Modification of Hypoeutectic Al–Cu, Al–Si and Al–Ni Alloys by Rheocasting*

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An investigation was made for the improvement of microstructures and mechanical properties at an elevated temperature in Al–Cu alloys containing 10, 24, 30 mass% copper, Al–8 mass% Si and Al–4 mass% Ni alloys rheocast with the rotation of a stirrer at a high speed in the range from 20 to 67 rev/s. It was found that the relation among the size, \( d \), of primary solid particles formed by fragmentation of dendritic crystals, copper content, \( C_0 \), and rotation speed of stirrer can be expressed by the following empirical equation:

\[ d = K (33 - C_0)^n \]

for \( n = 0.32 \), where \( K \) is a constant determined by the stirrer speed. The apparent viscosity of Al–Cu alloys was evaluated during the solidification with high-speed rotation of stirrer. Rheocast microstructures in Al–8%Si and Al–4%Ni alloys are compared with those in Al–Cu alloys on the basis of microscopic observations. The maximum stress and total elongation of the Al–30% alloy ingot rheocast at a rotation speed of 33 s\(^{-1}\) are 24.0–28.5 MPa and 71%, respectively, while those of the Al–24% alloys are 19.7–21.2 MPa and 63–86% at 33 s\(^{-1}\) and 21.2–22.7 MPa and 79–91% at 67 s\(^{-1}\), respectively.

(Received January 30, 1988)

Keywords: rheocasting, stir-casting, aluminum-copper alloy, aluminum-silicon alloy, aluminum-nickel alloy, primary solid particle, grain refinement, apparent viscosity, elevated temperature tensile test, elongation

I. Introduction

Alloy design methods are generally constructed for conventionally cast, directionally solidified columnar, single crystal and powder metallurgy processed alloys. However, no alloy design method has been developed for the improvement of microstructures and mechanical properties of alloys produced by rheocasting, because very few work is available in the literature which is useful for predicting a new material. It should be thus necessary to construct an alloy design method for the rheocast alloys because the rheocasting process has some great advantages. The advantages are to reduce manufacturing costs compared with the powder metallurgy process, to produce a homogenized and finely grained alloy only by casting, to improve material characteristics, to make the semi-solid alloy slurry into near net shape components directly and so on. Several fundamental studies(1)-(4) have been made on the solidification with a rotation of the stirrer at high speeds above 20 s\(^{-1}\) in order to construct an alloy design by rheocasting.

The first approach to constructing the alloy design in rheocast alloys is to determine the solidification parameters, i.e. the rotation speed of stirrer, the cooling rate during solidification, the size of primary solid particles formed by destruction of dendritic crystals, the solute content, the viscosity of semi-solid alloy, the amount of grain refiners, etc. The second approach to constructing the alloy design in rheocast alloys is to determine the mechanical properties, i.e. tensile strength, elongation, reduction of area, hardness, etc.

In the present work the relations among the size of primary solid particles, the solute content and the rotation of stirrer have been investigated in order to derive an empirical equation on rheocasting. The apparent viscos-
ity in Al–Cu alloy solidifying with rotation of the stirrer and the effect of grain refiners on microstructures are evaluated. Rheocast microstructure in Al–Cu alloy is compared with that in other rheocast microstructures in Al–Si and Al–Ni alloys. Tensile tests are carried out on the rheocast alloys to examine the relationship between the microstructures and the mechanical properties.

II. Experimental Procedure

The outline of an experimental apparatus used in this work is shown in Fig. 1. A chamber provided thereof in the front panel of a door for permitting insertion of a crucible into the chamber and inspection of the interior of the chamber constitutes a vacuum container. The interior of the chamber is partitioned into upper cooling and lower heating rooms by a shutter, which is made of molybdenum and designed to be opened and closed by an air cylinder. Inside the lower heating room is disposed a resistance heating furnace of molybdenum in which a graphite crucible on a support bar so as to be movable up and down automatically. The upper cooling room has a water-cooling outer tube therein and a graphite stirring bar is suspended downwardly into the water-cooling outer tube. The stirring bar has a generally square cross-section and tapered 0.030 m at the upper end to 0.025 m at the lower end, with the four corners cut as illustrated in Fig. 2. This stirring bar is so constructed that it may be rotated at the superhigh speed up to 75 s⁻¹ by a motor provided at the upper end thereof. The motor is provided on the rotary shaft with a torque detector and a rotation detector connected with a digital unit for displaying and recording both the torque of the stirring bar and the rotational speed to be detected simultaneously. A recorder attached to the aforementioned apparatus is also operated to record the changing cooling temperature in a continuous curve.

The experimental procedure is as follows. First of all, 99.99 mass% pure aluminum blocks together with 99.99 mass% pure copper plates, 99.97 mass% pure nickel globules or 98 mass% pure silicon granules are placed in the crucible. Then the chamber is evacuated with a vacuum pump and the shutter over the heating furnace is closed to heighten the efficiency of heating. When the degree of vacuum in the chamber attains 1 × 10⁻³ Pa, the material in the crucible is heated to produce a molten Al–10 mass%Cu, Al–24 mass%Cu,
Al-30 mass%Cu, Al-8 mass%Si, Al-4 mass%Ni alloy weighing 0.5 kg. After the alloy material in the crucible has been thoroughly melted at 100 K above the equilibrium liquidus temperature of the alloy, the shutter over the furnace is opened and the support bar supporting the crucible by the bottom thereon is raised by an elevating mechanism until the crucible is located inside the water-cooling outer tube. As a result, the stirring bar is gradually inserted, with the forward end thereof in the lead, into the molten material in the crucible until the leading end of the stirring bar reaches a distance of 0.01 m from the bottom wall of the crucible. At this time, the length of the stirring bar immersed in the molten alloy is about 0.1 m. Then, flow of cold water through the water-cooling outer tube is started to effect cooling of the molten alloy at a rate of 0.42 K/s, while the stirring bar is kept rotated at a low speed of 9 s⁻¹. The rotational rate of the stirring bar is raised in 10 s to a high speed in the four stages of 20, 33, 50 and 67 s⁻¹ after the alloy material has substantially reached the temperature for starting solidification in recorded cooling curve and torque. In this case, the rotational speed is increased at a fixed rate so as to prevent the semi-solid alloy from being scattered in consequence of a sharp increase in rotation speed. The rotational stirring at the fixed speed indicated above is continued until immediately before the temperature for completion of solidification is confirmed by the cooling curve of the recorder and the torque value on the digital display device. Then the support bar supporting the crucible is lowered by the falling mechanism until the leading end of the stirring bar reaches a distance of 0.20 m from the bottom wall of the crucible in order to prevent the stirring bar from adhering to the rheocast alloy ingot. As a result, dendritic crystals inherently formed by the alloy are finely divided into many primary solid particles.

A metallurgical observation is made in the specimens cut from the rheocast alloy ingot by optical and scanning electron microscopes. The size of primary solid particles is determined on the microphotograph by an automatic image analysis system.

**Fig. 3** Size and shape of test piece used for the present work.

Tensile test pieces are also machined from the rheocast alloy ingot as shown in Fig. 3. In the present tensile condition the test temperature and strain rate are 773 K and 1.19 × 10⁻³ s⁻¹.

### III. Experimental Results and Discussion

#### 1. Evaluation of apparent viscosity during the solidification with stirring

In order to evaluate quantitatively the viscous flow of Al–Cu alloys solidifying with the rotation of stirring bar, the value of apparent viscosity is calculated by the following equation⁹(6):

$$\eta = M(1 - \alpha^2) / 4\pi r^2 LR$$  \hspace{1cm} (1)

where $\eta$ is the apparent viscosity, $M$ is the torque, $\alpha$ is the ratio of the diameter of stirrer (practically the major axis at the transverse section of stirrer) to the inner diameter of crucible, $r$ is the radius of the stirrer (practically the half-value of major axis), $L$ is the length of stirrer immersed in the melt and $R$ is the rotation speed of the stirrer. Equation (1) is derived on the basis of laminar flow of an incompressible fluid in the space between two coaxial cylinders, the inner one of which is rotating with an angular velocity $R$. Thus fundamental errors must originate to some extent in the calculation of apparent viscosity by using eq. (1), because the stirrer used in the present experiment is not a rounded inner cylinder.

The effect of initial solute concentration on the apparent viscosity induced by the rotation of stirring bar is compared in Al–10%Cu, Al–24%Cu and Al–30%Cu alloy. As a result, a variation in apparent viscosity during the solidification of these alloys with the rotation...
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of stirring bar at a high speed of 33 s\(^{-1}\) is shown in Fig. 4. It is found that the level of apparent viscosity values in the initial stage of solidification increases with increasing initial solute concentration of the alloy. The apparent viscosity in Al–10%Cu alloy solidifying with the rotation of stirrer at a speed of 33 s\(^{-1}\) starts at about 7 Pa·s in the initial stage of solidification and is rapidly increased from the middle to the latter stage of solidification. But the apparent viscosity in Al–24%Cu alloy solidifying with the rotation of the stirrer at a speed of 33 s\(^{-1}\) remains almost unchanged at a constant value of 13 Pa·s during solidification. The apparent viscosity in Al–30%Cu alloy solidifying with the rotation of the stirrer at a speed of 33 s\(^{-1}\) is also decreased from a value of about 19 Pa·s to a value of 16 Pa·s during solidification after a rapid fluctuation at the initial stage of solidification. The difference in apparent viscosity during the solidification of Al–10%Cu, Al–24%Cu and Al–30%Cu alloys with the rotation of the stirrer at a speed of 33 s\(^{-1}\) is considered as follows. The level of apparent viscosity in the initial stage of solidification is mainly determined by the chemical composition, i.e. the solute content, in the Al–Cu alloy. However, the value of apparent viscosity in the middle and latter stages of solidification must depend on whether or not the coalescence of primary solid particles formed by fragmentation of dendritic crystals is accomplished as well as the chemical composition. The amount and size of primary solid particles formed just before the start of formation of eutectics and the freezing range in the phase diagram of Al–Cu alloy are decreased with increasing initial solute concentration. The decrease in the amount and size of primary solid particles and in the freezing range with increasing from 10 to 30%Cu implies the decrease in the degree of coalescence of primary solid particles during the violent flow of the solidifying alloy. Therefore the apparent viscosity is rapidly increased with the coarsening and coalescence of primary solid particles in the middle stage of solidification in the Al–10%Cu alloy, and is gradually increased with the coarsening and local coalescence in the latter stage of solidification in Al–24%Cu alloy. But the apparent viscosity in Al–30%Cu alloy remains almost unchanged without the coalescence of primary solid particles except a rapid change in the initial stage of solidification. The influence of the rotation speed of the stirrer in the Al–Cu alloy on the apparent viscosity induced by the rotational stirring is also investigated. As a result, a variation in apparent viscosity from the start to just before the end of solidification with the rotation speeds of 33, 50 and 67 s\(^{-1}\) in the Al–30%Cu alloy is shown in Fig. 5. The apparent viscosity values in the Al–30%Cu alloy solidifying with the rotation of stirrer at high speeds is almost unchanged during the solidification of the alloy except a rapid change at a rotation speed of 33 s\(^{-1}\) and a gradual increase at a speed of 50 s\(^{-1}\) in the initial stage of solidification. It is clearly recognized that the level of them decreases with increasing rotation speed of stirrer. As described above, the solidification of the Al–30%Cu alloy at speeds of 33 to 67 s\(^{-1}\) must proceed without the coalescence of primary solid particles. The rotating stirrer cannot be thoroughly wetted even at the minimum speed of 33 s\(^{-1}\) in the present study in the latter

Fig. 4 Variation in apparent viscosity during the rotation of stirrer at a speed of 33 s\(^{-1}\) from the start of solidification in Al–10%Cu, Al–24%Cu and Al–30%Cu alloy ingots.
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and final stages of solidification by the flowing Al-30%Cu alloy because the flow velocity of the semi-solid alloy is significantly decreased with the formation of eutectics. Therefore it is considered that the apparent viscosity in the Al-30%Cu alloy cannot be increased in the final stage of solidification.

2. Relation among solute content, rotation speed of stirrer and primary solid particle size

The sizes of primary solid particles in the Al-10%Cu, Al-24%Cu and Al-30%Cu alloys rheocast at a high rotation speed are evaluated by using an automatic image analysis system. As a result, the sizes of primary solid particle formed during solidification with the rotation of stirrer in Al-10%Cu, Al-24%Cu or Al-30%Cu alloy ingot are 101±31, 87±28 or 54±19 μm at a stirrer speed of 33 s⁻¹, 98±34, 75±30 or 52±21 μm at a stirrer speed of 50 s⁻¹ and 90±29, 61±32 or 46±14 μm at a stirrer speed of 67 s⁻¹. The relation between the average value of primary solid particles, 𝑑, and the solute content, 𝐶₀, at different rotation speeds of stirrer is represented in Fig. 6. The solid line is also drawn under the following relation:

\[ d = K (33 - C₀)^n \quad \text{for} \ n = 0.32 \quad (2) \]

where \( K \) is a constant determined by the rotation speed of stirrer \( R \), i.e. \( K = 110 R^{-0.29} \), and the initial solute concentration in Al-Cu eutectic alloy is 33 mass% copper. The calculated curves are consistent with the observed values at rotation speeds of stirrer above 50 s⁻¹, though some errors are seen at a low speed of 33 s⁻¹. Thus it is recognized that eq. (2) holds true. Therefore it is possible to estimate the size of primary solid particles in Al-Cu alloy ingots rheocast at a high rotation speed in the range of the solute contents from 10 to 33 mass% copper.

3. Rheocast microstructures in hypoeutectic alloy

In order to compare the rheocast microstructure in Al-24%Cu alloy with that in other hypoeutectic aluminum alloys, Al-8%Si and Al-4%Ni alloys are chosen under the same condition that the ratio of the amount of primary crystals to that of eutectic crystals is 1:2 on an equilibrium phase diagram, assuming that the solidification of these alloys with rotation of stirrer at a high speed of 67 s⁻¹ proceeds according to the phase diagram (see Appendix). Optical micrographs in Al-24%Cu, Al-8%Si and Al-4%Ni alloy ingots rheocast at a rotation speed of 67 s⁻¹ are represented in Fig. 7. Figure 7(a) is a rheocast microstructure in the Al-24%Cu alloy, in which the size of primary
solid particles is $61 \pm 32 \mu m$. Figure 7(b) is a rheocast microstructure in the Al-4\%Ni alloy, in which the primary solid particle size is $114 \pm 38 \mu m$. Figure 7(c) is a rheocast microstructure in the Al-4\%Ni alloy, in which the primary solid particle size is $118 \pm 40 \mu m$. Even if the rotation speed of stirrer and cooling rate during solidification were controlled under the same condition, the primary solid particle sizes in the Al-4\%Ni and Al-8\%Si alloys with lesser solute content are much larger than the particle size in the Al-24\%Cu alloy. It is then recognized that the relation among the size of primary solid particles, $d$, the solute content, $C_0$, and the freezing range, $F$, in Al-based alloy is expressed by the following empirical equation:

$$d = \beta (F/C_0)^m \text{ for } m = 2/3$$

where $\beta$ is an experimental constant determined to be $51 \mu m \cdot (\text{mass}\% / K)^{2/3}$ and $F$ is $31 K$ in Al-24\%Cu, $27 K$ in the Al-8\%Si or $14 K$ in the Al-4\%Ni alloy.

4. Effect of grain refiners on microstructure

Vacuum-melted Al-Cu alloys are violently agitated during solidification with increased addition of grain refiners of titanium and boron at high rotation speeds above $20 s^{-1}$. As a result, scanning electron microphotographs in Al-24\%Cu-1\%Ti-0.2\%B alloy ingots rheocast at rotation speeds of 20 and $50 s^{-1}$ are shown in Fig. 8. Black circles in this figure are primary solid particles and lamellar zones are eutectics. The size of primary solid particles in the alloy ingot rheocast at a rotation speed of $20 s^{-1}$ as shown in Fig. 8(a) is $52 \pm 20 \mu m$. As this value is half the size of primary solid particles, $94 \pm 34 \mu m$, in Al-24\%Cu alloy ingot rheocast at $20 s^{-1}$, the primary solid particles are considerably refined with addition of grain refiners. The primary solid particle size in Al-24\%Cu-1\%Ti-0.2\%B alloy rheocast at $50 s^{-1}$ as shown in Fig. 5(b) is $51 \pm 19 \mu m$. As it is small compared with the particle size of $75 \pm 30 \mu m$, the refining effect of addition of grain refiners on the microstructure is recognized.

Furthermore, the size of primary solid par-
5. Verification of high ductility in rheocast alloys

Vacuum-melted Al–Cu alloys are rheocast at a high rotation speed in the range from 33 to 67 s⁻¹. Test pieces are prepared from these rheocast alloy ingots as shown in Fig. 3. The tensile test is carried out under the following conditions. The test temperature is 773 K and the strain rate is 1.19 × 10⁻³ s⁻¹. As a result, the maximum stress and total elongation values in the rheocast Al–Cu alloys are also represented in Table 1. The maximum stresses in Al–30%Cu alloy rheocast at a rotation speed of 33 s⁻¹ show a value above 24 MPa and are large as compared with those in the Al–24%Cu alloy rheocast at 33 s⁻¹. However, the total elongation in the Al–30%Cu alloy rheocast at a rotation speed of 33 s⁻¹ is 71% and is approximately equal to that in the Al–24%Cu alloy rheocast at 33 s⁻¹. It is found that the total elongation increases with decreasing maximum stress in Al–24%Cu alloy rheocast at a rotation speed of 33 s⁻¹. It is also recognized that the total elongation in the Al–24%Cu alloy rheocast at a rotation speed of 67 s⁻¹ is larger than that in Al–24%Cu alloy rheocast at 33 s⁻¹. It is considered that the increase in total elongation with increasing rotation speed of the stirrer is largely responsible for the refinement of primary solid particles as shown in Table 1.

Microstructural observations are made on a longitudinal cross section of the test piece, which has been elongated to be a value of 86% and ruptured, in the Al–24%Cu alloy rheocast at a rotation speed of 33 s⁻¹. As a result, scanning electron micrographs at a distance of 0, 0.0015, 0.0030, 0.0045, 0.0060, 0.0080, 0.0100 and 0.0140 m from the ruptured position of the test piece are shown in Fig. 9. Spherical primary solid particles are completely transformed into elongated rods in the vicinity of the rupture.
of ruptured position and at a distance of 0.0015 m from the ruptured position. It is also observed that the rupture starts from hard plate-like θ phases. They are changed mostly into elongated rods and partly to elongated particles at a distance of 0.030 and 0.045 m from the ruptured position. Elongated particles are observed to be arranged along the tensile axis at a distance of 0.060 and 0.080 m from the ruptured position. The primary solid particles are almost unchanged to be spherical at a distance of 0.0100 and 0.0140 m from the ruptured position even after the elevated temperature tensile testing. Accordingly it is confirmed that the strain rate of $1.19 \times 10^{-3} \text{s}^{-1}$ is too fast for the test piece of the Al-24%Cu alloy rheocast at a rotation speed of 33 s$^{-1}$ to deform superplastically, because the average size of primary solid particles in the alloy is greater than 10 μm. However, it is possible that superplasticity will manifest itself with decreasing strain rate.

IV. Conclusion

Vacuum-melted hypoeutectic Al–Cu, Al–Si and Al–Ni alloys are vigorously agitated from the start to just before the end of solidification with rotation of a stirrer at fixed speeds from 20 to 67 s$^{-1}$ during continuous cooling at a rate of about 0.4 K/s inside the water-cooling outer tube, in order to obtain an ultra-finely grained material capable of manifesting superplasticity. The effects of viscous flow of solidifying alloy and rotation speed of stirrer on the size of primary solid particles originated by fragmentation of dendritic crystals and the refinement of eutectics during the rotational stirring are investigated. High ductility is also examined on the tensile test of these rheocast alloys at elevated temperatures.

(1) The apparent viscosity in the solidification with rotation of stirrer at a speed of 33 s$^{-1}$ starts at about 7 Pa·s in the initial stage of solidification and is rapidly increased from the middle to the latter stage of solidification in the Al–10%Cu alloy. But it remains almost un-
changed at a constant value of 13 Pa·s during solidification in the Al-24%Cu alloy. The apparent viscosity is also decreased from a value of about 19 Pa·s to about 16 Pa·s during solidification after a rapid fluctuation at the initial stage of solidification in the Al-30%Cu alloy. The apparent viscosity in the solidification with rotation of the stirrer at a speed of 50 or 67 s⁻¹ in the Al-30%Cu alloy remains almost unchanged at a value of about 10 or 5 Pa·s.

(2) It is found that the relation among the primary solid particle size, d, the copper content, C₀, and the stirrer speed can be expressed by the following empirical equation:

\[ d = K (33 - C₀)^n \quad \text{for } n = 0.32 \]

where \( K \) is a constant determined by stirrer speed \( R \), i.e. \( K = 110R^{-0.29} \).

(3) It is recognized that the relation among the primary particle size, d, the solute content, C₀, and the freezing range, F, in Al-based alloys is represented as follows:

\[ d = \beta (F/C₀)^m \quad \text{for } m = 2/3 \]

where \( \beta \) is a constant determined to be 51 \( \mu \text{m} \cdot (\text{mass} \%/K)^{2/3} \).

(4) The sizes of primary solid particles in the Al-24%Cu alloy ingot rheocast at the rotation speeds of 20 and 50 s⁻¹ with the addition of grain refiners of 1%Ti and 0.2%B are 52±20 and 51±19 \( \mu \text{m} \), respectively.

(5) Tensile tests are made on rheocast Al-Cu alloys under the condition of the test temperature of 773 K and the strain rate of 1.19×10⁻³ s⁻¹. The maximum stress and total elongation of the Al-24%Cu alloy rheocast at a rotation speed of 33 s⁻¹ are 19.7–21.2 MPa and 63–86%, respectively, while those of the alloy at 67 s⁻¹ are 21.2–22.7 MPa and 79–91%, respectively. The maximum stress and total elongation of the Al-30%Cu alloy rheocast at a rotation speed of 33 s⁻¹ are 24.0–28.5 MPa and 71%, respectively.

Acknowledgment

This work was performed as a part of the R & D Project of Basic Technology for Future Industries sponsored by Agency of Industrial Science and Technology, MITI.

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Appendix

Application of solidification with rotational stirring to equilibrium phase diagram

Conservation of solute in a small volume element of half the secondary arm spacing during conventional solidification is expressed as follows(7):

\[ f_s C_s + f_L C_L = C_0 \]  \( (1) \)

where \( f_s \) and \( f_L \) are the fractions of solid and liquid within the volume element, \( C_s \) and \( C_L \) are the local average solute concentrations in solid and liquid within the volume element, and \( C_0 \) is the initial solute concentration in liquid.

The solute concentration in liquid within the volume element is expressed by the following equation(8),

\[ C_L = C_0\left[1 - (1 - 2\gamma k_0)f_s\right]^{(k_0 - 1)(1 - 2\gamma k_0)} \]  \( (2) \)

where \( k_0 \) is the equilibrium distribution coefficient \( (0 < k_0 < 1) \). The solidification parameter, \( \gamma \), is expressed as follows:

\[ \gamma = D_s \theta_f / l^2 \quad \text{for } 0 < \gamma \leq 0.5 \]  \( (3) \)

where \( l \) is half the secondary arm spacing, \( D_s \) is the diffusivity of solute in solid and \( \theta_f \) is the local solidification time.

Substituting eq. (2) into eq. (1), the local average solute concentration in solid within the volume element is rewritten as follows:
The primary solid particles are formed and suspended in the remaining liquid by the fragmentation of dendritic crystals caused by rotational stirring. When the remaining liquid is perfectly mixed by the violent stirring in the present experiment, i.e. $\psi = 0.5$, the solute concentration in liquid within the volume element is expressed as follows:

$$C_L = \frac{C_0}{1 - (1 - k_0)\psi}. \quad (5)$$

And the average solute concentration in solid within the volume element is represented by the following equation:

$$\bar{C}_s = C_0 \frac{1 - (1 - f_s)}{\{1 - (1 - k_0)\psi\}^{(k_0 - 1)(1 - 2k_0)}}. \quad (4)$$

Assuming that the value of $C_L$ in eq. (4) increases from the $C_0$ value to the value of $C_0/k_0$ on the liquidus of the equilibrium phase diagram with the advance of solidification, the value of $C_s$ also increases from the value of $k_0C_0$ at the start of solidification to the $C_0$ value at the end of solidification on the solidus of the phase diagram. In other words, it is considered that the solidification with rotation of stirrer at super-high speeds proceeds according to the equilibrium phase diagram.