

Characterization of microstructural length scales in directionally solidified Sn–36%Ni peritectic alloy

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Abstract: Sn–36%Ni peritectic alloys were directionally solidified at different growth rates under a constant temperature gradient (20 K/mm), the dependences of microstructural characteristic length scales on the growth rate were investigated. Experimental results are presented, including primary and higher order dendrite arm spacings λ_1 , λ_2 , λ_3 and dendrite tip radius R of primary Ni_3Sn_2 phase. Comparisons between the theoretical predictions and the experimental results show that, for the primary dendrites, $\lambda_1=335.882v^{-0.21}$, which is in agreement with the Kurz–Fisher model; for the secondary dendrites, $\lambda_2=44.957v^{-0.277}$, which is consistent with the Bouchard–Kirkaldy model; for the tertiary dendrites, $\lambda_3=40.512v^{-0.274}$; for the dendrite tip radius, $R=22.7v^{-0.36}$. The experimental results also show that the λ_1/λ_2 changes greatly with increasing growth rate while the λ_1/λ_3 has no significant change, indicating that tertiary dendrite arms have a more similar growth characteristics to primary dendrites compared with secondary dendrites. The λ_1/R ranges from 2 to 2.3 with the increase of growth rate.

Key words: Sn–Ni alloy; directional solidification; dendrite arm spacing; dendrite tip radius

1 Introduction

The dendrite solidification microstructure has been observed in many alloys [1–4]. It is generally described by suitable length scales, such as primary and higher order dendrite arm spacing (λ_1 , λ_2 , λ_3) and dendrite tip radius (R) which have been characterized as functions of alloy tip growth rate, solute concentration and temperature gradient [5–7]. In peritectic solidification, dendrites of primary phase often grow in a matrix of peritectic phase over a wide range of growth rates, thus, investigation on the dependence of microstructural characteristic length scales on solidification processing parameters such as growth rate in peritectic alloys is expected.

The primary dendrite arm spacing (λ_1) and secondary dendrite arm spacing (λ_2) have been studied in many peritectic alloys such as Pb–Bi [8], Cu–Zn [9]. MA et al [9] also proposed that the values of $\lambda v^{1/2}$ for peritectics are generally two orders of magnitude higher than those for eutectics. They also held that the arm coarsening for primary phase should be suppressed by

the formation of peritectic phase surrounding the primary phase, which has been demonstrated in some peritectic alloys [8]. However, the tertiary dendrite arm spacing and dendrite tip radius have never been discussed in these investigations. Neither have the relations between these characteristic length scales been concerned up to now in peritectic solidification process, which is insufficient to describe the dependence of solidification condition on the characteristic length scales in peritectic alloys.

In the present work, Sn–Ni peritectic alloy which has attracted wide attention due to its extensive application of developing lead-free solders [10] and anode material for lithium ion battery with better cyclic performance and higher capacity [11] was chosen. Microstructural characteristic length scales of primary Ni_3Sn_2 phase were measured and compared with theoretical predictions in directionally solidified Sn–36%Ni (mole fraction) alloy at extensive growth rates (2–200 $\mu\text{m/s}$). First, these characteristic length scales of the primary Ni_3Sn_2 phase in directionally solidified Sn–36%Ni alloys were measured and compared with the theoretical predictions; then, relations

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among these characteristic length scales were investigated and compared with other alloys.

2 Experimental

2.1 Sample production process

The Sn–36%Ni alloy was induction melted from pure Ni and Sn (99.9%). As-cast rods of 3 mm in diameter and 110 mm in length were machined from the ingot by a spark machining. Experiments consisting of melting followed by directional solidification were carried out in a Bridgman-type furnace which consists of a resistance furnace, a water cooled liquid metal bath filled with liquid Ga–In–Sn alloy, and an adiabatic zone which is located between the heater and the cooler, as previously described. For each experiment, the furnace was heated to 1250 °C to melt the alloy, and then was held for 30 min to homogenize the melt. Solidification of Sn–36%Ni peritectic alloy was carried out at different growth rates (2, 5, 10, 15, 20, 40 and 200 $\mu\text{m/s}$) under a constant temperature gradient (20 K/mm) in the Bridgman-type furnace. After a predetermined distance of 30 mm reached, the samples were quenched into liquid Ga–In–Sn alloy quickly to preserve the solid/liquid interface. According to results by SMITH et al [12], the length of the initial transient zone can be obtained using $l=4D/vk$, where D is the solute diffusion coefficient in liquid, v is the pulling rate and k is the solute distribution coefficient. In the present work, for the lowest growth rate 2 $\mu\text{m/s}$, the length of the initial transient zone is 15 mm which is much smaller than 30 mm, thus the growth distance of 30 mm is long enough to achieve the steady-state.

2.2 Measurement of temperature gradient and microstructural length scales

The samples were placed into 99.99% pure alumina crucibles of 4/5.5 mm in inner/out diameter and 150 mm in length. The temperature gradient was measured by PtRh30–PtRh6 thermocouples that were placed near the outside surface of the alumina crucible. One thermocouple was placed 5 mm from the bottom of the sample where was near the solid/liquid interface. The other was placed 15 mm from the bottom of the sample where was the liquid region. The temperature gradient close to the solid/liquid interface was measured to be 20 K/mm. The temperature gradient can be changed by changing the temperature of the specimen. To keep the temperature gradient constant during directional solidification, the temperatures of the cooler and the hotter part of the furnace were kept constant by an automatic temperature controlling system.

The phases in the samples were identified by a Rigaku D/max-RB X-ray diffractometer with monochromatic Cu K_α radiation. Both optical and scanning electron microscopy (SEM) were used to characterize the microstructure of the specimens after polishing and etching by a solution of 10 g FeCl_3 +20 mL HCl +180 mL H_2O . Backscattered electron imaging (BSE) was used to identify the phases. Primary dendrite arm spacing was measured by $\lambda_1 = \sqrt{A/N}$ described by JACOBI and SCHWERDTFEGGER [13], where A is the area of the transverse section and N is the number of the primary dendrites counted. The secondary dendrite arm spacing (λ_2) was measured by averaging the distance between adjacent side branches on the longitudinal section of a primary arm. The tertiary value (λ_3) was measured in longitudinal section of samples. The primary dendrite tip radius (R) was measured by comparing the unperturbed tip region to a series of parabolas of known curvatures as described by SOMBOONSUK et al [6]. The procedure involved projecting a greatly enlarged tip image from the photographic negative onto the standard parabolas and adjusting the magnification until a best fit was obtained. At least 15 readings were taken for each sample and the values of these characteristic length scales were the mean of these measured values.

3 Results

3.1 Microstructure of directionally solidified Sn–36%Ni alloy

Equilibrium solidification process of Sn–36%Ni alloy [14] begins at $t_L=1040$ °C with a precipitation of primary Ni_3Sn_2 phase: $L \rightarrow \text{Ni}_3\text{Sn}_2$, followed by a peritectic reaction at $t_P=798$ °C: $L+\text{Ni}_3\text{Sn}_2 \rightarrow \text{Ni}_3\text{Sn}_4$, and the remaining liquid will solidify through the eutectic reaction at $t_E=231.15$ °C: $L \rightarrow \text{Ni}_3\text{Sn}_4+\text{Sn}$. Backscattered electron (BSE) images of microstructure of directionally solidified Sn–36%Ni peritectic alloy at different growth rates (2–200 $\mu\text{m/s}$) are shown in Fig. 1. The quenched liquid is behind the solid/liquid interface. Ahead of the interface is the mushy zone, where the solid and the liquid coexist.

As shown in Fig. 2, according to the EDX results of the solubility of components in each phase, the dark gray, bright gray and white phases correspond to the primary Ni_3Sn_2 phase, peritectic Ni_3Sn_4 phase and ($\text{Ni}_3\text{Sn}_4+\text{Sn}$) eutectic, respectively. The compositions of the two phases comprising the eutectic are 56.23% and 99.67% Sn, respectively, which means that the two phases are Ni_3Sn_4 and Sn. In the present work, developed dendrite morphology forms, as shown in Fig. 3(a); primary and higher order dendrites can be observed; tertiary dendrite arms initiating from secondary dendrite arms are clearly illustrated in Fig. 3(b).

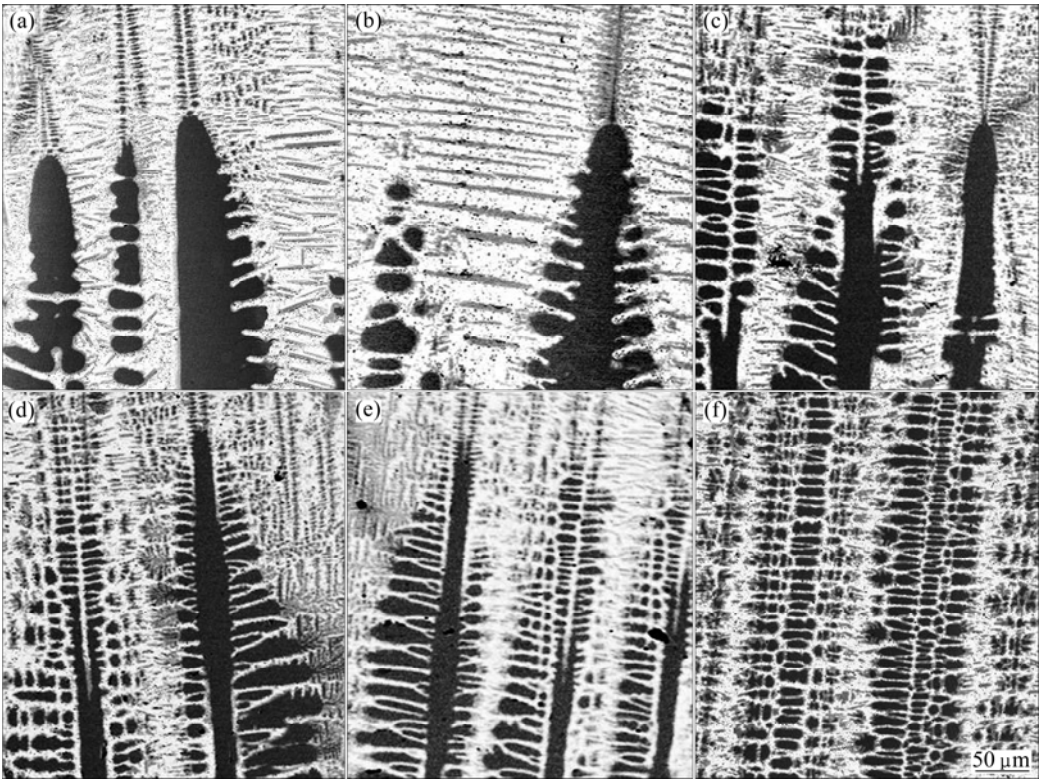


Fig. 1 SEM micrographs showing evolution of morphology of solid/liquid interface in directionally solidified Sn–36%Ni peritectic alloy: (a) $v=2\text{ }\mu\text{m/s}$; (b) $v=5\text{ }\mu\text{m/s}$; (c) $v=10\text{ }\mu\text{m/s}$; (d) $v=15\text{ }\mu\text{m/s}$; (e) $v=20\text{ }\mu\text{m/s}$; (f) $v=200\text{ }\mu\text{m/s}$

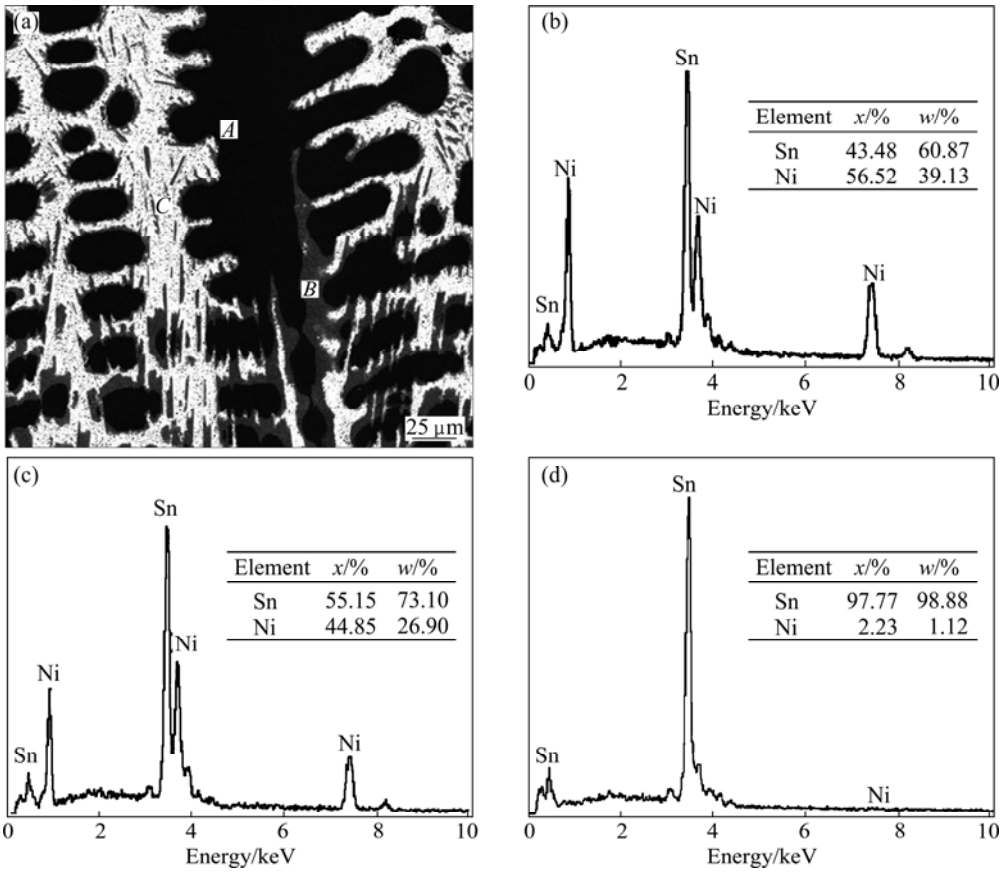


Fig. 2 Chemical composition analysis of Sn–36%Ni peritectic alloy by SEM-EDX: (a) BSE image of directionally solidified Sn–36%Ni peritectic alloy near peritectic interface ($v=10\text{ }\mu\text{m/s}$, $G=20\text{ K/mm}$); (b) Dark gray phase A; (c) Bright gray phase B; (d) White phase C

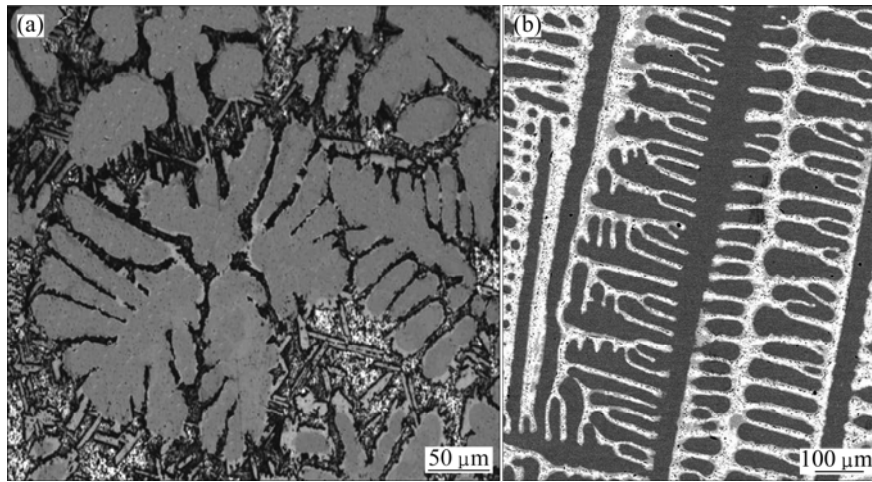


Fig. 3 Dendritic morphology in directionally solidified Sn-36%Ni peritectic alloy at growth rate of $v=15 \mu\text{m/s}$: (a) OM image of transverse section; (b) BSE image of longitudinal section

3.2 Primary dendrite arm spacing λ_1

Studies characterizing the variation of primary dendrite arm spacing with alloy composition, solidification rate (v), and temperature gradient (G) in the liquid involving solidification both in steady-state heat flow [15–18] and in unsteady-state regime [7] have been reported.

HUNT [15] derived a model for the primary spacing λ_1 :

$$\lambda_1 = \frac{2.83 \times (k\Delta T_0 D \Gamma)^{1/4}}{v^{1/4} G^{1/4}} \quad (1)$$

where k is the equilibrium distribution coefficient, D is the diffusion coefficient in the liquid, Γ is the Gibbs–Thomson coefficient, G is the temperature gradient and ΔT_0 is the equilibrium solidification temperature range given by

$$\Delta T_0 = \frac{m(k-1)C_0}{k} \quad (2)$$

where m is the liquidus slope and C_0 is the initial alloy composition.

Later, using a simplified solution to the wavelength instability, KURZ and FISHER [5] gave

$$\lambda_1 = \frac{4.3 \times (\Delta T_0 D \Gamma)^{1/4}}{k^{1/4} v^{1/4} G^{1/4}} \quad (3)$$

A good approximation for λ_1 was derived by KURZ et al [16,17] as

$$\lambda_1 = 4\pi \left(\frac{D\Gamma}{v\Delta T_0} \right)^{1/2} \quad (4)$$

HUNT and LU [18] proposed an analytical expression for corresponding cellular spacing from their numerical predictions of the lower limit of stable primary

dendritic/cellular spacing, namely:

$$\lambda_1 = 8.18k^{-0.335} \left(\frac{\Gamma}{mC_0(k-1)} \right)^{0.41} \left(\frac{D}{v} \right)^{0.59} \quad (5)$$

BOUCHARD and KIRKALDY [7] derived a spacing formula for steady-state solidification and have proved its utility in the unsteady regime. The formula is given by

$$\lambda_1 = 120 \left(\frac{16(1-C_0)^{1/2} G_0 \varepsilon \sigma T_M D}{(1-k)m\Delta H G v} \right)^{1/2} \quad (6)$$

The physical parameters used in calculations of the Sn–Ni peritectic system are illustrated in Table 1.

Table 1 Physical parameters for Sn–Ni system

Symbol	Parameter	Unit	Value	Reference
T_M	Melting temperature of solvent	°C	232	[14]
C_0	Composition of α at T_p	% (mole fraction)	36	[14]
m_α	Liquidus slope	°C/%	−14.235	[14]
k_α	Distribution coefficient of α		0.543	[14]
Γ	Gibbs–Thomson coefficient	m·K	3×10^{-7}	[19]
D	Diffusion coefficient in liquid	m ² /s	5×10^{-9}	[19]
ε	Scaling factor for surface tension		6	[19]
σ	Liquid–solid surface energy	J/m ²	0.25	[19]
ΔH	Heat of fusion	J/kg	1.5×10^5	[19]

The predictions of these models are compared with the experimental results shown in Fig. 4. Variation of λ_1 versus v is essentially linear on the logarithmic scale. It can be found from Fig. 4 that the data form straight lines and the models discussed above show different constancies: the Hunt model (Eq. (1)) and the Kurz–Fisher model (Eq. (3)) show constancy of $\lambda_1 v^{1/4}$ for a given C_0 whereas the Kurz–Giovanola–Trivedi (Eq. (4)) and Hunt–Lu (Eq. (5)) models indicate constancy of $\lambda_1 v^{1/2}$ and $\lambda_1 v^{0.59}$, for a given C_0 , respectively. The Kurz–Fisher model exhibits excellent agreement with experimental data for Sn–36%Ni alloy. Our results show that $\lambda_1 = 335.882 v^{-0.21}$. Both the Hunt–Lu model and Kurz–Giovanola–Trivedi model predict large deviations from our experimental results. It can be found that the exponent value of growth rate (0.21) in the present work makes a large difference compared with the eutectics which have been reported (close to 0.5) [20,21].

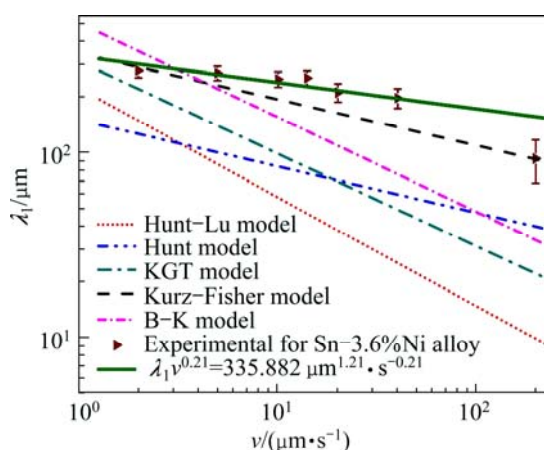


Fig. 4 Variation of primary dendrite arm spacing λ_1 with growth rate v under constant temperature gradient ($G = 20$ K/mm)

3.3 Secondary dendrite arm spacing λ_2

KATTAMIS and FLEMINGS [22] predicted that the secondary dendrite arm spacing λ_2 is proportional to the cube root of solidification time (t_f), and gave

$$\lambda_2 = 5.5(Mt_f)^{1/3} \quad (7)$$

$$M = \frac{\Gamma D \ln(C_1^m/C_0)}{m(k-1)(C_1^m - C_0)} \quad (8)$$

where C_1^m is the maximum concentration in the liquid. In the case of directional solidification, the local solidification time is given by $t_f = \Delta T / Gv$, where G is the temperature gradient, v is the growth rate, and ΔT is the non-equilibrium solidification range.

LANGER and MÜLLER–KRUMBHAAR [23] predicted a scaling law between secondary dendrite arm spacing λ_2 and the dendrite tip radius R as $\lambda_1/R=2$. With this scaling law, the variation in λ_2 under small Peclet

number conditions given by TRIVEDI and SOMBOONSUK [24] is

$$\lambda_2 = \left(\frac{8\Gamma DL}{kv\Delta T_0} \right)^{1/2} \quad (9)$$

For secondary dendrite arm spacing, BOUCHARD and KIRKALDY [7] also derived a formula:

$$\lambda_2 = 12\pi \left[\frac{4\sigma}{C_0(1-k)^2 \Delta H} \left(\frac{D}{v} \right)^2 \right]^{1/3} \quad (10)$$

The predictions of these models are compared with the experimental results shown in Fig. 5. The experimental results show that $\lambda_2 = 44.957 v^{-0.277}$ and it can be observed from Fig. 5 that the Bouchard–Kirkaldy (B–K) model exhibits excellent agreement with the experimental data. The commonly accepted Kurz–Fisher (K–F) model predicts larger deviation from our experimental results which can be attributed to the retard of coarsening of the secondary dendrite arms by peritectic reaction and transformation [7].

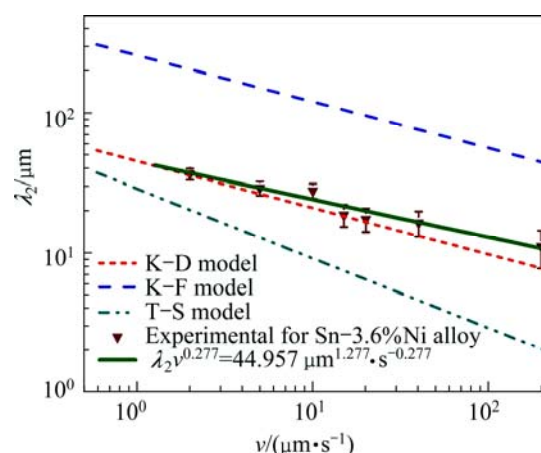


Fig. 5 Variation of secondary dendrite arm spacing λ_2 with growth rate v under constant temperature gradient ($G=20$ K/mm)

3.4 Tertiary dendrite arm spacing λ_3

Dendrites can adjust their primary spacing during growth without difficulty. If the primary spacing is too large, a tertiary arm initiating from the secondary branches will catch up to the growing primary tips and become one of them [25]. Investigations on tertiary dendrite arms are relatively scarce in the literature. They are more commonly mentioned on steady-state growth experiments where they are observed to grow past initiating secondary branches and go on to become primary arms [26]. GRUGEL [27] carried out experiments in directional steady-state growth of Al–Si alloys, and from the spacing measurements suggested a power law correlating λ_3 with local solidification

time (t_f):

$$\lambda_3 = 10(t_f)^{1/3} \quad (11)$$

Based on the experimental examination of both Sn–Pb and Al–Cu alloys, SÁ et al [28] proposed a 0.55 power law to characterize the variation of tertiary spacing with the cooling:

$$\lambda_3 = k(t_f)^{0.55} \quad (12)$$

where k is a coefficient which can be determined by regression analysis. The predictions of these models are compared with the experimental results in Fig. 6. By linear regression analysis we can obtain that $\lambda_3 = 40.512v^{-0.274}$. And it can be found from Fig. 6 that the 0.55 model ($\lambda_3 = 4.23t_f^{0.55}$) exhibits reasonable agreement with the experimental data at lower growth rates but predicts large deviations from the experimental results when v exceeds 20 $\mu\text{m/s}$.

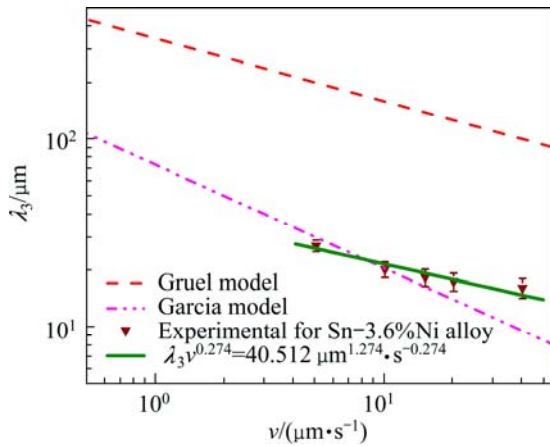


Fig. 6 Variation of tertiary dendrite arm spacing λ_3 with growth rate v under constant temperature gradient ($G = 20 \text{ K/mm}$)

3.5 Dendrite tip radius R

Numerous studies have been carried out on the dendrite tip radius R . HUNT [15] proposed that:

$$R = \left[\frac{2\Gamma D}{m(k-1)C_0 v} \right]^{1/2} \quad (13)$$

according to the Kurz and Fisher model [5]:

$$R = 2\pi \left[\frac{\Gamma D}{m(k-1)C_0 v} \right]^{1/2} \quad (14)$$

and according to the Trivedi model [24]:

$$R = \left[\frac{2k\Gamma DL}{m(k-1)C_0 v} \right]^{1/2} \quad (15)$$

where L is a constant which depends on the harmonic of perturbation. From the research of TRIVEDI, $L = l/[2(l+1)(l+2)]$, in which the $l=6$ harmonic is operative

for dendrite growth.

It can be seen from Eqs. (13)–(15) that the theoretical models for dendrite tip radius, R , are very similar and the differences among them are a constant only. The predictions of these models are compared with the experimental results shown in Fig. 7. It can be obtained through linear regression analysis that $R = 22.7v^{-0.36}$. It can be observed from Fig. 7 that the Trivedi model exhibits excellent agreement with the experimental data at low growth rates while the Kurz–Fisher model predicts small deviations from our experimental results when the growth rate v exceeds 20 $\mu\text{m/s}$.

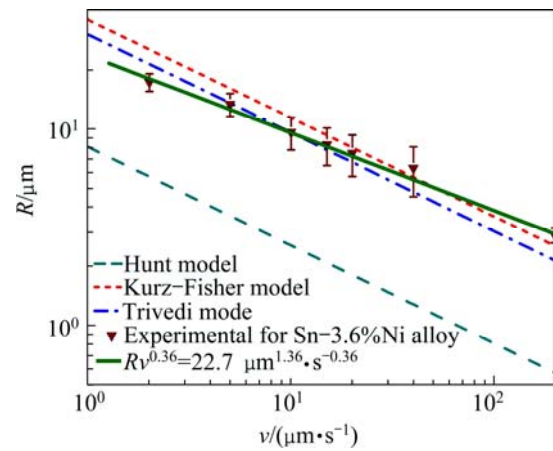


Fig. 7 Variation of dendrite tip radius R with growth rate v under constant temperature gradient ($G = 20 \text{ K/mm}$)

3.6 Relations between characteristic length scales

The λ_1/λ_2 has been used to estimate the permeability of the mushy zone, but only limited information is available in the literature about this ratio. CICUTTI and BOERI [29] developed an analytical model to estimate the λ_1/λ_2 and a roughly constant value was obtained for $(\lambda_1/\lambda_2):(\lambda_1/\lambda_2) \approx 2.6$. This is consistent with the values ranging from 2 to 4 suggested by WOLF [30]. But it can be observed from our experimental results in Fig. 8(a) that the λ_1/λ_2 is not constant but range from 7.6 to 14.1 with the increase of growth rate. This means that the primary dendrite arm spacing λ_1 decreases more slowly than the secondary dendrite arm spacing λ_2 when the growth rate increases. This is in consistent with our discussion in sections 3.2 and 3.3 that λ_1 and λ_2 exhibit power law of 1/4 and 1/3, respectively. For λ_2 exhibits much more dependence on interface curvature which has little effect on λ_1 , and the coarsening mechanism also has effect on λ_2 , thus the variations of λ_1 and λ_2 exhibit different tendencies.

The values of λ_1/λ_3 for Al–Cu, Pb–15%Sn (mass fraction) and Pb–30%Sn (mass fraction) alloys are 3.4, 5, 6, respectively [28]. The values of this ratio in this study are shown in Fig. 8(b), and it can be found that

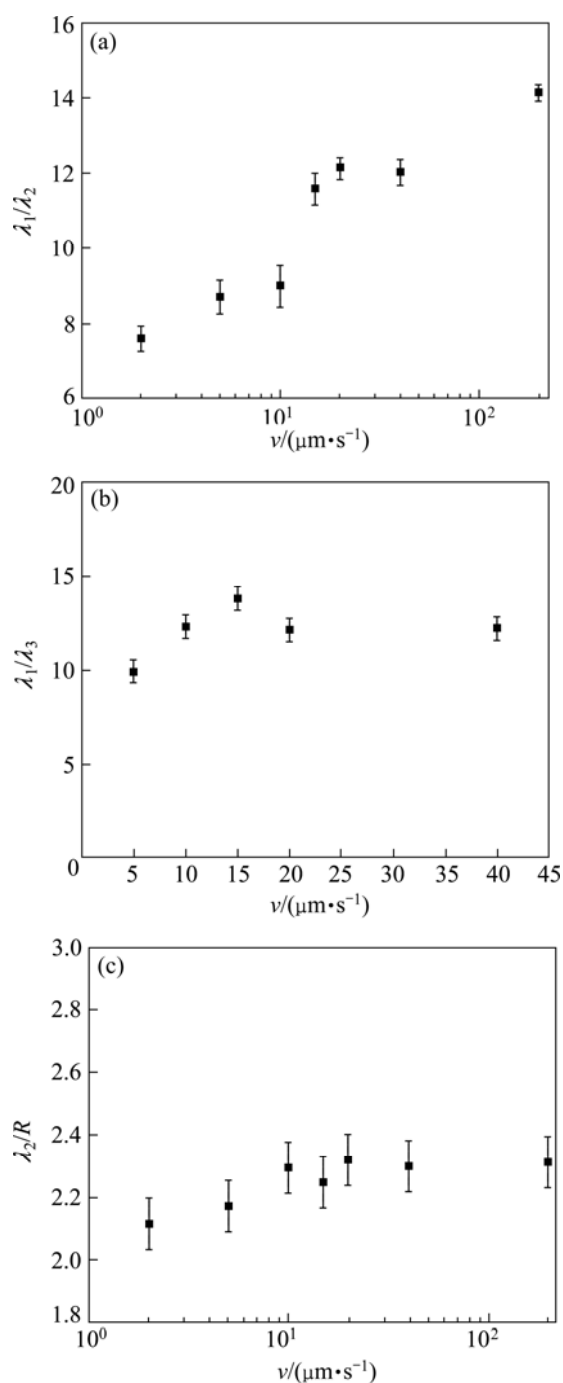


Fig. 8 Relation between characteristic length scales: (a) λ_1/λ_2 , (b) λ_1/λ_3 , (c) λ_2/R

λ_1/λ_3 does not remain constant but reaches its maximum at the growth rate of $15 \mu\text{m/s}$. This can be attributed to the initiation of tertiary dendrite arm which results from adjustment of primary dendrite arms. At lower growth rate, in order to reduce the undercooling between primary dendrite arms, tertiary dendrite arms originate from secondary dendrite arms. Since λ_2 decreases faster than λ_1 , λ_3 also decreases faster than λ_1 . But when the growth rate is above a threshold, according to the branching mechanism of primary dendrite arm, the interdendritic space for primary dendrite arm branching

is limited, thus λ_1/λ_3 has no significant change. And from this it can be found that despite the fact that tertiary arms develop from secondary branches, they have a growth characteristic similar to the primary dendrite arms.

A numerical analysis of the wavelength of instabilities along the sides of a dendrite was carried out by LANGER and MÜLLER-KRUMBHAR [23] and they predicted a scaling law as $\lambda_2/R \approx 2$. The results of numerous studies showed that although in most cases the λ_2/R might be constant, its value is different in different alloy systems, and ranges from 2 [24] to 4.86 [31]. It can be found from our experimental results in Fig. 8(c) that λ_2/R is not constant but ranges from 2 to 2.3 with the increase of growth rate.

4 Conclusions

1) For a given C_0 of Sn–Ni peritectic alloy, the values of λ_1 , λ_2 , λ_3 and dendrite tip radius R decrease as the growth rate increases. The relations between the microstructural parameters and the solidification parameters were obtained by linear regression analysis as: $\lambda_1 = 335.882v^{-0.21}$, $\lambda_2 = 44.957v^{-0.277}$, $\lambda_3 = 40.512v^{-0.274}$, $R = 22.7v^{-0.36}$.

2) The λ_1/λ_2 is not constant but ranges from 7.6 to 14.1 with the increase of growth rate; the λ_1/λ_3 does not remain constant but reaches its maximum at the growth rate of $15 \mu\text{m/s}$; the λ_2/R ranges from 2 to 2.3 with the increase of growth rate.

3) It can be observed from the variation of λ_1/λ_2 and λ_2/R with growth rate that the secondary dendrite arm spacing λ_2 is more sensitive to solidification parameters compared with primary dendrite arm spacing λ_1 . The variation of λ_1/λ_3 shows that tertiary dendrite arm has a similar growth characteristic to primary dendrite arm.

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定向凝固 Sn–36%Ni 包晶合金凝固组织特征长度的表征

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摘 要：通过对 Sn–36%Ni 包晶合金在恒定温度梯度($G=20\text{ K/mm}$)下进行的一定速度范围内($v=2\sim 200\text{ }\mu\text{m/s}$)的定向凝固实验，研究凝固组织特征尺度随生长速度 v 的变化；测量包括一次与高次枝晶间距及枝晶尖端半径在内的凝固组织特征尺度。通过实验结果与理论模型的对比，发现凝固组织特征尺度随生长速度的变化关系为：对于一次枝晶间距有 $\lambda_1=335.882v^{-0.21}$ ，且与 Kurz–Fisher 模型吻合；对于二次枝晶间距有 $\lambda_2=44.957v^{-0.277}$ ，且与 Bouchard–Kirkaldy 模型吻合；对于三次枝晶间距有 $\lambda_3=40.512v^{-0.274}$ ；对于枝晶尖端半径有 $R=22.7v^{-0.36}$ 。实验结果表明， λ_1/λ_2 随着生长速度的增加而增加， λ_1/λ_3 的变化明显较 λ_1/λ_2 的小，表明三次枝晶具有与一次枝晶类似的生长特征；而 λ_1/R 的比值随着生长速度的增加而由 2 增加到 2.3，变化很小。

关键词：Sn–Ni 合金；定向凝固；枝晶间距；枝晶尖端半径

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