## EFFECT OF CONCENTRATION DEPLETION ON KINETICS OF INTERMEDIATE PHASE FORMATION IN THE SUPERSATURATED ALLOY

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Abstract. The influence of the number of heterogeneous nucleation sites in the parent phase and of the thermodynamic parameters on nonsteady separation kinetics in the supersaturated binary solid solution is considered in details. The conservation of matter in the parent phase, which leads to constraints on nucleation and growth of new phase particle, is taken into account. Computer simulations show that depending on the number of heterogeneous nucleation sites there exist different number of evolution stages of separation.

#### I. Introduction

When a system is quenched into the two-phase region, separation proceeds via nucleation and growth of the new phase nuclei. Modeling of these first order phase transformations is important for understanding the processes in real systems. In experiments one can distinguish four subsequent stages of first order phase transition: nucleation, independent growth stage, crossover (or so-called intermediate) stage, coarsening (or Ostwald ripening) [1-3]. Recently the problems of nucleation and growth of precipitates were solved analytically as well as numerically [4-6]. Nevertheless, the dynamics of nucleation and crossover phenomenon is not yet well understood.

The aim of present paper is to study (by computer simulation methods) the influence of number of defects in the initially supersaturated alloy on separation kinetics.

It is known that there are two kinds of nucleation mechanism: heterogeneous and homogeneous. In this work we consider only first one, when the nucleation takes place on heterogeneous nucleation sites. We suppose that at the beginning all droplets of a new phase are overcritical because of rapid quenching and interfacial defects (say, due to "dirt").

The principal supposition has been made in [8-10] by considering the effective "supply region" which leads to Gibbs free energy of a system as a function of concentrations in depleted parent phase as well as in new phase particle. This paper continues solving the problem of unsteady-state nucleation kinetics with taking into account the time dependent depletion. The problem (master equation) is self-consistent: Gibbs free energy of a system is a function of concentration, concentration in its turn is a functional of the size distribution function and kinetics of the size distribution function is determined by Gibbs free energy dependence on concentration.

#### II. Method and model

We consider a supersaturated binary alloy consisting of atoms of two sorts: *A* and *B*, so that the total number of atoms  $N_{tot}=N_{A,tot}+N_{B,tot}$ , where  $N_{A,tot}$  and  $N_{B,tot}$  are the numbers of atoms A and B respectively in a system. We will use next assumptions: 1) parent phase is being depleted during the nucleation and growth of a new phase particle; 2) new phase is a "linear" intermetallic (strict stoichiometric composition AB) with concentration  $C_1=0.5$  of elements *B*; 3) parent phase is an ideal solid solution with initial concentration  $C_0$  less than  $C_1$ ; 4) clusters of a new phase are spherical; 5) concentration in the parent phase is the same throughout the volume and determined by the conservation law; 6) homogeneous nucleation does not take place; 7) heterogeneous nucleation sites arrange approximately symmetrically in space and total number of heterogeneous nucleation sites is constant in time.

Introduce the cluster size distribution function  $f(\tilde{N}, t)$ . It denotes the number of droplets of the new phase consisting of  $\tilde{N}$  structure units AB at moment *t*. The kinetics of clusters population will be described by "master equation":

$$\frac{\partial f(\widetilde{N},t)}{\partial t} = f(\widetilde{N}-1,t) \cdot \nu_{+}(\widetilde{N}-1,t) + f(\widetilde{N}+1,t) \cdot \nu_{-}(\widetilde{N}+1,t) - f(\widetilde{N},t) \cdot \nu_{+}(\widetilde{N},t) - f(\widetilde{N},t) \cdot \nu_{-}(\widetilde{N},t)$$

Here  $v_+(\tilde{N}, t)$  and  $v_-(\tilde{N}, t)$  - time dependent frequencies of attachment and detachment of monomer *AB* to a cluster of size  $\tilde{N}$ , t - time.

In our case  $C_0 < 0.5$ ,  $C_1 > C_0$  and  $C_1 = 0.5$ , so the number of building units  $\widetilde{N}$  in each nucleus coincides with the number of atoms *B* type  $\widetilde{N} = N_B$ . Under these assumptions the process will be controlled by *B* component mobility (depletion influence in a matrix). It yields:  $f(\widetilde{N}, t) \equiv f(N_B, t)$ ,  $v_{\pm}(\widetilde{N}, t) \equiv v_{\pm}(N_B, t)$  and for rate equation:

$$\frac{\partial f(N_B,t)}{\partial t} = f(N_B - 1, t) \cdot \nu_+(N_B - 1, t) + f(N_B + 1, t) \cdot \nu_-(N_B + 1, t) - f(N_B, t) \cdot \nu_+(N_B, t) - f(N_B, t) \cdot \nu_-(N_B, t)$$
(1)

The ratio between  $v_{(N_B,t)}$  and  $v_{(N_B,t)}$ .

$$\nu(N_B, t) = \nu_+(N_B, t) \cdot \exp\left(\frac{\Delta G(\widetilde{N}) - \Delta G(\widetilde{N} - 1)}{kT}\right)$$

where  $\Delta G(\tilde{N}) = \Delta G(N_B \mathcal{L}) = \Delta G(N \mathcal{L})$  - Gibbs free energy of forming a nucleus of size  $\tilde{N}$  consisting of  $N = N_B/C_I$  atoms, where the mean mole fraction in the depleted parent phase is equal to C. The difference  $\Delta G(\tilde{N}) - \Delta G(\tilde{N} - 1)$  is the change of the Gibbs free energy of a system when one monomer (AB) attaches to the new phase cluster and depletes the matrix.

The average mole fraction C (further concentration) in depleted parent phase during the process is a function of time and functional of size distribution function:

$$C = \frac{N_{tot} C_o - \sum_{N_B = N_o}^{\infty} N_B f(N_B, t)}{N_{tot} - \frac{1}{C_1} \sum_{N_B = N_o}^{\infty} N_B f(N_B, t)},$$

where  $N_{to}$  - total number of atoms in alloy,  $N_o$  - minimal number of *B* atoms on one heterogeneous nucleation site,  $C_o$  - initial concentration of B atoms (in mole fraction).

The density of energy (per one atom) of new linear phase is an isothermal Gibbs free energy of formation from pure solid components:

$$\Delta g_n(T,C) = \Delta g_1 + \alpha kT, (C = C_1).$$

Accordingly for the supersaturated parent phase we will use the Gibbs energy of mixing per atom in such form:

$$\Delta g_o(T,C) = kT \left( C \cdot Ln(C) + (1-C) \cdot Ln(1-C) \right).$$
<sup>(5)</sup>

Here k - Boltzmann constant, T - absolute temperature,  $\Delta g_1$  and  $\alpha > 0$  - new phase parameters, which determine the temperature dependence of bulk driving force. For thermodynamically advantageous new phase:  $\Delta g_n(T, C_1) < \Delta g_o(T, C_o) < 0$ .

An expression  $\Delta G(N_B + 1) - \Delta G(N_B)$  in equation (2) for the case  $d\tilde{N} = dN \cdot C_1 = 1$  may be found by formula [11]:

$$\Delta G(N_{B}+1) - \Delta G(N_{B}) = \frac{\Delta g_{n}}{C_{1}} - \frac{\Delta g_{o}(C)}{C_{1}} + \frac{C - C_{1}}{C_{1}} \frac{\partial \Delta g_{o}(C)}{\partial C} + 4\pi\sigma \left(\frac{3}{4\pi n C_{1}}\right)^{\frac{2}{3}} \left( (N_{B}+1)^{\frac{2}{3}} - (N_{B})^{\frac{2}{3}} \right)$$

 $f(N_o,\tau) = W - \sum_{N_o=N+1}^{\infty} f(N_B,\tau)$ 

Here  $\sigma$  is the surface energy, *n* is a number of atoms per unit volume (taken the same for both phases),  $\frac{\Delta g_n}{C_1} - \frac{\Delta g_o(C)}{C_1} + \frac{C - C_1}{C_1} \frac{\partial \Delta g_o(C)}{\partial C} = \Delta g_{ef} - \text{effective bulk driving force of transformation,}$   $\Delta g_n = \Delta g_n(T, C), \quad \Delta g_0(C) = \Delta g_0(T, C). \quad \text{Equation (6) formally coincides with first derivative}$   $\partial \Delta G(\tilde{N}, C) / \partial \tilde{N} = \frac{\partial \Delta G(N, C)}{C_1 \partial N}.$ 

When the time of attaching of a monomer to the surface of a cluster is small with respect to the time of transporting of monomer to the cluster then attachment probability per unit time  $v_+(N_B, t)$  will be determined:  $v_+(N_B, t) = K \cdot C \cdot (N_B)^{\frac{1}{3}}$  [5]. Here K – proportionality coefficient. It depends on diffusion coefficient of B atoms and size of a building unit AB [5,12].

Further we use new variable for time:  $\tau = K \cdot t$ .

Numerical calculations of problem (1-6) were performed by assuming the constant total number of nuclei in ensemble (noncontact nucleation and noncontact growth mode, when nonmoving precipitates grow only due to diffusion in solid solution). We receive the boundary condition:

The initial conditions are:

 $f(N_B,0) = \begin{cases} W, & N_B = N_o \\ 0, & N_B > N_o \end{cases}$ That is, nuclei may decrease their sizes until reaching the minimal number of atoms  $N_{\min}=N_o/C_1$ , but can not vanish completely and they can start to grow only from size  $N_{\min}$ . Here W is the number of heterogeneous nucleation sites. According to our supposition, when one nucleation site may nucleate one nucleus, one may interpret the quantity W as a number of new phase particles in ensemble (constant in time).

#### **III. Results and discussion**

Computer simulations have been realized for next set of parameters:  $C_1 = 0.5$ ,  $C_o = 0.3$ , T = 550K,  $\alpha = 2.4$ ,  $\Delta g_1 = -3 \cdot 10^{-20} J$ ,  $\sigma = 0.05 Jm^{-2}$ ,  $N_o = 7$ ,  $N_{tot} = 10^{22}$ ,  $n = 7 \cdot 10^{28} m^{-3}$ . Further we will use the number of atoms per one nucleation site determined by ratio  $N_{tot} / W$  instead of the number of atoms per unit volume (hereby total volume is  $1.4 \cdot 10^{-5} m^3$ ).

Such assumptions and thermodynamic parameters give the opportunity to distinguish all previously mentioned stages of separation.

In present paper we will consider only the influence of defects number W on a process of isothermal annealing. All further analysis may be reformulated in the terms of limiting the initial volume of an alloy. If initial volume of the system will decrease (at fixed W) then it effectively corresponds to the increasing the number of heterogeneous nucleation sites. The results presented in all figures have been obtained for following six cases: case 1 -  $W_1=10^{14}$  (so

(6)

that  $W_1/N_{tot}=10^{-8}$ ; case 2 -  $W_2=10^{18}$  ( $W_2/N_{tot}=10^{-4}$ ); case 3 -  $W_3=5\cdot10^{18}$  ( $W_3/N_{tot}=5\cdot10^{-4}$ ); case 4-  $W_4=10^{19}$  ( $W_4/N_{tot}=10^{-3}$ ); case 5-  $W_5=5\cdot10^{19}$  ( $W_5/N_{tot}=5\cdot10^{-3}$ ); case 6-  $W_6=10^{20}$  ( $W_6/N_{tot}=10^{-2}$ ).

The most important time dependent values for analysis are: average concentration of the parent phase; effective bulk driving force; average number of atoms in a new phase particle; average droplet radius of the new phase particle; critical nucleus size; mean radius of overcritical new phase particle; average radius of the new phase particle; number of atoms corresponding to the maximum and minimum of size distribution function; Guinier radius for comparing with experimental results of small angle neutron scattering.

The detailed analysis of all important characteristics will be discussed elsewhere. Here we will show only some of them in order to see the main results.

Note that we neglect the first nucleation stage (at the beginning all new phase particle are overcritical) due to heterogeneous nucleation and start the process with independent growth stage.

The modeling was performed using Euler algorithm described in details in [11,12]. This algorithm was checked by the Runge-Kutta numerical method which gave the same result.

From numerical analysis we obtained that during the evolution the maximum of the distribution function  $f(N_R, t)$ 

appears very fast and moves to the direction of bigger sizes  $N_B$ . This general behavior of  $f(N_B,t)$  is clear from our assumptions. It means that all initially overcritical nuclei grow independently until supersaturation and driving force of transformation decreases to equilibrium values.

Let us find the equilibrium concentration for problem (1-6) at fixed T. This concentration  $C_{eq}^{\infty}$  corresponds to the separation in the matrix and may be found by parallel tangent rule when depletion of the parent phase is taken into account [8-10]. This rule is a sequence of thermodynamic approach when Gibbs energy is minimized  $\left(\frac{\partial \Delta G(N,C)}{\partial C}\right)_N = 0$ . Assuming that the second equilibrium concentration is known (C<sub>1</sub>=0.5) and using the

equilibrium condition for the case of infinite system this rule gives the common tangent rule [13]:

$$\Delta g_{o}(T, C_{eq}^{\infty}) + \frac{\partial \Delta g_{o}(T, C)}{\partial C} \bigg|_{C_{eq}^{\infty}} \left( C_{1} - C_{eq}^{\infty} \right) = \Delta g_{n}(T, C_{1}) \quad (9)$$

The transcendent equation (9) after some algebra may be rewritten in such form:

$$C_{eq}^{\infty} = 0.5 \left( 1 - \sqrt{1 - 4 \exp\left(2\left(\frac{\Delta g_1}{kT} + \alpha\right)\right)} \right).$$

Hereby the second root appears due to symmetry of the problem with respect to  $C_1 = 0.5$  and corresponds to cases  $C_o > 0.5$ . It yields  $C_{eq}^{\infty} = 0.0041$ . The tendency to equilibrium in mentioned respect is shown in figures 1 and 2.



Fig. 1 Mole fraction of B atoms in the parent phase versus logarithm of time for different number of heterogeneous nucleation sites. The initial concentration  $C_0=0.3$ . Parameters are given in the main text.



Fig. 2 Evolution of effective bulk driving force  $\Delta g_{ef}/kT$  for different number of heterogeneous nucleation sites. The value of  $\Delta g_{ef}/kT$  tends to zero. Crossover horizontal parts of lines 3 and 4 correspond to the intermediate stages.

As one can see from figures 1 and 2 there are four different regions with different time intervals corresponding to

different stages of separation. Furthermore effective driving force  $\Delta g_{ef}$  and mean molar fraction C are correlating quantities.

Obviously, in general the decrease of the Gibbs free energy of the system is possible due to decreasing the total surface energy and increasing total volume of all new phase particles. The competition between these two processes with taking into account of matter conservation law and collective effects (of ensemble of particles) leads to the complex multistage process of separation, in which at each stage the certain way (volumetric or surface) is prevailed.

At a stage of independent growth when the new phase particles are small (and the supersaturation is high) then the bulk effects are dominating.

At last stage of coarsening when supersaturation in alloy tends to equilibrium the efficiency of surface effects became greater and dominating.

In crossover stage the volumetric and surface changes give the equal effects (first of all in supersaturation in the parent phase respect), which in turn lead to very slow changing driving force  $\Delta g_{ef}$ . It is clear seen in cases 3 and 4 in figure 2. The crossover horizontal parts of lines 3 and 4 in figure 2 represent the intermediate stage between diffusional growth and Ostwald ripening. Initial horizontal parts show the independent growth stages.

$$=\sum_{N_B=N_o}^{\infty} R(N_B) \cdot f(N_B, \tau)$$

The average droplet radius  $\langle R \rangle$ 

As one can see the evolution of the value  $\langle R \rangle$  differs in each case, but in general it is approximately constant at intermediate stage and decreases when coarsening starts. The time dependence of mean droplet radius  $\langle R \rangle$  is parabolic for the case 1 of small numbers of heterogeneous nucleation sites in initially supersaturated alloy at independent growth stage.

In standard Lifshits-Slyozov-Wagner theory [13] the contribution to size distribution function by subcritical nuclei is neglected (they disappear very fast) so the mean radius of the new phase particles always increases. In our consideration we take into account all overcritical as well as subcritical nuclei (which don't disapper) and see that the corresponding contribution of the number of subcritical nuclei to size distribution function and mean radius  $\langle R \rangle$  may be not negligible in highly defective finite alloy (cases 3-4 in our consideration). So mean radius  $\langle R \rangle$  may increase as well as decrease at different stages of evolution. When the average droplet radius  $\langle R \rangle$  becomes approximately constant then the intermediate stage starts. One may mention that the coarsening starts when average droplet radius  $\langle R \rangle$  starts to increase. This criterion is not strict for the case of big number of heterogeneous nucleation sites, when there is no intermediate stage.

One needs to mention that the total volume of a new phase particles (or/and average number of atoms  $\langle N \rangle$  in the particles) always increases at all stages of separation. It changes fast at first stage and then very slowly with the approximate rate  $\tau^{0.0056}$  at coarsening.

Another essential quantity is critical nucleus size  $N^*$ . The critical value  $N^* = N_B^* / C_1$  was found as a crossover point, where the first derivative of Gibbs free energy changes the sign in equation (6):  $\partial \Delta G(N, C)/\partial N = 0$ . This time dependent quantity is shown in figure 4.

Existence of the independent growth, crossover and coarsening stages on  $N_B^*$  time dependence are clearly observable. Comparison of  $N_B^*$  with the overall number of atoms in a new phase particle

 $< N_B >= \sum_{N_B=N_o}^{\infty} N_B \cdot f(N_B, \tau) / W$  gives the additional *in situ* information about transformation. This numerical

in situ consideration will be fully discussed in a future work; some of these results have been obtained in [6].

56



Fig. 3 Logarithmic dependencies of average droplet radius of the phase particle  $\langle R \rangle$  on logarithm of time for different number of heterogeneous nucleation sites: line 1 is plotted for small number of defects - case 1. Crossover horizontal parts of lines correspond to the intermediate stages. Asymptotically  $< R > \tau^{0.5}$ for the case 1 where independent growth mode

takes place.  $P = \frac{1}{3} \ln \left( \frac{3}{4\pi n C_{\star}} \right).$ 

Fig. 4 Dependencies of size of the critical nucleus consisting of  $N^* = \frac{N_B^*}{C_1}$  atoms on logarithm of time for different numbers W.

Crossover interval  $5 \le ln(\tau) \le 10$  for 3-6 cases shows the intermediate stages. Asymptotically  $N_B^* \sim \tau^{0.87}$  - for coarsening.

Representation of mean radius  $\langle R_{ocr} \rangle = \frac{\sum_{N_B = N_B^*}^{\infty} R(N_B) \cdot f(N_B, \tau)}{\sum_{N_B = N^*}^{\infty} f(N_B, \tau)}$  of overcritical new phase particles shows

mentioned stages (figure 5). Asymptotic lines have different behaviors:  $\langle R_{ocr} \rangle \sim \tau^{0.51}$  for small number of defects (and independent growth stage) and  $\langle R_{ocr} \rangle \sim \tau^{0.29}$  ( $\tau^{0.29} \approx \tau^{\frac{1}{3}}$ ) for last coarsening stage (or for big number of

defects).

It is conventional to define the coarsening as a stage of transformation when concentration and bulk driving force have reached their equilibrium values. Comparing the figure 5 with figures 1 and 2 one may mention that the coarsening starts ( $ln(\tau) \approx 10$ , C $\approx 0.06$ ) far from equilibrium concentration (in our presentation  $C_{eq}^{\infty} = 0.0041$ ) and zero bulk driving force are reached. This numerical result allows to suppose the understanding of stage of Ostwald ripening by

two following ways (say, A and B):

A. It is a stage when the simultaneous redistribution of matter and growth of precipitates give



Fig. 5 Evolution of mean radius  $\langle R_{ocr} \rangle$  of the overcritical new phase particles for different given W. Cases 1 and 2 are indistinguishable in figure. Parameters are given in the main text.  $P = \frac{1}{3} \ln \left( \frac{3}{4\pi n C_1} \right)$ .

the change of composition and driving force until equilibrium will be reached.

B. Coarsening seems to be consisting of two consequent stages: first, where redistribution of matter between precipitates leads to further changing the composition of parent phase until equilibrium reached and second, where the prolongation of the precipitates growth takes place in a matrix with almost constant by composition matrix due to decreasing the surface energy, when the total volume of the new phase particles is approximately constant.

At present time we don't know what way (single or twostep) takes place. The detailed analysis of this problem will be presented elsewhere.

If one increases the number of nucleation centers W more  $(W_7>W>W_6, W_7=3.744\cdot10^{20})$  then the intermediate stage disappears: the system undergoes Ostwald ripening without independent growth stage. This fact is clear from comparing the cases 1 and 6 in all figures. The subsequent increasing the number of heterogeneous nucleation sites (at fixed other parameters) consequently gives vanishing the intermediate stage and temporary delay in separation. Hereby the concentration C in the parent phase tends to the equilibrium. An absence of the scatter of the particle sizes at the initial size redistribution function gives the stability of a dispersed system against coarsening. This fact was noted also in [7,12,14].

The greater the W the more close is mole fraction C to equilibrium. One can easily check from formula (3) that for

previously given set of parameters and W<sub>7</sub>=3.744·10<sup>20</sup> the equilibrium concentration  $C = C_{eq}^{\infty} = 0.0041$  in the

parent phase is reached at first step of calculation. For other  $W>W_7$  simulations gave the delay. This is the so-called "mob" ("traffic jam") effect [8-10] in which nuclei try to grow simultaneously. It means the prohibition of separation in this "mob" case because of the nuclei became subcritical [10-12].

Work is now in progress within our group in order to take into account the homogeneus nucleation simultaneously with the heterogeneus. This would lead to the comparison of these two mechanisms and their efficiency.

Another modification is necessary as well, that is including the coagulation mechanism of growth which seems to be important in the case of big number of nuclei and at intermediate and coarsening stages for real systems.

#### **IV. Resume**

The present model indicates that:

1. In the case of small density of heterogeneous nucleation sites the process undergoes three stages: independent growth, intermediate stage and Ostwald ripening stage. Hereby the time dependence of mean radius of supercritical new phase particles is parabolic during independent growth stage, and satisfies characteristic power law with 1/3 exponent at the coarsening stage.

2. The intermediate stage is characterized by lower growth rate of the mean cluster size than typical rates for independent growth stage and asymptotic ripening as it was found experimentally as well as theoretically.

3. The increasing the number of nucleation centers leads to disappearing the independent growth stage and reducing the time interval of the intermediate stage. In such cases the process directly undergoes phase separation by Ostwald ripening. If one increases the number of heterogeneous nucleation sites still more then it yields total prohibition of separation.

4. According to the numerical results, coarsening ( $< R_{ocr} > \tau^{0.29}$ ) starts far from equilibrium concentration is

reached. Ostwald ripening may consist of two consequent stages. At first the redistribution of matter between precipitates leads to further changing the composition of parent phase until equilibrium reached and at second the prolongation of the precipitates growth takes place in matrix with nearly constant composition.

5. In standard Lifshits-Slyozov-Wagner theory the mean radius of a new phase particles always increases. Taking into account all supercritical as well as subcritical nuclei leads to not negligible contribution of the number of subcritical nuclei to size distribution function and mean radius  $\langle R \rangle$ . The mean radius  $\langle R \rangle$  may increase as well as decrease at different stages of evolution. When the average droplet radius  $\langle R \rangle$  becomes approximately constant then the intermediate stage starts.

In usual theory of homogeneous nucleation and separation the coarsening starts when average droplet radius  $\langle R \rangle$  starts to increase. This criterion is not strict for the case of big number of heterogeneous nucleation sites, when there is no intermediate stage.

6. Presented analysis may be interpreted as the influence of finite size of a system.

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