Pulverization of Molten Sn and Sn-Cu by a Bladed Rotator Method*

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Synopsis

In present work, molten Sn and Sn-Cu have been pulverized by an impact atomizer which has a bladed rotator. The powder properties, especially mean particle size, specific surface area and particle shape, have been related with the operating conditions for atomization. The relations give the informations for the size and shape controls of the powder particles on the atomizing method.

Key words: powder fabrication; atomization; bladed rotator; metallic powder; operating condition; water curtain; particle size distribution; powdery shape; specific surface area; dimension analysis; spherification; solidification.

I. Introduction

Atomization method is one of the most popular technics for the pulverization of molten metals and alloys. According to the types of atomizing media and principles applied, the method is classified into three categories; water or oil, gas, and various centrifugal atomizations.

To fully specify the fabrication methods, it is necessary to know the characteristic properties of powder products, such as the particle size distribution, the shape, the microstructure, *etc.* It is also desirable that these properties can be independently controlled in the process. The present work, therefore, aims at seeking the possibility of the independent control. For pulverization an impact atomizing way was devised with a bladed rotator. This bladed rotator method is, in principle, of the impact atomizations,^{1,2)} but has a particular way where molten metal is ejected toward the centripetal direction from a nozzle settled over an uprightly rotating disk and atomized with the blades of the disk. Particular interest is concerned in the effects of the operating conditions on the powder properties, especially the size distribution and particle shape.

II. Experimental Procedure

1. Pulverization Apparatus and Procedure

Figure 1 is a schematic illustration of the pulverization apparatus used in the present work. It consists of three parts; for melting and ejecting the metallic sample, atomizing the molten metal, and collecting the powder.

Figure 2(a) shows the one for melting and ejecting. A metallic sample (Sn or Sn-Cu) of about 50 g was put in a quartz tube having a nozzle on the bottom.

The nozzle hole was closed with an alumina stopper. The other end of the tube was sealed up with a brass cap. Under the atmosphere of an Ar stream, the sample was heated up to a given temperature and melted in a Ni-Cr wire resistance furnace. The temperature of the molten metal was detected with a thermocouple in the nozzle stopper tube. After the pressure of the atmosphere had been increased up to 0.16 MPa, the melt sample was ejected down through the nozzle to the atomizing device that was settled below the nozzle.

Atomization was carried out by collision of the molten metal with the blades of an uprightly rotating disk. A mild steel-made disk with a diameter of 80 mm and width of 5 mm has thirty six or twelve flat blades with a height of 10 mm and thickness of 2 mm. The blades are set in an equal interval on the periphery of the disk which is rotationally driven by an electric motor. The distance between the nozzle end and motor shaft was about 55 mm. The disk revolution was controlled by adjusting the voltage of the motor and detected with a photomicro sensor. The atomization was observed on the pictures which were taken by flashing light for about 4 μ s with a strobe.

The powdery particles atomized were splashed forward against and carried downward with a water curtain or a filmy water flow, then collected in a water pool. The curtain was formed of water free falling through a slit of 100 mm long and 4 mm wide, and settled at a horizontal distance of about 120 mm apart from the nozzle position.

Fig. 1. Schematic diagram of the pulverizing apparatus.

* Manuscript received on September 22, 1987; accepted in the final form on January 14, 1988. © 1988 ISIJ

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After rinsed in methyl alcoho and dlried, the collected powder was submitted to sieve analysis and scanning electron microscope (SEM) observation.

2. Sample Materials and Operating Conditions

The materials used are granular Sn (purity 99.8 %) and fragmentary Sn–Cu alloys. The latter was prepared as follows. After the pure Sn and pure Cu (purity 99.85 %) were mixed in given proportions, the mixtures were premelted by high-frequency induction heating. The ingots obtained were cut off fragmentarily as the Sn–Cu samples.

The variables mainly selected in the present work were 4 independent operating conditions; the initial temperature of melt (T_i), the central angle between blades (θ_b), the peripheral velocity of rotating disk (V_d) and the diameter of nozzle hole (D_n). The ejecting velocity (V_e), that is the stream rate through the nozzle, depends on the diameter of the nozzle hole and the properties of the melt at the temperature, under the constant ejecting pressure. These operating conditions are summarized in Table 1 together with the sample materials.

Fig. 2. Assembly of the melt ejector (a), and the bladed disk (b).

III. Results

1. Atomization

Atomizing scenes of the ejected melt (sample No. 3) are shown in Fig. 3; (a) before atomizing, (b) just atomizing, and (c) after atomizing. These pictures indicate that:

- (1) The molten metal stream is disintegrated into acicular droplets at a moment of collision with a blade.
- (2) According to positional relationships between main groups of flying droplets and blades, the flying speeds of the droplets can be estimated to be nearly equal to the peripheral velocity of the rotating disk.

2. Particle Size Distribution

Attention has been given to the size distributions of the powders produced under the operating conditions. Figures 4 to 7 show the relationships between the size distribution *vs.* the initial temperature of the melt, the central angle between the blades, the peripheral velocity of the rotating disk or the diameter of the nozzle hole, respectively. The effects of the operating conditions are summarized as follows.

- (1) The initial temperature of the melt has no significant effect on the particle size distribution. But the higher the temperature is, the smaller the mean particle size.
- (2) The central angle of 30° between the blades leads to smaller mean particle size and narrower size distribution than that of 10°, but this effect is relatively small.
- (3) The peripheral velocity of the rotating disk has a large effect on the distribution. The higher the velocity is, the smaller the mean particle size and the narrower the distribution width.
- (4) For the high peripheral velocity, the diameter of the nozzle hole has a strong effect on the distribution. The smaller diameter results in the smaller mean particle size and the narrower width of the distribution.

Table 1. Sample materials and operating conditions.

Sample No.	Sample materials	Operating conditions				
		Independent operating conditions				V_e (m·s ⁻¹)
		T_i /(K)	θ_b (deg (rad))	V_d (m·s ⁻¹ (r.p.m.))	D_n (10 ⁻³ m)	
1	Sn	873	10 ($\pi/18$)	41.89 (8 000)	0.5	6.15
2	Sn	873	10 ($\pi/18$)	41.89 (8 000)	1.0	4.19
3	Sn	873	30 ($\pi/6$)	41.89 (8 000)	0.5	6.15
4	Sn	873	10 ($\pi/18$)	10.47 (2 000)	0.5	6.15
5	Sn	873	10 ($\pi/18$)	10.47 (2 000)	1.0	4.19
6	Sn	673	10 ($\pi/18$)	10.47 (2 000)	1.0	4.99
7	Sn	673	10 ($\pi/18$)	41.89 (8 000)	1.0	4.99
8	Sn	673	10 ($\pi/18$)	10.47 (2 000)	0.5	7.27
9	Sn	673	10 ($\pi/18$)	20.94 (4 000)	0.5	7.27
10	Sn	673	10 ($\pi/18$)	31.42 (6 000)	0.5	7.27
11	Sn	673	10 ($\pi/18$)	41.89 (8 000)	0.5	7.27
12	Sn-20 wt%Cu	973	30 ($\pi/6$)	41.89 (8 000)	0.5	8.49
13	Sn-20 wt%Cu	1 173	30 ($\pi/6$)	41.89 (8 000)	0.5	7.51
14	Sn-60 wt%Cu	1 023	30 ($\pi/6$)	41.89 (8 000)	0.5	14.33

Fig. 3. Atomization of the ejected melt; (a) before atomizing, (b) just atomizing and (c) after atomizing.

Fig. 4. Effect of the initial temperature of the melt on the particle size distribution under the different peripheral velocities of the rotating disk.

3. Powdery Shape

The SEM photographs reveal the aspects of the particles obtained. Figure 8 indicates the shapes in

Fig. 5. Effect of the central angle between the blades on the particle size distribution.

Fig. 6. Effect of the peripheral velocity of the rotating disk on the particle size distribution.

Fig. 7. Effect of the diameter of the nozzle hole on the particle size distribution under the different peripheral velocities of the rotating disk.

the three size ranges of the tin particles (sample No. 3). They are of non-spherical in all the size ranges. In the sample of Sn-20 wt%Cu (sample No. 12), the fine size group consists of the nearly spherical particles, while the coarse group the irregular ones, as shown in Fig. 9. The powders of Sn-60 wt%Cu (sample No. 14) have the nearly spherical shapes in every size range according to Fig. 10.

IV. Discussion

1. Relations between Powder Properties and Experimental Conditions

1. Mean Particle Size and Peripheral Velocity of Rotating Disk

The mean particle size of the powder is closely sub-

Fig. 8. Observations of the powdery shapes in three size ranges by scanning electron microscopy (sample No. 3).

Fig. 9. Observations of the powdery shapes in three size ranges by scanning electron microscopy (sample No. 12).

Fig. 10. Observations of the powdery shapes in three size ranges by scanning electron microscopy (sample No. 14).

ject to the peripheral velocity of the rotating disk, as shown in Fig. 6. An attempt is made to interrelate them quantitatively. Figure 11 indicates the relations by the logarithmic plot, where $\bar{D}_p$ is the cumulative 50 wt% particle size (μm) and V_d the peripheral velocity of the rotating disk ($\text{m}\cdot\text{s}^{-1}$). Solid line A shows the relation obtained from the data of sample

Nos. 8 to 11 in this work. Solid line B is of the result which Katoh *et al.*³⁾ have obtained by the centrifugal atomization of Ni-base superalloy. Ohnaka *et al.*¹⁾ proposed the relation of the line C by the impact atomization of $\text{Fe}_{40}\text{Ni}_{40}\text{B}_{20}$ alloy, where the molten alloy jet was atomized with a toothed-disk rotating horizontally. In spite of difference in the materials

Fig. 11. Relations between the mean particle size and the peripheral velocity of the rotating disk.

and atomizing apparatus, all the three results indicate essentially the straight lines having almost the same slope as shown in Fig. 11. This might imply similarity of the atomization mechanism. The following equation for the above relation is obtained by the least square method in this work.

$$\bar{D}_p = 3.6 \times 10^{-3} \cdot V_d^{-0.89} \dots\dots\dots(1)$$

2. Specific Surface Area and Experimental Conditions

As a property with respect to the particle size, the size distribution is an important concept as well as the mean value. Therefore, our attention is directed to the specific surface area, which is closely dependent on both the size and distribution, and also to the most interesting property of the powder having a large surface area. Trials were made of quantitative expressions for the effects of the experimental conditions on the area.

In this work, the specific surface area was calculated from the data of the particle size distribution. As a formula to express the size distribution, the modified Rosin-Rammler distribution function,⁴⁾

$$R(D_p) = \frac{100}{\alpha - \beta} \left[\exp \left\{ - \left(\frac{D_p}{D_e} \right)^m \right\} - \beta \right] \dots\dots\dots(2)$$

is used, where *R* is the cumulative oversize (wt%), *D_p* the particle size (μm), *m* the distribution constant (—), and *D_e* the absolute size constant (μm). The modification is based on the fact of particle sizes limited within the maximum particle size (*D_{max}*) and the minimum one (*D_{min}*). The constants *α* and *β* are defined as follows;

$$\alpha = \exp \left\{ - \left(\frac{D_{min}}{D_e} \right)^m \right\} = 0.999, \\ \beta = \exp \left\{ - \left(\frac{D_{max}}{D_e} \right)^m \right\} = 0.001.$$

An analysis of the particle size distribution with Eq. (2) gives *m* and *D_e*, two parameters of a modified Rosin-Rammler distribution. The specific surface area can be calculated by substituting *m* and *D_e* into the following equation,⁵⁾

Fig. 12. Relations between the specific surface area and the peripheral velocity of the rotating disk.

$$S_v = \frac{\phi}{D_e} \left\{ \Gamma(K) - \frac{0.0010^k}{K} \right\}, \quad K = 1 - \frac{1}{m} \dots\dots\dots(3)$$

where, *S_v*: the specific surface area (m⁻¹)
Γ(K): the gamma function about *K* (—)
φ: the specific surface shape factor (—).

In this case, the shape factor *φ* is 6 according to an assumption that all the particles in a powder are spherical. Figure 12 shows the relations between the specific surface areas with Eq. (3) and the peripheral velocities of the rotating disk by way of log-log plots. The open symbols are the present results and the solid black ones the results by Schmidt and Knauss²⁾ for the atomization of the mercury drops with the rotating blades, where *D_o* is the diameter of mercury drop. Every group of the results indicates a good linearity and a good agreement on the slope as well as in Fig. 11. This agreement originates in a similar atomization mechanism as above mentioned.

Next, an examination is made on how each of the experimental conditions affects the specific surface area. There are 4 directly effective conditions, the distance between the blades *R_dθ_b* (m), the diameter of the nozzle hole *D_n* (m), the peripheral velocity of the rotating disk *V_d* (m·s⁻¹) and the ejecting velocity *V_e* (m·s⁻¹). The initial temperature of the melt *T_i* (K) is the indirect one because of having the effects on the density *ρ_m* (kg·m⁻³), the viscosity *μ_m* (Pa·s) and the surface tension *σ_m* (N·m⁻¹) of the material.

The final relation among these experimental conditions and the specific surface area is obtained by a dimension analysis based on the *π* theorem. In the theorem,

$$F(S_v, R_d \theta_b^{k_1}, D_n^{k_2}, V_e^{k_3}, V_d^{k_4}, \rho_m^{k_5}, \mu_m^{k_6}, \sigma_m^{k_7}) = 0 \dots\dots\dots(4)$$

is held. When the 8 physical quantities are arranged for the basic units of mass, length and time, the 3 factors of *k₅*, *k₆* and *k₇* can be exchanged with the equations which consist of *k₁*, *k₂*, *k₃* and *k₄*. Consequently Eq. (4) can be rearranged into the fol-

lowing form,

$$F\left\{\left(\frac{\mu_m^2}{\rho_m \sigma_m} S_v\right), \left(\frac{\rho_m \sigma_m}{\mu_m^2} R_d \theta_b\right)^{k_1}, \left(\frac{\rho_m \sigma_m}{\mu_m^2} D_n\right)^{k_2}, \left(\frac{\mu_m}{\sigma_m} V_e\right)^{k_3}, \left(\frac{\mu_m}{\sigma_m} V_d\right)^{k_4}\right\} = 0 \quad \dots\dots\dots(5)$$

Finally,

$$\frac{\mu_m^2}{\rho_m \sigma_m} S_v = C \left(\frac{\rho_m \sigma_m}{\mu_m^2} R_d \theta_b\right)^{k_1} \left(\frac{\rho_m \sigma_m}{\mu_m^2} D_n\right)^{k_2} \times \left(\frac{\mu_m}{\sigma_m} V_e\right)^{k_3} \left(\frac{\mu_m}{\sigma_m} V_d\right)^{k_4} \quad \dots\dots\dots(6)$$

is obtained. This equation consists of the 5 dimensionless groups, where C , k_1 , k_2 , k_3 and k_4 are constants. From the operating conditions and the specific surface areas estimated with Eq. (3), these constants can be determined by the successive approximation based on the least squares method.⁶⁾ In this work they are as follows;

$$\begin{aligned} C &= 4.5836 \times 10^{-12} \\ k_1 &= 3.6579 \times 10^{-1}, \quad k_2 = 1.9172 \\ k_3 &= 3.5208, \quad k_4 = 1.0815 \end{aligned}$$

Each dimensionless group in the right-hand side of Eq. (6) indicates the positive effect on the increment of the specific surface area. Especially the group which contains the ejecting velocity V_e seems to be most effective. Further examination, however, should be necessary for the confirmation because the pressure for ejection was kept constant in this work so that the ejecting velocity was not varied in the sufficiently wide range. Since this work was mainly carried out with the various changes in the peripheral velocity, the exponent k_4 could be precisely determined for the dimensionless group containing the peripheral velocity of the rotating disk.

In order to examine the adequacy of Eq. (6), a comparison is made between the two specific surface areas (S_v and S'_v) which are calculated by different ways. S_v is calculated from the data of the particle size distribution with Eq. (3), and S'_v is done with Eq. (6). The results are shown in Fig. 13, where the circular symbols indicate the relations for Sn samples, the triangular ones for Sn-20wt%Cu and the quadrangular one for Sn-60wt%Cu. These plots settle closely along the solid straight line, indicating the agreement between S_v and S'_v . Therefore the relation between the specific surface area and the experimental conditions may be expressed quantitatively with Eq. (6).

2. Powdery Shape

As observed in Figs. 8 to 10, the powdery shape depended on the sample material and the particle size. Then concerns require a discussion on the variation. The factors to be considered are the time durations necessary for the particle to solidify and to transform into a spherical one. Another should be taken into consideration in the present work that is the interval elapsing while the particle flies to the water

Fig. 13. Comparison between the two specific surface areas calculated by Eqs. (3) (S_v) and (6) (S'_v).

curtain, because the collision gives rise to deformation in shape before solidification.

The first estimation is of the time necessary for the particle to become a spherical shape. The calculation was carried out by using the model based on the following assumptions,

- (1) In a moment of atomization the melt droplets are acicular or cylindric shape as shown in Fig. 3.
- (2) The initial cylindric droplet have a dimension with a length to diameter ratio equal to 10 in order to simplify the calculation.
- (3) During the transformation the temperature of the melt is constant ($= T_i$).
- (4) The transformation is not disturbed by the air flow.

Consequently an equation⁷⁾,

$$t_{\text{sph}} = \frac{3\pi^2 \mu_m}{4 V_m \sigma_m} (r_1^4 - r_2^4), \quad \dots\dots\dots(7)$$

can be obtained, where t_{sph} is the transformation time to spherical shape (s), V_m the volume of a molten droplet, r_1 the droplet radius of the resultant sphere and r_2 the radius of the initial cylindric droplet, respectively.

The next calculations are of the solidification time according to the following assumptions.

- (1) The metal droplets have spherical shapes.
- (2) The heat is taken away from the particle surface only by convection.
- (3) There is no temperature gradient in the particle.
- (4) There is no change in the physical properties of the particle during cooling.

On these assumptions, two equations,⁸⁾

$$t_{\text{sol}s} = \frac{D_p \rho_m}{6h} C p_m \ln \left(\frac{T_i - T_g}{T_m - T_g} \right) \quad \dots\dots\dots(8)$$

$$t_{\text{sol}f} = \frac{D_p \rho_m}{6h} \left\{ C p_m \ln \left(\frac{T_i - T_g}{T_m - T_g} \right) + \frac{\Delta H_m}{T_m - T_g} \right\} \quad \dots\dots\dots(9)$$

can be obtained, where $t_{\text{sol}s}$ and $t_{\text{sol}f}$ are the initial

and final times of the solidification (s), h the heat transfer coefficient ($\text{J}\cdot\text{m}^{-2}\cdot\text{s}^{-1}\cdot\text{K}^{-1}$), Cp_m the heat capacity of metal ($\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$), T_i the initial temperature of molten metal (K), T_g the temperature of cooling gas (K), T_m the melting point of metal (K), and ΔH_m the specific latent heat of fusion of metal ($\text{J}\cdot\text{kg}^{-1}$), respectively.

Since the following conditions,

$$1 < Re \left(= \frac{D_p V_p \rho_g}{\mu_g} \right) < 7 \times 10^4$$

and

$$0.6 < Pr \left(= \frac{Cp_g \mu_g}{\lambda_g} \right) < 400$$

are satisfied in the present work, the heat transfer coefficient h is determined by the equation,⁹⁾

$$h = \frac{\lambda_g}{D_p} (2.0 + 0.6 Re^{0.5} \cdot Pr^{0.33}) \dots\dots\dots(10)$$

where, Re : Reynolds number (—)
 Pr : Prandtl number (—)
 V_p : the flying velocity of particle ($\text{m}\cdot\text{s}^{-1}$)
 λ_g : the thermal conductivity of gas ($\text{J}\cdot\text{m}^{-3}\cdot\text{s}^{-1}\cdot\text{K}^{-1}$)
 ρ_g : the density of gas ($\text{kg}\cdot\text{m}^{-3}$)
 μ_g : the viscosity of gas ($\text{Pa}\cdot\text{s}$)
 Cp_g : the heat capacity of gas ($\text{J}\cdot\text{kg}^{-1}\cdot\text{K}^{-1}$).

V_p is also assumed to be equal to the rotating velocity of disk as shown in Fig. 3.

The flying time to the water curtain t_w (s) is calculated by an equation,

$$t_w = \frac{L_w}{V_p} \dots\dots\dots(11)$$

where, L_w : the distance between the water curtain and the atomizing point (m).

The calculations of t_{sph} , $t_{sol\ s}$, $t_{sol\ f}$ and t_w are carried

out according to the data of Sn, Sn-20wt%Cu and Sn-60wt%Cu (sample Nos. 3, 12 and 14) and the results are shown in Figs. 14 to 16. The broken line indicates the transformation time, the one-dot chain line the moment when the solidification starts, the solid line the one when it finishes and the dotted line the arrival time at the water curtain. The three times of t_{sph} , $t_{sol\ s}$ and $t_{sol\ f}$ increase with the particle size, while t_w remains constant despite of the size variation. The ordinate is scaled on the left-hand side when $t_{sol\ s}$ or $t_{sol\ f}$ is shorter than t_w , and on the right-hand side otherwise.

Those three figures give the following informations,

(1) In every case the spherification time is much shorter than the two solidification times. Therefore,

Fig. 15. Time-particle size diagram (sample No. 12); t_{sph} is calculated with Eq. (7), $t_{sol\ s}$ with Eq. (8), $t_{sol\ f}$ with Eq. (9), and t_w with Eq. (11).

Fig. 14. Time-particle size diagram (sample No. 3); t_{sph} is calculated with Eq. (7), $t_{sol\ s}$ with Eq. (8), $t_{sol\ f}$ with Eq. (9), and t_w with Eq. (11).

Fig. 16. Time-particle size diagram (sample No. 14); t_{sph} is calculated with Eq. (7), $t_{sol\ s}$ with Eq. (8), $t_{sol\ f}$ with Eq. (9), and t_w with Eq. (11).

the spherical shape can be completed during the flight.

(2) The solidification occurs after the arrival at the water curtain under the condition where the particle size is over a certain limit. Under this condition, the droplet once transformed to spherical shape further changes into irregular shape by the water flow.

(3) The limitation in the particle size, where the irregular particles are produced, is different among the materials. The widest range is of Sn, the second of Sn-20wt%Cu and the last of Sn-60wt%Cu.

These calculation results are agreed with the observations shown in Figs. 8 to 10. More complicated factors, however, should be considered in the actual shape changes. For example the droplet seems to become spherical more slowly due to the temperature difference between the surface and interior during cooling and also to the solidified film formed on the surface. Therefore, the resultant powder particles have not only spherical but also ligamental or tear drop shapes.

V. Conclusion

In the present work the metallic powders have been produced by impacting molten metal and alloys with a bladed rotator. Interest is taken in the powder properties with respect to the operating conditions.

The results are summarized as follows.

(1) The mean particle size of the atomized powder varies with the peripheral velocity of the rotating disk as expressed by Eq. (1) quantitatively.

(2) The specific surface area which is estimated from the particle size distribution with Eq. (3) is re-

lated to the 4 main effective conditions, the distance between the blades $R_d\theta_b$, the diameter of the nozzle hole D_n , the peripheral velocity of the rotating disk V_d and the ejecting velocity V_e . The quantitative relationship (Eq. (6)) can be obtained by a dimension analysis.

(3) The powdery shape depends on the times necessary for the atomized melt drop to solidify, to spherify and to fly to the water curtain. These calculated results shown in Figs. 14 to 16 are well agreed with the observations in Figs. 8 to 10, respectively.

Acknowledgements

The authors are indebted to Prof. A. Otsuka and Dr. K. Mori, Nagoya University, for their guidance on the SEM observation.

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