APPLICATIONS.

Transition-zone.

Interfacial zone is often very complex to study. 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 solid and liquid. In other cases, for sufficiently high values of pulling velocity, microstructures resulting from an instability process appear, but the interface remains relatively smooth. 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 irreversible thermodynamics can be written in each case, both for the interface and for the bulk phases. A critical problem for numerical resolution is the presence of an interfacial discontinuity at the boundary between phases. To avoid this difficulty, some authors have replaced the discontinuity by a continuous description of the transition region. This is the concept of phase field. Two classes of phase field method, which differ by the scale of the description (macroscopic, and microscopic -or mesoscopic, but not molecular) are presented here.

One of the macroscopic method is applied to dendritic-like porous structures. The main difference with the other cases is that a mushy zone between fully solid and liquid phases is considered [14, 15]. In this method of prescribing a Darcy source term the velocity value arising from the solution of the momentum equation are inhibited, reaching values close to zero on complete solid formation. The Darcy

source term $-\frac{\mu_1 \rho}{K \rho_1}(\vec{v}-\vec{v}_s)$ appears on the right-hand

side of momentum equation where K denotes the isotropic permeability and represents the direct link between microscopic morphology and the macroscopic equations of conservation. Here, K is a scalar for simplicity [14]. It is expressed as a function of the volume fraction f_1 of the liquid. In more

general case it becomes a second order tensor. Closure relations where given [15, 16] for the isotropic case, and more recently for the non isotropic one. There is not place to give more details on this method.

In the following, basic equations of a macroscopic model, which is called enthalpy method, and some results of its application to particular cases, are given. Then the phase field model for microstructures is briefly presented.

The two methods give continuous balance equations that are valid in the whole considered volume, containing solid and fluid phases.

Enthalpy method.

Benielli et al. [4], El Ganaoui [17] and Guérin et al.[18] consider a single set of homogenous balance equations adapted to the problem of phase change in a 2D configuration:

$$\frac{\partial p}{\partial t} + \nabla \cdot (\rho \vec{v}) = 0 \quad (6)$$

$$\frac{\partial \rho C}{\partial t} + \nabla \cdot (\rho \vec{v} C) = \nabla \cdot (\rho D_1 \nabla C_1) + \nabla \cdot (\rho (C_1 - C)(\vec{v} - \vec{v}_s)) \quad (7)$$

$$\frac{\partial \rho u}{\partial t} + \nabla \cdot (\rho \vec{v} u) = -\frac{\partial p}{\partial x} + \nabla \cdot (\mu \frac{\rho}{\rho_1} \nabla u) - \frac{\mu}{K \rho_1} \rho (u - u_s) \quad (8)$$

$$\frac{\partial \rho v}{\partial t} + \nabla \cdot (\rho \vec{v} v) = -\frac{\partial p}{\partial y} + \nabla \cdot (\mu \frac{\rho}{\rho_1} \nabla v) - \frac{\mu}{K \rho_1} \rho (v - v_s) \quad (9)$$

$$+ \rho \frac{g \beta}{T_F} (T - T_F)$$

$$\frac{\partial \rho h}{\partial t} + \nabla \cdot (\rho \vec{v} h) = \nabla \cdot (\frac{k}{C} \nabla h) + \nabla \cdot (\frac{k}{C} \nabla (h_s - h)) + \nabla \cdot (\rho (h_1 - h)(\vec{v} - \vec{v}_s)) \quad (10)$$

where 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: $C = f_1 C_1 + (1 - f_1) C_s$.

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.

Fig. 2 is obtained in a case where the porous term is not relevant [4]. Position of numerical isotherms is in good agreement with front recoil measurements performed on pure succinonitrile (SCN). This result

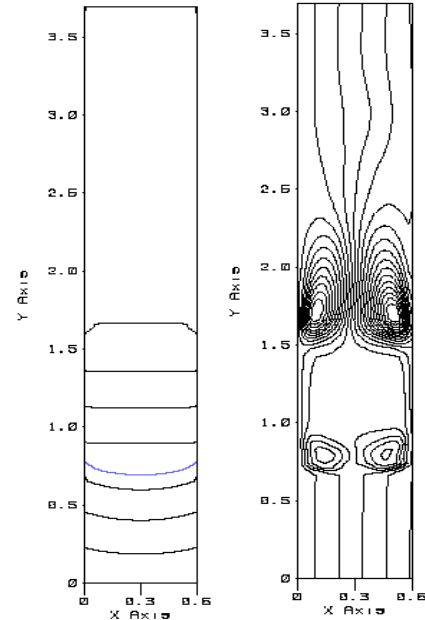


Fig. 2 : Isotherm and streamline plots obtained with SNC for pulling velocity $V_t = 10 \mu\text{m s}^{-1}$, and thermal gradient $G \approx 30 \text{ K/cm}$. Interface position around $y=0.75$ corresponds to the gray line [4].

gives information on the contribution of thermal convection to this recoil. However, the authors point out discrepancy between purely diffusive model and experimental data. Finally, it appears that

comparative studies with and without convection are required to clarify and quantify convection influence, therefore underlining the great interest of microgravity (DECLIC spatial project) for solidification studies.

Phase field model for microstructures.

To explain microstructures in constrained growth, and recently in eutectic colony formation [13], Plapp and Karma have studied the problem first analytically [19], and then numerically, using a phase field model [20]. They study a ternary alloy close to a binary eutectic which, for simplicity, they assume to have a symmetric phase diagram. They introduce 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 vary continuously across the smooth, diffuse interface. They start from a dimensionless Helmholtz free energy functional, and they deduce, by minimization, three local equations between three variables, which are valid everywhere under appropriate assumptions:

$$\tau \frac{\partial \phi}{\partial t} = W_\phi \bar{\nabla}^2 \phi + \frac{\phi}{2} - \frac{\phi^3}{2} + h(\phi) (f_{\text{liq}} - f_{\text{sol}}) \quad (11)$$

$$\frac{\partial u}{\partial t} = \bar{\nabla} \cdot \left[M \left(\frac{\partial^2 f_{\text{liq}}}{\partial u^2} \bar{\nabla} u + W_u \bar{\nabla} (\bar{\nabla}^2 u) \right) \right] \quad (12)$$

$$\frac{\partial \tilde{C}}{\partial t} = \bar{\nabla} \cdot \left(\tilde{M} \frac{\partial^2 f_{\text{liq}}}{\partial \tilde{C}^2} \bar{\nabla} \tilde{C} \right) \quad (13)$$

In these equations, u is a 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, $f_{\text{liq}}(u, \tilde{C}, T)$ and $f_{\text{sol}}(u, \tilde{C}, T)$ are the free energy densities for liquid and solid, respectively, W_u and

W_ϕ are characteristic thicknesses of the solid-solid and of the solid-liquid interface, respectively, and $M(\phi, u, \tilde{C})$ and $\tilde{M}(\phi, u, \tilde{C})$ are the mobility functions of the eutectic component and the ternary eutectic, respectively.

Numerical simulations which have been performed with this phase field model, have enabled the authors to carry out large-scale simulations of colony formation starting from arrays of up to 40 lamella pairs. They found a good overall agreement with the theoretical predictions of linear analysis [19], which

predicts a destabilization front by long-wavelength modes that may be stationary or oscillatory. But they find that the growth of the lamellae is not exactly normal to the large scale envelope of the composite interface, a rule used in the linear stability analysis [19].

The phase field model must be extended to 2D interfaces in 3D configuration [21]. Akamatsu et al. [22] compare 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 solidification front presents a curvature because the isotherms are curved for well-known technical reasons. This curvature is of course influenced by gravity but it exists also in microgravity. Thermosolutal convection will also lead to modifications in local dynamics of eutectic fronts and can also cause a macro-segregation. Other specific phenomena are pointed out for 2D fronts: varying orientations of lamellae between eutectic grains, global slow rotation of the microstructure around z axis, presence of "faults". The authors propose to compare benchmark space experiments to ground ones, and also to study the intermediate case of semi-massive samples.

Second gradient.

Some fluid-fluid interfaces have been studied by second gradient methods [23, 24]. In second gradient model, internal energy by unit mass depends not only on the entropy and the density but also on the magnitude of the density gradient [25]

$$e = e(s, \rho, |\bar{\nabla} \rho|^2) \quad (14)$$

The associated Gibbs relation is

$$de = T ds - p d(1/\rho) + (\lambda/\rho) \bar{\nabla} \rho d(\bar{\nabla} \rho) \quad (15)$$

where λ is a coefficient called the "capillarity coefficient". Using average quantities, and introducing the volumetric 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 [25, 26].

OTHER RELEVANT PROBLEMS.

To complement previous sections and conclude this paper, some solidification problems relevant for microgravity experiments, and comparison with theoretical and numerical results are quoted.

Melting and thermal stabilization stages preparatory to directional solidification greatly affect the initial state on which growth is started [27], and then directional solidification [13]. It is pointed out that free melting is not the mere reversal of free crystal growth. Two phenomena have been observed in situ and in real time during 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. Benielli et al. [27] analyze the dynamics of melting morphology at the solid-liquid interface. The criterion for morphological instability in directional melting reads

$$V > V_{MI} = (kD_s + D_l)G / [mC_0(k-1)] \quad (16)$$

with V_{MI} the critical melting velocity, C_0 the initial solute concentration. The authors emphasize the interest of carrying out experiments in the limit of diffusive transport of solute in the liquid, that imposes to work in the absence of gravity. They participate to DECLIC project of CNES.

Berne, Pasturel & Vinet [28] study crystalline nucleation and phase selection in refractory metals and alloys in the 50-meters high Grenoble drop-tube of CEA, they emphasize the interest of microgravity and ultra-vacuum to ensure absolute no-contact conditions and purification by evaporation of really spherical droplets.

Camel & Dupouy [29] study 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.

Nguyen Thi et al. [30] stress the importance of microstructures for the resulting properties of 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 preparatory phases of melting and stabilization, and the initial directional-solidification transient, so that the measured concentration variation in solidified samples in the AGHF (Advance Gradient Heating Facility) can be explained.

The problem of solidification front curvature is very important and experiments will be performed on transparent material for a better knowledge of this phenomena in DECLIC instrument.

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 [31].

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, for instance [32].

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NOMENCLATURE

Gr Grashov number

Pr Prandtl number

Ra Rayleigh number

Re Reynolds number

Sc Schmidt number

c specific heat of the material

C solute concentration, average mass fraction

\tilde{C} concentration of impurities

D mass diffusivity

e internal energy per unit mass

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

p pressure

s entropy per unit mass

t time

T, T_F mixture temperature, melting temperature,

u x velocity component, scaled concentration

\bar{U} local fluid velocity

v z velocity components

V, V_t melting velocity, pulling velocity

\vec{v} local velocity

W characteristic thickness

x horizontal coordinate

y, z vertical coordinate

β_T thermal expansion coefficient of the liquid

Δ jump

λ thermal diffusivity, capillarity coefficient

μ, ν dynamical viscosity, kinematical viscosity

ρ density

ϕ phase field

Subscripts

E eutectic

F melting

l, s, sl liquid, solid, transition zone

α, β solid phases in a binary eutectic

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