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WALDEMAR WOŁCZYŃSKI *, MANUEL BOBADILLA **,
ANDRZEJ DYTKOWICZ ***

SEGREGATION PARAMETERS FOR CELLS OR COLUMNAR DENDRITES OF ALLOYS WITH δ/γ TRANSFORMATION DURING SOLIDIFICATION

PARAMETRY SEGREGACJI DLA KOMÓREK LUB DENDRYTÓW KOLUMNOWYCH W STOPACH Z TRANSFORMACJĄ δ/γ PODCZAS KRYSTALIZACJI

An application of the equations governing the microsegregation phenomenon with back-diffusion has been presented and referred to the competitive growth of the δ and γ phases during peritectic reaction. It allows for independent description of the solute microsegregation and redistribution within both analysed phases. The mode of calculation of both microsegregation and redistribution after back-diffusion has been suggested when non equilibrium partition ratio is taken into account. It requires to calculate the displacement of the liquidus line and to recalculate the value of non equilibrium partition ratio, which should be used in theoretical predictions of parameters associated with microsegregation and redistribution.

Podano zastosowanie równań opisujących zjawisko mikrosegregacji z dyfuzją wsteczną w odniesieniu do konkurencyjnego wzrostu faz δ i γ podczas przemiany perytektycznej. Pozwoliło to na stworzenie niezależnych opisów mikrosegregacji i redystrybucji składnika stopowego w obydwu analizowanych fazach. Zasugerowano modyfikację metody obliczeń mikrosegregacji i redystrybucji przy uwzględnieniu nierównowagowego współczynnika rozdziału. Wymaga to określenia przesunięcia linii likwidus i wyznaczenia wartości liczbowej nierównowagowego współczynnika rozdziału, który powinien być następnie użyty w teoretycznych przewidywaniach parametrów związanych z mikrosegregacją i redystrybucją.

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* INSTYTUT METALURGII I INŻYNIERII MATERIAŁOWEJ PAN, 30-059 KRAKÓW, UL. REYMONTA 25

** INSTITUT DE RECHERCHE DE LA SIDERURGIE FRANCAISE, 57214 MAIZIERES-LES-METZ, VOIE ROMAINE,

*** POLITECHNIKA ŚLĄSKA, KATEDRA NAUKI O MATERIAŁACH, 40019 KATOWICE, UL. KRASIŃSKIEGO 8.

NOTATION

D_L — coefficient of diffusion in the liquid, [m^2/s],
D_S — coefficient of diffusion in the solid, [m^2/s],
G — temperature gradient at the solid/liquid interface, [K/m],
G_C^j — solute concentration gradient at the solid/liquid interface for δ or γ — phase, [mole fr./m],
H — height of columnar dendrite or cell, [m],
k — equilibrium partition coefficient, [mole fr./mole fr.],
L — half the dendrite/cell spacing of ideally organised hexagonal dendrite/cell array, [m],
m_j — slope of the liquidus line for the j-phase, $j = \delta, \gamma$, [K/mole fr.],
N — solute concentration, [mole fr.],
N_0 — nominal concentration of solute in a given alloy, [mole fr.],
$N_L(x; \alpha)$ — solute concentration in the liquid, [mole fr.],
$N_S(x; \alpha)$ — solute concentration at solid/liquid interface, [mole fr.],
$\bar{N}_S(x; \alpha)$ — average solute concentration within the solid, [mole fr.],
$N_S^\beta(x; x_0, \alpha)$ — solute redistribution within the solid, [mole fr.],
R_j — dendrite/cell tip radius of the j-phase, $j = \delta, \gamma$,
t_f — local solidification time, [s],
T — temperature, [K],
v — growth rate in the *Bridgman* system, [m/s],
v_p — rate of thickening, [m/s],
x — amount of growing crystal, [mole],
x_0 — amount of crystal at a given stage of solidification, [mole],
$x_K(\alpha, N_0)$ — total amount of crystal at the end of solidification, just before the start of precipitation process, [mole],
$x_1(\alpha, N_0, N_1)$ — amount of primary δ-phase, just before the peritectic reaction, [mole],
$x_1^{\min}$ — amount of primary crystal, δ just after peritectic reaction, [mole],
$x_1^{\max}$ — amount of both primary crystal, δ, and γ — phase resulting from peritectic reaction, [mole],
α — parameter of back-diffusion into the solid across s/l interface,
β_1 — coefficient of extent of solute redistribution within the solid,
β_2 — coefficient of intensity of solute redistribution within the solid,
Γ_j — capillary parameters of the solid/liquid interface of the j-phase, $j = \delta, \gamma$, [K m],
λ_S^j — marginal stability wavelength at the solid/liquid interface of the j-phase, $j = \delta, \gamma$, [m],
Ω — solute supersaturation.

1. Introduction

Some measurements of solute redistribution within the dendrites of low carbon steel prove that back-diffusion phenomenon takes place, [1]. It is especially important in the steels for which the competitive growth δ/γ is observed. In binary peritectic alloys with a composition in the range of the peritectic plateau two phases can form as the primary phases directly from the melt: stable δ and metastable γ, [2]. Both phases form dendrites and compete with other. It is evident that with increasing growth rate rather the γ — metastable phase forms than the δ-phase. The subject of competitive growth of both considered phases is of great importance, because precise control of the contribution of both phases in a produced material is required from the viewpoint of the final properties of the product.

Fredriksson [3] analysed the segregation phenomenon of iron-base alloys with respect to the primary phase selection that is δ — ferrite or γ — austenite. Fredriksson [3] was also able to explain the transition from table δ-dendrites to metastable γ-dendrites due to peritectic reaction in Fe–Ni alloys with increasing growth rate. Bobadilla, Lacaze and Lesoult [4] applied the above approach to stainless steels growth and developed the theory shown by Kurz and Fisher [5] to ternary alloy solidification. Rappaz, David, Vitek and Boatner [6] extended the model for multiconstituent alloys given by Bobadilla, Lacaze and Lesoult [4] to the region of high solidification rates.

Rapid solidification, for example strip casting or planar flow casting has gained much interest. Such a process leads to solidification rates equal to several cm/s. The diffusion within solid/liquid system as well as accompanying non-equilibrium phenomena become important at the above solidification rates. A model for dendrites growth which includes high rate phenomena has been developed by Kurz, Giovanola and Trivedi [7] and Trivedi and Kurz [8].

According to analysis performed by Trivedi and Kurz [8] two types of peritectic phase diagrams can be selected:

1. the first is typical for steels, where the control of the transition from δ to γ — phase is important for the quality of product,
2. the second is typical for rare earth permanent magnet alloys or superconductors.

In the continuous casting of carbon steels the quality of surface and heat flux to the mold strongly depend on the phase which forms at first, [9]. In the welding or strip casting of the 18/8 Cr/Ni steel the cracking can be avoided or strongly reduced when the ratio of amounts of both phases δ/γ is high enough, [10]. In the ingot casting of the Nd-Fe-B permanent magnets the preferred alloy compositions are close to the metastable peritectic γ, that is $Nd_2Fe_{14}B$ and the stable primary phase δ. It means that Fe — phase should be eliminated, [2]. In directional growth of the Y-Ba-Cu oxide superconductors the metastable 123 γ — phase ($YBa_2Cu_3O_7$), should form rather than the stable 211 phase δ, [2].

In order to control the appearance of δ or γ phase the segregation parameters should be described in function of solidification rate. It is evident that at low solidification rates the stable phases are preferred by the solid/liquid system. At high rates the smaller solute rejection of the metastable phase leads to the lower interface undercooling, in comparison with the stable phase undercooling. This promotes the appearance of metastable γ — phase which is thermodynamically justified. The transition from δ to γ — phase occurs at certain critical growth rate. The above competition is also observed in other situations. For example, it can be controlled in the case of transformation of binary $TiAl_3$ compound into ternary $Ti(Al, Zn)_3$ compound during inoculation in the Zn-25 Al alloy modified by Al-Ti master alloy, [11], [12] adequately to the rate of heat transfer within the solid-liquid system.

Moreover, interaction between both competitive phases δ and γ is the mutual pheonomenon, which occurs during mechanical and thermal treatments, as shown by Ratuszek, Ryś and Chruściel, [13] for the duplex steel. This interaction can improve the material properties due to recrystallization and precipitation processes, [13].

The subject of the present paper is to show the behaviour of some parameters describing the segregation phenomenon in steel — type peritectic diagrams. It is not only important to control the competition between δ and γ phase appearance but the solute redistribution within the dendrites and the amount of precipitates as well. This is because the phenomena of segregation influence the quality of a given alloy. Especially, magnetic properties are essentially dependent on solute redistribution within crystal, [14-16].

The microsegregation is directly connected to solute content and solidification rate as studied by Shin, Suzuki and Umeda [17] in the case of carbon steel with δ/γ transformation. The solidification of steel is usually accompanying by inclusions and precipitates. Both above phenomena are associated with segregation, [18–19]. The competitive growth and solute segregation as well as solute redistribution are especially important in the case of columnar dendrite formation of duplex steels in which δ/γ transformation is always observed, [20], [21]. Some theoretical treatments involving the study of competitive phase undercooling and application of the marginal stability criterion to the columnar dendrite growth of steels are also known, [22–25]. The mentioned solutions will be introduced into the proposed model of segregation.

2. Equations governing the solute microsegregation and redistribution

Recently, a new equation for microsegregation has been formulated, [26]

$$d\big(x\bar{N}_S(x;\alpha)\big) = N_S(x;\alpha)dx + \frac{D_S}{Lv_p}x\frac{dN_S(x;\alpha)}{dx}dx. \tag{1}$$

The l.h.s. of equation (1) presents the amount of solute, which entered inside the crystal due to back-diffusion, when the newly growing dx — layer appeared; the first term of r.h.s. of equation (1) presents the amount of solute, which is actually within this layer, (solid/liquid interface); the second term of r.h.s. of equation (1) presents the amount of solute which passed across the solid/liquid interface and entered interior of the crystal.

The definition of back-diffusion parameter α, known in Brody and Flemings [17] theory can be separated from equation (1).

$$\alpha = \frac{D_S t_f}{L^2}, \tag{2}$$

Finally, eqn (1) is integrated:

$$N_L(x;\alpha) = N_0(1+\alpha kx - x)^{\frac{k-1}{1-\alpha k}}, \qquad x \in [0, x_K], \qquad N_L(0;\alpha) = N_0. \tag{3}$$

The equation (3) is dealing with microsegregation controlled by back-diffusion and can described the solute content in consequently appearing layers (s/l interfaces) as follows:

$$N_S(x;\alpha) = kN_0(1+\alpha kx - x)^{\frac{k-1}{1-\alpha k}}, \qquad x \in [0, x_K]. \tag{4}$$

The solute redistribution is strongly influenced by back-diffusion and can be defined by the following equation, in which microsegregation is taken into account:

$$N_S^{\beta}(x;x_0,\alpha) = N_S(x;\alpha) + \beta_1(x;x_0)\beta_2(x_0;\alpha)N_L(x;\alpha), \qquad x \in [0, x_0], \qquad x_0 \in [0; x_K]. \tag{5}$$

The coefficient of the extent of the solute redistribution, β_1 and the coefficient of the intensity of the solute redistribution, β_2 are also precisely determined. Both coefficients are defined in the following way:

$$\beta_1(x;x_0) = \frac{k(1-k)(x_0-x)}{1+kx_0-x_0} \tag{6}$$

and

$$\beta_2(x_0,\alpha) = \frac{\alpha(1+k-2\alpha k)(1+kx_0-x_0)\left[(1+kx_0-x_0)(1+\alpha kx_0)(1+\alpha kx_0-x_0)^{\frac{k-1}{1-\alpha k}}-1\right]}{(1-k)\left[kx_0(1-\alpha)+(1+\alpha kx_0-x_0)\left((1+\alpha kx_0-x_0)^{\frac{k-\alpha k}{1-\alpha k}}-1\right)\right]}. \tag{7}$$

3. Back-diffusion phenomenon referred to experiment of the oriented growth

The definitions for solute microsegregation and redistribution are based on the scheme of cell/dendrite geometry, which allows some morphological parameters to be defined, Fig. 1. Taking into account that the L — parameter has been defined in the Fig. 1 as one half the intercellular/dendritic spacing, which was suggested by Umeda, Matsuyama, Murayama and Sugiyama [28], the definition of α-parameter, equation (2) can be expressed by oriented growth rate v. The modified definition is as follows:

$$\alpha = \frac{D_S H}{L^2 v}. \tag{8}$$

The above model takes into account the constant partition ratio, which is equilibrium coefficient given by equilibrium phase diagram. However, in the case of more rigorous calculations the undercooling is to be also taken into account. It results in a replacing of the equilibrium partition ratio by the non-equilibrium partition ratio coming from the displacement of liquidus line caused by mentioned undercooling.

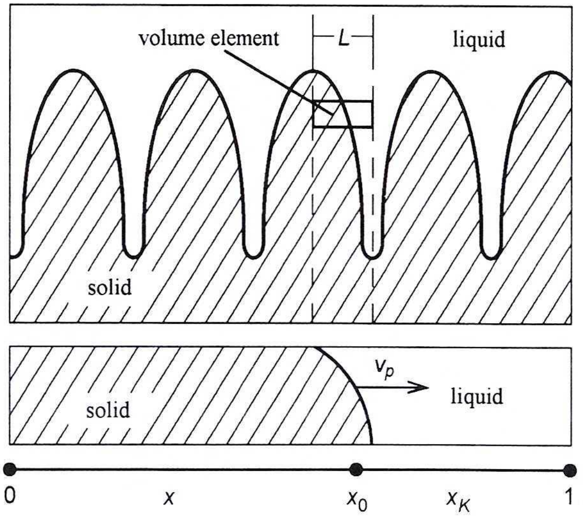

Fig. 1. Schematic presentation of the geometry of columnar dendrite formation

4. Competitive growth of the δ and γ phases

The competitive growth can be easily observed in laboratory experiment under conditions of directional solidification. At first appears the phase δ or γ according to the criterion of higher temperature of the tip of columnar dendrite, [4]. This temperature depends on growth rate, which influences also the back-diffusion parameter, equation (8). It is evident that the difference between undercooling of the tips of both phases δ and γ depends significantly on the growth rate. This difference changes its algebraic sign at a critical growth rate for δ/γ competition. Therefore, the undercooling for both phases have to be determined independently in function of growth rate. In order to obtain the definition of undercooling, a model for columnar dendrites growth should be derived, [29]. The competition between δ and γ phases is strongly influenced by the addition of third elements, as discussed in details by Ueshima, Mizoguchi, Matsumiya and Kaijoka, [30] and Sugiyama, Umeda and Matsuyama [31]. Moreover, solute microsegregation as well as redistribution are also influenced not only by the addition of third element but by the partition ratio as it can be deduced from equations (4) – (7). Generally, there are some segregation parameters which make difference in solute redistribution:

$$k_\delta \neq k_\gamma, \qquad D_S^\delta \neq D_S^\gamma. \tag{9}$$

In consequence, for the δ and γ phases formation from the liquid

$$\alpha_\delta \neq \alpha_\gamma, \quad \beta_1^\delta \neq \beta_1^\gamma, \quad \beta_1^\delta \neq \beta_2^\gamma. \tag{10}$$

So, it is evident that two independent equations governing the solute microsegregation as well as redistribution have to be written:

a) for δ-phase formation from the liquid

$$N_L^\delta(x;\alpha) = N_0(1+\alpha k_\delta x - x)^{\frac{k_\delta - 1}{1-\alpha k_\delta}}, \quad x \in [0, x_1], \quad N_L^\delta(0;\alpha) = N_0 \tag{11}$$

b) for γ-phase formation from the liquid

$$N_L^\gamma(x;\alpha) = N_1(1+\alpha k_\gamma x - x)^{\frac{k_\gamma - 1}{1-\alpha k_\gamma}}, \quad x \in |x_1^{\max}, x_K|, \; N_L^\gamma(x_1^{\max};\alpha) = N_1 \tag{12}$$

where N_1 is defined in Fig. 2.

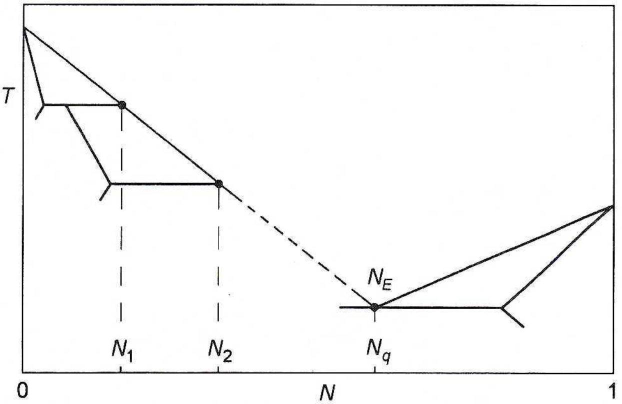

Fig. 2. Scheme of multipertitectic-eutectic phase diagram

c) for the solute concentration at the δ-solid/liquid interface

$$N_\delta^L(x;\alpha) = k_\delta N_0(1+\alpha k_\delta x - x)^{\frac{k_\delta - 1}{1-\alpha k_\delta}}, \quad x \in [0, x_1] \tag{13}$$

d) for the solute concentration at the γ-solid/liquid interface

$$N_\gamma^L(x;\alpha) = k_\gamma N_1(1+\alpha k_\gamma x - x)^{\frac{k_\gamma - 1}{1-\alpha k_\gamma}}, \quad x \in |x_1^{\max}, x_K| \tag{14}$$

e) for the solute redistribution in the primary δ-phase

$$N_\delta^\beta(x;x_0,\alpha) = N_\delta^L(x;\alpha) + \beta_1(x;x_0)\beta_2(x_0;\alpha)N_L^\delta(x;\alpha), \quad x \in [0, x_0], \quad x_0 \in [0, x_1] \tag{15}$$

f) for the solute redistribution in the primary γ-phase

$$N_{\gamma}^{\beta}(x;x_0,\alpha)=N_{\gamma}^{L}(x;\alpha)+\beta_1(x;x_0)\beta_2(x_0;\alpha)N_{L}^{\gamma}(x;\alpha),\quad x\in[0,x_0],\quad x_0\in|x_1^{\max},x_K|. \tag{16}$$

However, the competition between the primary δ-phase and γ-phase from the peritectic reaction is observed. The occurrence of the peritectic reaction is preceded by the formation of primary δ-phase, Fig. 3. Some part of primary δ-phase is disappearing. The amount of disappeared primary δ-phase $\Delta\delta$ is equal to:

$$\Delta\delta(\alpha,N_0,N_1)=x_1(\alpha,N_0,N_1)-x_1^{\min}(\alpha,N_0,N_1). \tag{17}$$

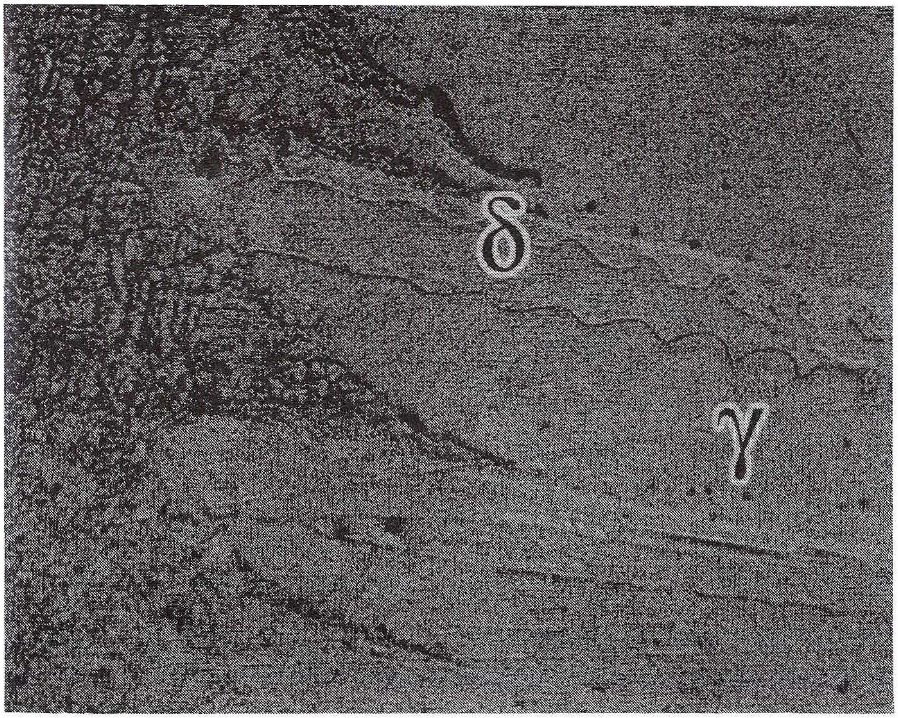

Fig. 3. Competitive growth of δ-phase and γ-peritectic phase, [36]

The amount of peritectic γ-phase $\Delta\gamma$ is given by

$$\Delta\gamma(\alpha,N_0,N_1)=x_1^{\max}(\alpha,N_0,N_1)-x_1^{\min}(\alpha,N_0,N_1). \tag{18}$$

It is evident that

a) for $\alpha=0$

$$x_1(\alpha,N_0,N_1)-x_1^{\min}(\alpha,N_0,N_1)=0 \tag{19}$$

b) for $\alpha=1$

$x_1(\alpha, N_0, N_1) - x_1^{\min}(\alpha, N_0, N_1)$, equation (19), is the maximum value from among all possible values of this difference. Notice that the solute concentration within the appearing peritectic γ-phase due to peritectic reaction is treated as equal to $k_\gamma N_1$.

5. Non equilibrium parameters for the growth of δ and γ phases

In the case of more rigorous calculation the segregation parameters should be calculated taking into account undercooling of the solid/liquid interface. It results in a translation of the liquidus line. Finally, the equilibrium partition ratio, k, can be substituted by non equilibrium partition ratio, k_T^j, $(j = \delta,\ \gamma)$. However, the solid/liquid interface undercooling must be known. The following mode of calculation is proposed to determine the undercooling. At the beginning it can be written that for cells or columnar dendrites growth the following equation is valid:

$$\Omega = I(L_P), \tag{20}$$

where $I(L_P)$ is defined by the equation given by Ivantsov [32]:

$$I(L_P) = L_P \exp(L_P) E_1(L_P), \tag{21}$$

where

$$L_P = \frac{v R_j}{2 D_L}. \tag{22}$$

Some measurements made for the low carbon steel, [33], proved that the following approximation can be applied in this description according to the analysis shown by Kurz and Fisher [34].

$$\Omega = I_2(L_P). \tag{23}$$

Next, applying the marginal stability criterion developed by Langer and Muller-Krumbhaar [35]

$$R_j = \lambda_S^j, \quad (j = \delta, \gamma) \tag{24}$$

the final equation for the tip radius, R_j, $(j = \delta, \gamma)$ can be reached, [29]. It is as follows

$$R_\delta = 2\pi \left(\frac{\Gamma_\delta}{m_\delta G_C^\delta - G} \right)^{0.5}. \tag{25}$$

Analogously

$$R_\gamma = 2\pi \left(\frac{\Gamma_\gamma}{m_\gamma G_C^\gamma - G} \right)^{0.5}. \tag{26}$$

Finally, the undercooling of the solid/liquid interface can be determined. The presented definitions contain capillary parameters connected with the interface curvature.

a) for δ-phase formation

$$\Delta T_\delta = m_\delta\left(N^L_\delta(x,\alpha) - N_0\right) - \frac{2\Gamma_\delta}{R_\delta} \tag{27}$$

b) for γ-phase formation

$$\Delta T_\gamma = m_\gamma\left(N^L_\gamma(x,\alpha) - N_1\right) - \frac{2\Gamma_\gamma}{R_\gamma}. \tag{28}$$

The calculated undercooling allows for determination of the displacement of liquidus line and finally to calculate the effective non equilibrium partition ratios, k^δ_T and k^γ_T, in equations (11)–(16) instead of equilibrium partition ratio, k_δ and k_γ, respectively.

6. Concluding remarks

The substitution of equations (20)–(26) into (27) and (28) allows to express them in terms of growth rate, which is contained in the definition of the *Peclet* Number, L_P, equation (22). It is as follows,

a) for the δ-phase formation

$$\Delta T_\delta = m_\delta N_0\left[1 - \frac{R_\delta v + D_L}{R_\delta v + D_L - (1-k_\delta)R_\delta v}\right] - 2\frac{\Gamma_\delta}{R_\delta} \tag{29}$$

b) for the γ-phase formation

$$\Delta T_\gamma = m_\gamma N_1\left[1 - \frac{R_\gamma v + D_L}{R_\gamma v + D_L - (1-k_\gamma)R_\gamma v}\right] - 2\frac{\Gamma_\gamma}{R_\gamma}. \tag{30}$$

According to equations (29) and (30) the displacement of the liquidus line and finally the calculation of the non equilibrium partition ratios can be made in terms of crystal growth rate, v, which is imposed for a given experiment carried out in the *Bridgman* system.

The displacement of the liquidus line can be deduced from equations (29) and (30) and a given phase diagram:

a) for the δ-phase formation

$$\Delta N_\delta = \frac{\Delta T_\delta}{m_\delta} \tag{31}$$

a) for the γ-phase formation

$$\Delta N_\gamma = \frac{\Delta T_\gamma}{m_\gamma}. \tag{32}$$

Finally, the definitions for the non equilibrium partition ratios are:

a) for the δ-phase formation

$$k_T^\delta = \frac{N_0 - \Delta N_\delta}{k_\delta N_0} \tag{33}$$

b) for the γ-phase formation

$$k_T^\gamma = \frac{N_1 - \Delta N_\gamma}{k_\gamma N_1}. \tag{34}$$

Both definitions (33) and (34) should be used in calculation of segregation parameters instead of the equilibrium partition ratio, k_j, $(j = \delta, \gamma)$, as suggested above.

It should be emphasised that the competitive growth of both δ and γ phases can take place in two situation:

a) between primary δ-phase and primary γ-phase,

b) between primary δ-phase and peritectic γ-phase.

However, in the second situation the appearance of the peritectic γ-phase is accompanying by the partial disappearance of the primary δ-phase. The system chooses one from above mechanisms and it depends on temperature of cell/dendrite tip. The higher is temperature of the tip of a given phase the first it appears.

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