Production of Fine Metallic Powders by Hybrid Atomization Process*

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Hybrid Atomization is a recently developed powder-making process that combines effectively free-fall gas atomization and centrifugal atomization. This technique can produce very fine spherical powders with mean diameters of around 10 micrometers, and in high yields. The present report discusses the concept and basic principles of hybrid atomization. Process experiments were carried out and the optimal processing conditions were obtained. The results show that the influences of processing parameters and optimum conditions differ greatly between the proposed and the conventional atomization processes. A new correlation of atomization equation applicable to hybrid atomization is proposed and discussed.

Key Words: Fine Powders, Perfectly Spherical Powders, Narrow Size Distributions, Low Oxidation, Hybrid Atomization

1. Introduction

Very fine metal powders, with diameters of around 10 μm or less, often exhibit unique structures or properties that are not attainable in larger sizes⁽¹⁾. Demand for such powders is increasing rapidly as new technologies emerge that can make use of their unique characteristics⁽²⁾. Some examples of these rapidly growing applications are metal injection molding, electronic solders and conductive inks. However, the requirements for powder characteristics in these applications are very strict, and conventional atomization techniques cannot meet them economically. In an

attempt to overcome this problem, a new powder-making process, Hybrid Atomization, was developed recently to produce very fine metal powders with high quality and at low production cost⁽²⁾.

2. Concept and Basic Principles

The new idea is that controlling the liquid film formation on the rotating disk can facilitate the production of fine powders. In centrifugal atomization, the powder sizes are usually very coarse even under very high rotating speeds. The main reason for this coarseness was found to be the formation of a thick liquid film on the rotating disk before melt breakup. The thicker the film, the coarser the mean particle size becomes. Meanwhile, the more unstable the flow type of the film, the wider the powder size distribution will be. If the formation of film on the disk can be controlled well to obtain a very thin but stable film, finer powders should be produced with a tighter size distribution. However, it will be difficult or impractical to do so merely by adjusting processing parameters in conventional centrifugal atomization, as this would require a much higher rotating speed or a very low liquid supply rate. So a new way has been sought to control the film formation effectively and economically.

The basic concept of hybrid atomization is to

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hybridize free-fall gas atomization with centrifugal atomization. In our work, low-pressure gas atomization was utilized to promote the formation of a very thin stable film before centrifugal breakup and to provide a better preparation for the final pulverization of melts. According to our theoretical analysis, the liquid film thickness in hybrid atomization is about 50–60% of that in corresponding centrifugal atomization. In most cases of hybrid atomization, the basic flow type of liquid on disk is laminar, and it is more stable than that in conventional centrifugal atomization. Since the melt has been re-distributed by a gas spray before its final atomization, it should break down more easily into finer particles under the centrifugal force.

3. Process and Experimental

A schematic diagram of our hybrid atomization device is shown in Fig. 1. The melt is atomized by a free-fall gas atomizer into coarse liquid droplets. These droplets are sprayed onto a high-speed rotating disk and form a thin liquid film on the disk. This film is then pulverized by centrifugal force. The solidified powders are collected by a vacuum cleaner at the bottom of the atomization chamber.

Our preliminary experiments showed that the device's main processing parameters are metal superheat, disk speed, spray distance and melt diameter. Other parameters, such as gas pressure and melt diameter, were found to have no significant influence on the powder characteristics. Hybrid atomization was carried out for Sn-9 wt% Zn, with the variation ranges of the main parameters as follows: melt temperature 573–823 K; disk rate 524–3141 rad/s; spray distance 50–160 mm; disk diameter 50–100 mm. Other constant parameters were: gas pressure 0.6 MPa (Nitrogen), gas atomization angle $2\pi/9$ rad (40°) and nozzle diameter 2 mm. The disk material was Sialon (by Hitachi Metals). The powder size and size distribution were measured by the sieving and

Coulter Counter methods. The shapes and microstructures of the powders were observed by SEM (JSL5400) and by OM.

4. Results and Discussion

4.1 Powder characteristics

As shown in Fig. 2 and Table 1, hybrid atomization can produce finer spherical powders with a higher yield and a narrower size distribution than gas atomization can. For example, in hybrid atomization the yield of powder fraction under $26\mu\text{m}$ is 82–90%, whereas in conventional gas atomization it is less than 10% under the same conditions. If only the gas atomizer works, the powders obtained have an average particle size of $64\mu\text{m}$, with a geometric standard deviation of about 2.0. If only the rotating disk runs, the average particle size is 80–140 μm and the geometric standard deviation 1.3–1.5. If the whole hybrid atomization device is used, the average powder size can decrease to 10–15 μm , with a corresponding geometric standard deviation of only 1.3–1.7.

As shown in Fig. 3, most of the powder particles are spherical or nearly spherical. Few particles are

Table 1 Comparison among atomization methods

Method	Powder Size(μm)	Size Distribution	Powder Shape
WA	5~150	broad $\sigma_g: 1.7 \pm 2.4$	irregular
GA	20~100	Log-Normal $\sigma_g: 2.0 \pm 0.3$	good sphere
CA	25~85	tight $\sigma_g: 1.3 \pm 1.5$	sphere
HA	10~15	tight $\sigma_g: 1.3 \pm 1.7$	sphere

Fig. 1 Schematic diagram of hybrid atomization device

Fig. 2 Comparison of results between hybrid atomization and gas atomization

Fig. 3 Powder characteristics of hybrid atomization under different conditions

dumbbell-shaped or granular. In addition, few satellites are found around main particles under typical hybrid atomization conditions.

4.2 Influences of processing variables

4.2.1 Melt temperature According to Fig. 4, when melts are atomized at a low temperature and with a low disk rate, the mean powders are coarse, with two peaks in their size distribution. When the melt temperature increases to a moderate temperature (673 K), the powders become finer and the minor peak in the size distribution curve shifts to a finer particle range. However, if the melt temperature is too high (823 K), this trend of powder refinement with increasing superheat will not continue. On the contrary, the average powder size and geometric standard deviation will increase. Also, as shown in Fig. 3, the higher the superheat, the more spherical the powders become. In addition, it seems that a proper superheat can reduce the presence of satellites around main particles.

4.2.2 Disk rate and size As shown in Fig. 5, when disk rate increases from 524 to 2 094 rad/s, the mean powder diameter decreases and the size distribution changes from one peak to two peaks. Also in Fig. 5, when disk diameter increases from 50 mm to 100 mm, the curve of the powder size distribution changes from two peaks to almost entirely one sharp peak. Compared with other parameters, the influences of disk speed (including disk rate and size) on both powder size and size distribution are quite remark-

Fig. 4 The influence of superheat on hybrid atomization results

Fig. 5 The effects of disk rate and size on hybrid atomization results

Fig. 6 The effect of spray distance on hybrid atomization results

able.

4.2.3 Spray distance As shown in Fig. 6, when spray distance increases from 50 mm to 100 mm, the powders obtained become finer and the size distribution tighter. However, according to our previous work⁽³⁾, if the spray distance continues to increase, say, from 100 mm to 160 mm, the powders become coarse again. In addition, when the spray distance is too small (50 mm), a lot of deposits will solidify on the rotating disk. However, if the spray distance is too large, many droplets will not be atomized centrifugally after their gas atomization. So there exists an optimum value or range for spray distance, corresponding with the best atomization results.

4.3 Process optimization

The optimum processing conditions for hybrid atomization of Sn-9 wt% Zn alloy are as follows: melt temperature 673 K (superheat 200 K); disk rate 2 094 rad/s; disk diameter 100 mm; spray distance 100 mm; melt diameter 2.0 mm; gas pressure 0.6 MPa (Nitrogen).

The characteristics of powders atomized under the above conditions are as follows: powder mass median diameter 11 μm; geometric standard deviation 1.3; spherical shape; the yield of powders under 45 μm is 98%.

4.4 Powder size correlation

Based on the results of our theoretical studies that will be published elsewhere⁽⁴⁾, the mass median diameter of powders in hybrid atomization obeys the following correlation.

$$d_m = 3.25(P/P_0)^{-1/2} [1 + K \sin(\theta/2)]^{-1/2} \left(\frac{\sigma}{\rho D \omega^2} \right)^{1/2} \quad (1)$$

Where d_m is the mass median diameter of powders; P and P_0 are the pressure of gas jet and atmosphere ($P_0=0.1$ MPa); K is a constant and about 23.4 under our present experimental conditions; θ is the gas atomization angle; σ , ρ , D and ω have the same meanings as in centrifugal atomization.

The calculated mean powder sizes in hybrid atomization are about 8–20 μm and agree well with our experimental data, i.e. 10–15 μm in typical cases. The mean particle size is 1/6–1/8 of that obtained from centrifugal atomization. However, the effects of disk rate and size on powder size diminish when the disk speed is very high. This is because the atomization mode shifts from DDF mode to a mixed mode (DDF+LF) under high disk speed⁽⁴⁾. In addition, according to our experiment results, melts can be broken down efficiently into droplets of around 10 μm at the disk linear speed of about 100 m/s in hybrid atomization. Powders become only a little finer with higher disk speed. Since the above equation was deduced from the DDF mode, there will be a deviation between mean powder size prediction and actual atomization results with very high rotating speeds.

5. Discussion

In hybrid atomization, the main role of gas atomization is to produce a much thinner and more stable liquid film on disk before its final breakup. Gas atomization provides a proper spray dispersion of melt, enhances the liquid flow on disk and increases the stability of liquid film flow⁽⁵⁾. In centrifugal atomization, the flow type of the liquid film depends on specific conditions and is sometimes turbulent⁽⁶⁾. But under most typical hybrid atomization conditions the flow should be laminar, since its Reynolds number based on surface flow is much smaller than that in centrifugal atomization.

On the other hand, in hybrid atomization several main processing parameters show different influences on atomization results from those in conventional atomization processes. Among them, disk rate and size have very remarkable effects on powder size and size distribution. This is because disk speed affects not only the liquid formation on disk but also the atomization mode. When the disk linear velocity increases, the breakup ability of the centrifugal atomizer becomes stronger. The liquid film becomes thinner and its main disintegration mode tends to be the DDF+LF or even LF mode. Thus, the resulting powders become finer. As for the powder size distribution, it depends heavily on the stability of both the liquid film and the atomization procedure. The more stable the liquid film and the final centrifugal breakup procedure, the tighter the powder size distribution. In addition, both melt superheat and spray distance are important factors. They first influence the characteristics of liquid film on disk and then influence the droplet formation. These influences are attributed to the close relationship between temperature variation and the melt thermo-physical properties, such as

surface tension, viscosity and density. In any case, it is known that the temperature dependence of surface tension and viscosity should not be pronounced for metals and alloys⁽⁷⁾. If enough superheat and proper spray distance can be adopted to avoid the premature solidification of melt before its final pulverization, the hybrid atomization results will not change much with further variation of either melt temperature or spray distance. So the optimal conditions should be a good combination between enough superheat and proper spray distance. However, powders are formed by centrifugal force in hybrid atomization, and their characteristics are determined mainly by the final breakup. Consequently, parameters such as gas pressure, gas atomization angle and melt diameter—which govern atomization results in gas atomization—affect the final results of hybrid atomization only indirectly. Their influences are minor in this new process, and are far less pronounced than in conventional gas atomization.

6. Conclusions

(1) A new process, Hybrid Atomization, was developed to hybridize effectively free-fall gas atomization with centrifugal atomization. This technique can produce very fine powders with high yields and at low cost.

(2) The influences of several main processing parameters on atomization results are different from those in conventional atomization processes.

(3) Under the optimal conditions, spherical Sn-9 wt% Zn powders can be produced with a mass median

diameter of 11 μm and a standard deviation of 1.3, with about 90% yields of particles less than 26 μm and 98% under 45 μm .

(4) A new atomization equation applicable to hybrid atomization was presented, and it agrees well with experimental data.

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