



Article Study on the Phase Selection and Debye Temperature of Hyper-Peritectic Al-Ni Alloy under High Pressure

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Abstract: The phase selection of hyper-peritectic Al-47wt.%Ni alloy solidified under different pressures was investigated. The results show that Al_3Ni_2 and Al_3Ni phases coexist at ambient pressure, while another new phase α -Al exists simultaneously when solidified at high pressure. Based on the competitive growth theory of dendrite, a kinetic stabilization of metastable peritectic phases with respect to stable ones is predicted for different solidification pressures. It demonstrates that Al_3Ni_2 phase nucleates and grows directly from the undercooled liquid. Meanwhile, the Debye temperatures of Al-47wt.%Ni alloy that fabricated at different pressures were also calculated using the low temperature heat capacity curve.

Keywords: high pressure; Al-Ni alloy; grain growth; phase selection; debye temperature



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1. Introduction

As high temperature materials, the potential application value of intermetallic compounds has attracted extensive attention. Among these intermetallic compounds, the Al Ni series is one of the most important and promising candidate materials in high temperature environment. It is mainly used in the fields of aviation, blades, aerospace vehicles, etc. [1]. According to the phase diagram of Al-Ni alloy, there are abundant intermetallic compounds, such as AlNi, Al₃Ni, AlNi₃ and Al₃Ni₂, in the composition range of peritectic reaction. Meanwhile, Peritectic alloys cover some important structural and functional materials, including Ti-Al intermetallic compound [2], Fe-Cr-Ni stainless steel [3], Nd-Fe-B magnetic material [4] and high temperature superconducting material [5,6]. Three stages are often identified during peritectic solidification: peritectic reaction, peritectic transformation and direct solidification of peritectic phase. It has been reported that cooling velocity can dramatically influence the nucleation and growth of the peritectic phase [7]. Löser et al. [8] reported that for Co₇₅Si₂₅ peritectic alloy, the equilibrium solidification will be replaced by the direct growth of peritectic phase after exceeding the critical undercooling.

However, most work focuses on the peritectic system, in which merely the temperature is considered. Another thermodynamic parameter, pressure, is normally ignored. According to the phase law, pressure is also a vital parameter that can influence the solidification process dramatically, including the melting temperature [9], coefficient of solute diffusion [10], generation of new phases [11] and liquidus temperature [10]. Meanwhile, the bond energy also be intensively affected by the pressure as the melting point of the metal materials will change after solidification under pressure, thus, in turn influencing the physical properties.

Debye temperature is one of the important physical properties of metal materials. It is not only the direct characterization of atomic force, but is also closely related to the atomic interaction potential energy, coefficient of thermal expansion, heat capacity, elastic modulus [12–14], etc. Therefore, the main purpose of this work is to investigate the effects of high pressure on phase selection and physical properties of Al-47wt.%Ni hyper-peritectic alloy, which will further enrich the theory of high pressure solidification.

2. Materials and Methods

Al-47wt.%Ni alloy was synthesized by conventional casting process using pure Al and Ni (Purity 99.9%). Then, the samples were cut into cylinders (Φ 4.5 mm × 8 mm). The 700 ton multi anvil apparatus (Japan) is the equipment needed for high pressure solidification. The calibration of pressure is based on the phase transition point (2.55 GPa) of Bi from type I to type II. At the same time, the R-type thermocouple was inserted in the center of the assembled sample to calibrate the temperature [15,16]. The pressures were set as 2 GPa, 3 GPa and 4 GPa. The graphite tube was used as heating furnace, pyrophyllite was used as encapsulant and pressure transmitting medium and ZrO₂ was used as insulation layer. Afterwards, the assembly block was placed into the chamber. The next steps were to increase the pressure to the target value, keep the temperature at 1723 K for 50–60 min, then cool down to the room temperature and relieve the pressure.

The phases of the alloy after different solidification pressures were characterized by a Rint 2200 XRD (Rigaku Corporation, Tokyo, Japan) with monochromatic Cu-K_{α} radiation. Morphologies and compositions were examined on SEM (SU8010, Hitachi, Tokyo, Japan) operated at 15 kV equipped with an Energy Dispersive Spectrometer (EDS) (SU8010, Hitachi, Tokyo, Japan). The low temperature heat capacity property was measured with Physical Property Measurement System (PPMS, Quantum Design Company, Tokyo, Japan).

3. Results and Discussion

Figure 1 shows the phase composition of Al-47wt.%Ni alloy solidified under different pressures. It can be seen that Al₃Ni₂ and Al₃Ni phases coexist when solidified at ambient pressure. However, when the pressure is 2 GPa, 3 GPa and 4 GPa, additional new α -Al peaks present at the diffraction angles of 38.4°, 38.86° and 38.96°, respectively, indicating the formation of new phase, and the peaks of the α -Al shift to the higher angle. Meanwhile, the (102) diffraction peak of Al₃Ni₂ phase disappears when solidified at 4 GPa.



Figure 1. X-ray diffraction patterns of Al-47wt.%Ni alloy synthesized under different pressures.

Based on the equilibrium phase diagram of Al-Ni alloy, the solidification of Al-47wt.%Ni starts with the nucleation of AlNi phase from the metastable liquid. When cooling down to 1392 K, peritectic reaction $L + AlNi \rightarrow Al_3Ni_2$ will occur. When it con-

tinues cooling down to 1107 K, another peritectic reaction $L + Al_3Ni_2 \rightarrow Al_3Ni + Al_3Ni_2$ happens. At the end of solidification, the peritectic Al₃Ni and Al₃Ni₂ phase coexist as can be seen in Figure 2. Figure 3 shows the backscattered electron (BSE) microstructure of Al-47wt.%Ni alloy solidified under different pressures. The results demonstrate that the microstructure is composed of gray and white phases at ambient pressure as shown in Figure 3a,b. When it solidifies under high pressures, there are grey, white and black phases existing simultaneously, as can be seen in Figure 3c-h. Furthermore, when the pressure increases to 4 GPa, there is a layer generated around the white phase (Figure 3g,h). The EDS analysis of each phase in Figure 3 is shown in Table 1. Combined with the XRD results, it can be obtained that the grey phase is Al₃Ni, the black one is α -Al phase and the white phase refers to Al_3Ni_2 . Meanwhile, the composition of the layer is 50.43 wt.%, indicating that it is the combination of Al₃Ni₂ and Al₃Ni phase. The Al₃Ni₂ phase is completely wrapped in the Al₃Ni phase. However, not all Al₃Ni phases contain the Al₃Ni₂ phase. The Al_3Ni phase, which does not contain Al_3Ni_2 phase, is located at the inter-granular of Al₃Ni. It is worth pointing out that when the peritectic reaction cannot be carried out due to the inability of the primary phase to direct contact with the liquid phase, the growth of the peritectic phase can only be realized by two mechanisms: the solid phase diffusion (peritectic transformation) and the direct solidification from the undercooled liquid phase. Furthermore, as the solute diffusion is greatly suppressed under pressure [17], it is reasonably speculated that the peritectic transformation stage cannot be completely carried out. The hyper-eutectic reaction will occur in the remnant undercooled liquid. Meanwhile, as the existence of a large number of Al₃Ni phases, the divorced eutectic reaction takes place and the α -Al phase grows alone at the location of inter-granular of Al₃Ni phases.



Figure 2. The phase diagram of Al-Ni alloy.

There are two kinds of competition between the primary and peritectic phases during peritectic solidification: nucleation competition and growth competition [18]. Under the same solidification conditions, the phase with the minimum undercooling nucleates preferentially. Meanwhile, previous studies [19–22] revealed that the phase growth velocity is determined by the interfacial temperature. Therefore, the maximum interfacial temperature criterion is adopted in the determination of the stable phase growth. It should be pointed out that before the application of the maximum interface temperature criterion, one should be sure that all the phases involved have the nucleation possibility. Firstly, the undercooling of each phase involved was calculated. Then, the interface temperature for both the primary phase and the peritectic phase were calculated. Finally, the interface temperature under the



same solidification conditions was compared to detect which stage is the peritectic reaction going through.

Figure 3. The microstructure of Al-47wt.%Ni alloy solidified at different pressures: (**a**,**b**) ambient pressure, (**c**,**d**) 2 GPa, (**e**,**f**) 3 GPa, (**g**,**h**) 4 GPa.

Pressure	Phase Color	Ni wt.%
Ambient pressure	Gray	42.8
	White	57.5
2 GPa	Black	1.03
	Grey	43.0
	White	58.1
3 GPa	Grey	43.09
	White	60.04
	Black	1.29
4 GPa	Grey	43.06
	White	60.52
	Black	1.87
	Layer	50.43

Table 1. The EDS results of Al-47wt.%Ni in Figure 2.

The growth temperature of the interface (T_i) can be expressed as [23,24]:

$$T_i = T_L - \Delta T \tag{1}$$

According to the model established by Boettinger, Coriell and Trivedi, the undercooling can be given as follows [25]:

$$\Delta T = \Delta T_t + \Delta T_c + \Delta T_r + \Delta T_k \tag{2}$$

The kinetic undercooling of intermetallic compounds with low atomic diffusion rate and dendrite growth with high solidification rate cannot be ignored [26]. However, in the case of constitutional undercooling, the situation may be totally different for the intermetallic compound phase. As the necessary condition for the existence of constitutional undercooling is that the actual temperature gradient at the front of the solid/liquid interface is smaller than that caused by the redistribution of solute, it is worth pointing out that the solute redistribution for the phase with nil solubility may be totally different. Hence, the constitutional undercooling condition for the intermetallic compound phase should be inferred in the first place.

According to the solid solubility, the intermetallic compounds in Al-Ni alloy system can be divided into two types: those with high solid solubility and those with almost zero solid solubility. For the intermetallic compounds with a certain solubility, the solute distribution characteristics can be referred to the solid solution phase. However, when the solid solubility is nil, the solute distribution characteristics are completely different. The solute distribution of solid solution has been widely studied [27]. However, there are few studies on intermetallic compounds with nil solid solubility.

Therefore, the status of the constitutional undercooling at the frontier of Al_3Ni interface is considered first. The solute distribution at the frontier of Al_3Ni interface is shown in Figure 4, and the corresponding equation derived by Liu [28] is shown as follows:

$$C_L = C_0 + (C_L^* - C_0)exp\left(-\frac{Vz'}{D}\right)$$
(3)

According to the mass balance law, the distribution of the solute at the frontier of Al₃Ni is written as below:

$$C_{L}^{*} = (C_{0} - C_{\beta}) \frac{V}{D} z + C_{0}$$
(4)



Figure 4. (a) The effect of high pressure on the interfacial undercooling of AlNi, Al₃Ni₂ and Al₃Ni phase. (b) The effect of high pressure on the interfacial temperature of AlNi, Al₃Ni₂ and Al₃Ni phase.

The result shows that C_L^* decreases linearly with the increase of z when $C_0 < C_\beta$. However, when $C_0 > C_\beta$, with the increase of z, C_L^* increases linearly. The solute concentration remains unchanged far away from the solid liquid interface. Therefore, the interface concentration gradient is negative. Based on the equilibrium phase diagram, the temperature gradient in liquid at the frontier of solid liquid interface caused by the solute redistribution is also negative. Therefore, the conclusion that there is no constitutional undercooling exists for Al₃Ni phase can be obtained.

Then, the interface undercooling of each phase in Al-47wt.%Ni alloy is expressed as below: $\Delta T \dots = \Delta T + \Delta T + \Delta T + \Delta T_{t}$

$$\Delta T_{AINi} = \Delta T_c + \Delta T_r + \Delta T_t + \Delta T_k$$

$$\Delta T_{Al_3Ni_2} = \Delta T_c + \Delta T_r + \Delta T_t + \Delta T_k$$

$$\Delta T_{Al_2Ni} = \Delta T_r + \Delta T_t + \Delta T_k$$
(5)

The undercooling of the dendrite tip can be given as follows [23]:

$$\Delta T_c = mc_0 \left[1 - \frac{1}{1 - I_{\nu}(P_c)(1 - k_P)} \right]$$

$$\Delta T_t = \frac{\Delta h_f}{R} I(P_t)$$

$$\Delta T_r = \frac{2f}{R}$$

$$\Delta T_k = \frac{U}{\mu}$$

(6)

Meanwhile, the marginal stable wavelength of perturbations at the solid liquid interface with constitutional undercooling is shown as follows [23]:

$$R = \frac{\Gamma/\sigma^{*}}{\frac{P_{t}\Delta h_{f}}{c}\xi_{t} - \frac{2P_{c}C_{0}(1-k_{P})m}{1-(1-k_{P})I_{v}(P_{c})}\xi_{c}}$$

$$\xi_{t} = 1 - \frac{1}{\sqrt{1 + \frac{1}{\sigma^{*}P_{t}^{2}}}}$$

$$\xi_{c} = 1 + \frac{2k_{P}}{1-2k_{P} - \sqrt{1 + \frac{1}{\sigma^{*}P_{c}^{2}}}}$$
(7)

Meanwhile, for the phase with no constitutional undercooling at the solid liquid interface, the marginal stable wavelength of perturbations can be deduced as below:

Firstly, assume that the perturbation displays a sinusoidal ripple. The interface temperature T_{Φ} can be derived from the assumption of local equilibrium:

$$T_{\Phi} = T_f + m\Delta C - V_{\Phi}/\mu - \Gamma K_{\Phi}$$

= $T_f - m(C_0 - C^*) - V_{\Phi}/\mu - \Gamma K_{\Phi}$ (8)

Therefore, the interface temperature at the peak (*t*) and the valley (*d*) can be expressed

as:

as:

$$T_{t} = T_{f} - m(C_{0} - C_{t}) - V_{\phi} / \mu_{t} - \Gamma K_{t}$$

$$T_{d} = T_{f} - m(C_{0} - C_{d}) - V_{\phi} / \mu_{d} - \Gamma K_{d}$$
(9)

Corresponding kinetic coefficient and curvature can be written as follows [26]:

$$\mu_t = \frac{V_0 \Delta H_f}{R_g T_f T_t}$$

$$\mu_d = \frac{V_0 \Delta H_f}{R_g T_f T_d}$$
(10)

The curvatures of the peak and valley can be determined by evaluating the second derivative of the interface shape function at $y = \lambda/4$ and $y = 3\lambda/4$:

$$K_t = -K_d = \frac{4\pi^2 \varepsilon}{\lambda^2} \tag{11}$$

Then, the difference between the peak and valley interface temperatures can be written

$$T_t - T_d = m(C_t - C_d) - \left(\frac{V_\phi}{\mu_t} - \frac{V_\phi}{\mu_d}\right) - \Gamma(K_t - K_d)$$
(12)

Since $T_t - T_d = -2\varepsilon G$, $C_t - C_d = 2\varepsilon G_C$, substituting Equation (10) into the above equation, Equation (13) can be obtained:

$$\lambda = 2\pi \left(\frac{\Gamma}{mG_C - G\left(\frac{V_{\phi}R_g T_f}{V_0 \Delta H_f} + 1\right)} \right)^{1/2}$$
(13)

Meanwhile, the radius of the dendrite tip *R* can be expressed as [29]:

$$R = \lambda \tag{14}$$

Furthermore, the temperature gradient *G* in the liquid phase at the stable growing dendrite front can be written as shown below [29]:

$$G = \frac{2P_t \Delta h_f}{cR} \tag{15}$$

The concentration of the liquid and solid phase at the interface is C^* and C_β , respectively, when the solid-liquid interface of Al₃Ni move forward with the local solidification velocity *V*. When $C_0 > C_\beta$, m > 0, k > 1, $V(C^* - C_\beta) = DG_C$. Then,

$$G_{C} = \frac{V(C^{*} - C_{\beta})}{D} = \frac{2P_{C}(C^{*} - C_{\beta})}{R}$$
(16)

Finally, the interface concentration gradient in liquid G_C for Al₃Ni with nil solubility is shown as follows:

$$G_{\rm C} = \frac{2P_{\rm C}}{R} \frac{C_0 - C_{\beta}}{1 - I(P_{\rm C})} \tag{17}$$

Substituting Equations (15) and (17) into Equation (13), the expression of dendrite tip radius can be simplified as:

$$R = \frac{4\pi^2 \Gamma}{m\left(\frac{2P_C(C_0 - C_\beta)}{1 - I(P_C)}\right) - \left(\frac{2P_t \Delta h_f}{c}\right) \left(\frac{V_\phi R_g T_f}{V_0 \Delta H_f} + 1\right)}$$
(18)

The relationship between high pressure and the solute distribution coefficient k, the diffusion coefficient D_L , and the interface solidification velocity is also been considered for calculation [29]. The effect of high pressure on the undercooling of each phase can be calculated by combining the parameter in Table 2, and the results can be seen in Figure 4a. It shows that ΔT_{Al3Ni2} is less than $\Delta T_{\beta-Al3Ni}$, followed by the undercooling of AlNi phase. Therefore, the peritectic reaction L + AlNi \rightarrow Al₃Ni₂ will not occur, and the Al₃Ni₂ phase directly nucleates from the undercooled liquid, while peritectic reaction L + Al₃Ni₂ \rightarrow Al₃Ni will happen. Combined with Figure 3, it is clear to see that peritectic transformation is the stage it undergoes. The effect of high pressure on the interface temperature of AlNi, Al₃Ni₂ and Al₃Ni is shown in Figure 4b. It can be seen that the interface temperature of Al₃Ni₂ is higher than Al₃Ni. That also proves that part of the Al₃Ni phases grow by the peritectic transformation mechanism.

Physical Quantities	Al-47%Ni
Liquidus temperature T _L (K)	1392
Density (Al _{solid}) ρ_{SAl} (g/cm ³)	2.7
Density (Al _{liquid}) P_{LAl} (g/cm ³)	2.375
Density (Ni _{solid}) ρ_{SNi} (g/cm ³)	8.908
Density (Ni _{liquid}) P_{LNi} (g/cm ³)	7.81
κ_{Al} (W/m·K)	235
K_{Ni} (W/m·K)	91
Eutectic structure contact angle	-
Liquidus slope of α , $m_{L\alpha}$ (K/%)	-3.51
Liquidus slope of β , $m_{L\beta}$ (K/%)	18.5
<i>m_{LA13Ni2}</i> (K/%)	23.25
m_{LAINi} (K/%)	29.8
$D_L (\mathrm{m^2/s})$	$0.78 \times 10^{-7} \times \exp(-7.44 \times 10^{-20}/K_BT)$ [30]

Table 2. The physical parameters used for calculation.

The low temperature heat capacity *C* (*T*) of Al-47wt.%Ni alloy synthesized under different pressures in the temperature range of 2–300 K is shown in Figure 5. The insets in Figure 5 show a linear *C*(*T*)/*T* versus T^2 behavior for $2 \le T \le 15$ K. According to previous research [31], the Debye temperature under different pressures can be obtained, as shown in Table 3. It can be concluded that the Debye temperatures at ambient pressure, 2 GPa and 4 GPa are 518.63 K, 524 K and 594.15 K, respectively.

 Table 3. The Debye temperature of hyper-peritectic Al-47wt.%Ni alloy solidified under different pressures.

Pressure (GPa)	β ^a (mJ/mol·K)	Θ_D/K
0	0.01672	518.63
2	0.01621	524
4	0.01112	594.15

 β^{a} reflects the low-*T* Debye *T*³ lattice contribution together with a three-dimensional atomic force microanalysis (AFM) spin-wave contribution.



Figure 5. Heat capacity *C* vs. temperature *T* and fits of C_{latt} (T) to the data over restricted temperature intervals for (**a**) ambient pressure; (**b**) 2 GPa; (**c**) 4 GPa. The respective insets show C(T)/T vs. T^2 for 2 K < *T* < 15 K.

4. Conclusions

The microstructure and phase composition of hyper-peritectic Al-47wt.%Ni alloy solidified under different pressures were investigated. Meanwhile, the phase selection between primary and peritectic phase and Debye temperature were also calculated. The following conclusions can be obtained:

- (1) The new diffraction peak of α -Al phase generates during solidification under high pressure.
- (2) A dendrite tip radius model for the growth of intermetallic compounds with nil solubility was established.
- (3) There is no constitutional undercooling that exists at the frontier of Al_3Ni interface.
- (4) Peritectic Al₃Ni₂ phase nucleates and grows directly from the metastable liquid.

- (5) When Al-47wt%Ni alloy synthesized at high pressure, $\Delta T_{AlNi} < \Delta T_{Al3Ni2} < \Delta T_{\beta-Al3Ni}$.
- (6) The Debye temperatures of Al-47wt%Ni alloy synthesized at ambient pressure, 2 GPa and 4 GPa are 518.63 K, 524 K and 594.15 K, respectively.

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