# Production of Ultra Fine Lead-Free Solder Powders by A New Atomization Technology 

K. Minagawa, H. Kakisawa, S. Takamomi, Y. Osawa and K. Halada<br>Ecomaterials Research Center, National Institute for Materials Science, 1-2-1 Sengen, Tsukuba City, Ibaraki 305-0047, JAPAN.<br>Fax: 81-029-859-2601, e-mail: MINAGAWA.Kazumi@nims.go.jp


#### Abstract

A new atomizing technique has been required to obtain a spherical, small and clean powder for soldering economically. To produce such lead-free solder balls, a novel powder-making process, "hybrid atomization" that combines free fall gas atomization and centrifugal atomization, was used. This technique produces very fine and spherical tin alloy powders with a mean diameter of about ten micrometers and very narrow size distribution with few satellites at low production cost. Taking $\mathrm{Sn}-9 \mathrm{mass} \% \mathrm{Zn}$ alloy as an example, process experiments were carried out, and the effect of temperature, spray distance and disk rotating speed on the resultant powder properties were examined. The optimal processing conditions were determined from the results; the influences of the processing parameters on the properties of the obtained powders were quite different from those in conventional atomization processes. The spherical powder with a mean diameter of $10.6 \mu \mathrm{~m}$ and a standard deviation of $1.3 \sim 1.7$ was obtained in the determined optimum condition.


Key words: Hybrid Atomization, Lead-Free Solder, Fine Powder, Ecomaterials

## 1. INTRODUCTION

Lead-containing tin alloy solders have been used extensively in interconnecting and packaging of components for micro-electronic applications [1]. Since the end of last century, considerable effort has been focused on the research and development of lead-free solders for the sake of necessary environmental protection and increasing health concerns [2]. Although remarkable progresses have been achieved in searching several promising candidate alloys, the economical production of fine uniform solder balls will be required for their possible industrial applications [3]. For example, lead-free solder powders for electronic applications have a very demanding specification; they must have a very narrow size distribution and be perfectly spherical and satellite-free, they must be very low in oxygen content ( $\sim 100 \mathrm{ppm}$ ). Attempts to make this product with inert gas atomization have now virtually ceased as yields are as little as $5 \%$ and the avoidance of satellites is very difficult [4]. Thus up to now it seems difficult or uneconomical to produce highmquality powders for lead-free solders with conventional atomization. So a new method should be devised to make acceptable lead-free solder balls.

## 2. HYBRID ATOMIZATION CONCEPT

Some powder processes combining gas atomization and centrifugal atomization have been reported. Oguchi et al [5]. produced
amorphous flake powders by spraying high pressure gas atomized melts on a rotating cone type copper disk for rapid solidification. Chen et al [6]. established multistage atomization in which melted droplets broken by gas jet were further broken by pelting a rotating disk with them. In our hybrid atomization method, gas atomization is used for spraying melts uniformly on a rotating disk, and forming a thin melt film on it. Then the fine and spherical powder with a narrow size distribution is produced by centrifugal atomization.

## 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 $\mathrm{Sn}-9 \mathrm{wt} \% \mathrm{Zn}$ (melting point 472 K ), with the variation ranges of the main parameters as follows: melt temperature $573 \sim 823 \mathrm{~K}$; disk rate $524 \sim 3141 \mathrm{rad} / \mathrm{s}$; spray distance $50 \sim 160 \mathrm{~mm}$; disk diameter $50 \sim 100 \mathrm{~mm}$. Other constant parameters were: gas pressure 0.6 MPa (nitrogen), gas atomization angle $2 \pi / 9 \mathrm{rad}\left(40^{\circ}\right)$ 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 a scanning electron microscope (SEM) and by an optical microscope (OM).

## 4. RESUITS AND DISCUSSION

The comparison of $\mathrm{Sn}-9 \% \mathrm{Zn}$ powder characteristics (mean particle size, shape and geometric standard deviation) by hybrid atomization and by gas atomization is shown in Table 1 and Fig. 2. As shown in Table 1 and Fig. 2, hybrid atomized powders have finer particle sizes, narrower size distributions than conventionally gas atomized powders.

### 4.1 Effect of Superheat

As shown in Fig. 3, when superheat increases,
Table 1 Comparison of atomization methods.

| Atomization <br> Method | Powder Size <br> $(\mu \mathrm{m})$ | Size <br> Distribution $(\mu \mathrm{m})$ | Powder Shape |
| :---: | :---: | :---: | :---: |
| GAS <br> Atomization | $20 \sim 100$ | Log-Normal <br> $\sigma g: 2.0 \pm 0.3$ | Sphere |
| Hybrid <br> Atomization | $10 \sim 20$ | Tight <br> $\sigma g: 1.3 \sim 1.7$ | Sphere |



Fig. 2 SEM comparison of powders by hybrid atomization and gas atomization.


Fig. 3 The influence of superheat on hybrid atomization.
the mean particle size decreases and the size distribution becomes narrower. But after superheat is above 200 K (melt temperature 673 K ), the effects of superheat on the powder mean size and size distribution become little.

Superheat can influence the surface tension and viscosity of melts. When superheat is greater, the surface tension and viscosity of melts become smaller, so the powder size also becomes smaller. Thus, superheat can influence the powder size indirectly in this way.

Moreover, during hybrid atomization superheat will affect directly the liquid film formation and final breakup procedure of metallic melts and the cooling \& solidification procedure of droplets. When superheat increases enough, the liquid film becomes thinner, more stable and flows better on the disk and is also easier for further breakup during final centrifugal atomization so that the powder size becomes finer. However, this refinement effect has some limit. After superheat is above a certain value, the powders will not become further finer because the melt film does not become thinner remarkably but the evaporation of zinc would accelerate under high temperature.

### 4.2 Effect of Disk Rotating Speed

As shown in Fig. 4, the higher the rotating speed, the smaller the powder mean size and the geometric standard deviation, that is, the size distribution becomes narrower. This effect is very remarkable. The final particle size obtained by hybrid atomization is mainly controlled by the centrifugal disintegration. The higher disk rate bears a thinner liquid film on the disk. Thus the melt film breaks up more completely.


Fig. 4 The effects of disk rate and size on hybrid atomization.


Fig. 5 The effect of spray distance on hybrid atomization.

### 4.3 Effect of Spray Distance

As shown in Fig. 5, there exists an optimum value for spraying distance at which the powder mean size reaches minimum. When the spraying distance increases or decreases, the powders obtained become coarser. When the spray distance is too short, most of the gas-atomized droplets will hit and concentrate in the central area of the disk. So the liquid film will be very thick at the disk center and very thin near the disk edge. Actually it looks like centrifugal atomization to some degree. But if spray distance is very long, the atomization results will be bad. The film on disk will be in a semisolid state because some droplets have premature solidification before centrifugal breakup. Also, only a part of the film will be broken down into powders while the solidified part will form a deposit instead during atomization. On the other hand, the percentage of only gas-atomized powders will increase remarkably when spray distance increases and the spray cone is larger than the disk. The powders obtained in this case will be very coarse with low powder yields. So one pre-condition for successful hybrid atomization is a proper spray distance. The optimum spray distance should ensure the continuous re-formation of a thin, evenly distributed liquid film from gas-atomized droplets on the disk during hybrid atomization procedure. In this study the optimal value of spray distance was 100 mm , which corresponds with the best atomization result. Together with other main parameters such as superheat and disk rotation, spray distance plays an important role as a combination parameter in hybrid atomization.

### 4.4 Process Optimization

In this study, the optimum process parameters were decided as follows: superheat 200 K (metal temperature 673 K ), spraying height 100 mm , gas pressure 0.6 MPa , disk rotating speed $2094 \mathrm{rad} / \mathrm{s}$. The characteristics of powders obtained under the above optimal conditions were as follows: mean particle diameter $11 \mu \mathrm{~m}$; geometric standard deviation $\sigma$ 1.3; spherical shape; few satellites; high yields of very fine powders: $90 \%$ under $26 \mu \mathrm{~m}$ and $98 \%$ under $45 \mu \mathrm{~m}$.

### 4.5 Discussion on Hybrid Atomization Mechanism

## and Control

The key concept behind hybrid atomization is the formation of a thin and stable liquid film on the rotating disk before the final centrifugal breakup of the melts. Gas spray in hybrid atomization does not play a role in powder making directly but in thinner liquid film re-formation. Powder characteristics are thus controlled by both gas atomization and centrifugal atomization. As shown in Fig. 6, the mean particle diameters ( $\mathrm{d}_{\mathrm{m}}$ ) of hybrid atomized powders are closely associated with not only the centrifugal atomization parameters (such as disk rotation speed, disk angular velocity: $\omega$, disk radius: R , melt surface tension: $\sigma$ and viscosity: $\mu$ on disk, etc.) but also with the gas atomization parameters (such as degree of superheating and spray distance). When the superheating, spray distance, and disk rotation speed increase properly, powders become finer. Together with the melt superheating and disk rotation, spray distance plays a key role in combining the gas atomization and the centrifugal atomization effectively. As an example shown in Fig. 6, in case A with a short spray distance and a moderate superheat, the hybrid atomization was assumed to be similar to centrifugal atomization since the liquid film thickness was large, and the powders obtained were coarse. In case B with a proper spray distance and a low superheat, the powder sizes were in the middle range because of the possibility of premature solidification and the formation of semisolid, unstable film on the disk. In case $C$ with a proper spray distance and a moderate superheat, very fine powders could be obtained especially under high disk speed. By the way, if the spray distance is very large, the results of hybrid atomization will be similar to those of gas atomization. In contrast, if the spray distance is excessively short, the results will be similar to those of centrifugal atomization. Therefore, the optimum atomization results depend on the proper selection and most advantageous combination of these main processing parameters.
According to the guiding concept of hybrid atomization [7-8], particle size and size distribution will be controlled primarily by the liquid film's characteristics (thickness, flow state, etc.) on the disk and by the centrifugal atomizer's

breakup ability, both of which can determine the main atomization mode and the liquid film instability before the final breakup. Very fine and uniform powders can be obtained in the case of very thin stable liquid film formation and strong atomizer breakup abilities. If the atomization mode is Direct Drop Formation (DDF), the particle size distribution will normally express bimodal distribution (two peaks). But in the case of Ligament Formation (LF) mode, there will be a very tight particle size distribution (mono-modal) with one peak [9]. Based on the disintegration mode diagram of hybrid atomization [6], the DDF mode is the main disintegration mode in typical hybrid atomization, and laminar flow is the basic type of liquid film flow on the disk, while the powders usually show bimodal distributions with a main peak and a minor peak, which agreed closely with the above experimental results.

By our experiments it was found that hybrid atomization can easily produce spherical powders. Theoretically speaking, the powder shapes can be controlled mainly by both the shape of the liquid droplets immediately after final centrifugal breakup and the following cooling \& solidification [9]. Since the liquid film on the disk is thinner than that in corresponding centrifugal atomization [6], the duration of the disintegration from liquid film to droplets will be shorter, which leads to a small and spherical droplets.

## 5.CONCLUSIONS

(1) Hybrid atomization was devised as a
promising candidate technology to produce lead-free solder powders efficiently and industrially.
(2) Very fine spherical $\mathrm{Sn}-9 \% \mathrm{Zn}$ powders with narrow size distributions and few satellites were obtained.
(3) Spray distance, superheat and disk rotating speed are three main processing parameters in hybrid atomization. Their influence laws on the hybrid atomization results and optimum conditions are different from those in conventional centrifugal atomization.

## 6. REFERENCES

[1] Y. Kariya, N. Williams, C. Gagg and W. Plumbridge: JOM, Vol. 53(2001), p. 39-42.
[2] S. K. Kang: JOM, Vol. 53(2001), p. 16.
[3] S. K. Kang: JOM, Vol. 54(2002), p. 25-26.
[4] J. J. Dunkley: Atomization, ASM Handbook, (ASM International, 1998), pp. 35-48.
[5] M. Oguchi, A. Inoue, T. Masumoto and K. Suzuki: J. Mater. Sci. 29(1994), p.1825-1832.
[6] Z. H. Chen, Y. Wang and P. Y. Huang, et al.: Chinese Patents No. 88212137.5, 1988, 2, 6.
[7] K. Minagawa, Y. Z. Liu, H. Kakisawa and K. Halada: Abstracts of The 2001 Japan National Autumn Conference on Powder and Powder Metallurgy, (Japan Society of Powder and Powder Metallurgy, 2001), 3-25A, pp. 84.
[8] K. Minagawa, Y. Z. Liu, H. Kakisawa and K. Halada: JSME International Journal, Series A, Vol. 46, No.3, (2003), p.260-264.
[9] K. Halada, and H. Suga: Powder and Powder Metallurgy(in Japanese). 37(1990), p. 405-408.

