# Effects of process parameters on surface morphology of metal powders produced by free fall gas atomization

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There are a number of process parameters which affect the characteristics of metal powders produced by free fall gas atomization. In the following work effects of various process parameters like apex angle of atomizer, focal length of atomizer, number of nozzles, diameter of nozzles, diameter of liquid metal delivery tube, superheat of liquid metal and type of metal etc. were studied on their surface morphology. It was observed that shape of powder particles depends on apex angle, superheat of liquid metal, type of metal and particle size range within a powder collective. Other parameters like focal length of atomizer, number of nozzles, diameter of nozzles and diameter of liquid metal delivery tube were found to have no effect on the shape of powder particles. However, Surface porosity and solidification shrinkage were observed on almost all types of metal powders. 

## 1. Introduction

Metal powders are widely produced in industries by free fall gas atomization. In this type of atomization liquid metal stream falls freely up to a distance before entering the gas field created by the gas nozzles of atomizer. That is why it is termed as free fall gas atomization. The disintegration of liquid metal stream takes place in the gas field of widely varying gas velocity [1–4]. The disintegrated liquid metal droplets solidify in the gas field downstream the geometric point and collect as metal powder [5]. The surface morphology (e.g. particle shape and porosity at the surface) of produced powder may be affected by various atomization parameters related to atomizing gas, liquid metal and atomizer etc [6, 7]. The surface morphology is important as it affects the flow and apparent density of powders [8].

Most of the work available in the literature on surface morphology of powders is produced by confined type atomization [9–11]. See and Johnston [12] atomized lead and tin by nitrogen gas using a free fall type atomizer. It was observed that the produced powder particles were not spherical. To explain it they calculated the spherodization and solidification time of powder particles. It was found that the solidification time was greater than the spherodization time. Therefore, they argued that the particles should have been spherical but practically it was not the case. So, the oxidation of particles [13] was reported as the reason for non spherical particles.

The aim of the present study was to find the effects of some parameters like plenum pressure, apex angle of atomizer, focal length of atomizer, number of gas nozzles, superheat of liquid metal, diameter of liquid metal delivery tube, type of metal and particle size range on powder particles surface morphology e.g. particle shape, surface porosity and surface shrinkage. Flight distance for complete solidification of different size but spherical shape particles was also measured in order to study the formation of spherical/non-spherical shape of particles. According to this study all particles should be spherical but practically it was not the case.

## 2. Experimetal

### 2.1. Atomization unit

In Fig. 1 a schematic sketch of the atomization unit is shown which was employed to produce metal powder. In the same set-up solidification distances of droplets in their flight were determined to study shape of particles by fixing an additional arrangement of trays, which is shown in Fig. 2. As shown in Fig. 1, a resistance type of furnace

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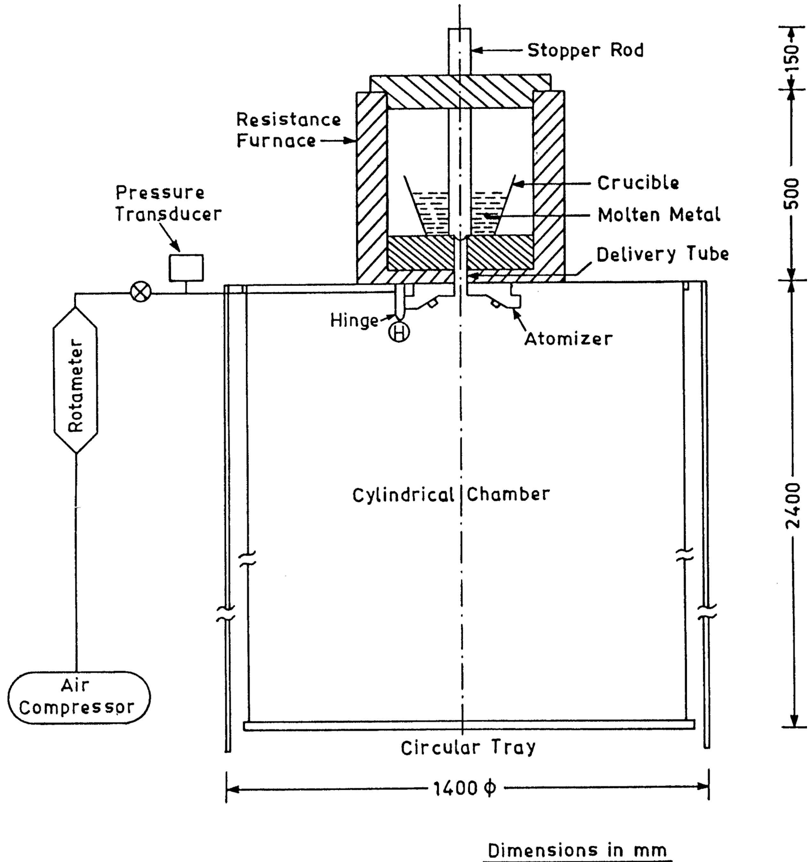



*Figure 1* Schematic sketch of the atomization unit.

was used to heat the graphite crucible. At center of the bottom of crucible a liquid metal delivery tube was fitted to produce the liquid metal stream. A stopper rod either opens or closes the liquid metal delivery tube. Various metal delivery tubes of different diameters varying from 3.5 to 12.0 mm were used. The atomizer was mounted on the inner side of a circular plate, while the furnace with the crucible was placed on the outer side such that the axis of the liquid metal delivery tube passes through the geometric point of atomizer. The vertically falling liquid metal stream was atomized in the cylindrical chamber of 1.4 m diameter and 2.5 m height. Fig. 3 shows a schematic sketch of the design of atomizer used in the present study. The atomizer consists of a manifold having a number of holes for fitting convergent type nozzles and gas supply arrangements. The holes were designed to be positioned at equal circumferential distances and inclined conically making an apex angle $\alpha$ such that the geometric point (it is the point of intersection of the axes of holes) lies on the central axis of the manifold. The distance between exit of nozzle and geometric point is termed as "Focal length" of the atomizer. Atomizers of different apex angle ((), focal length ($F$), number ($N$) and diameter ($D$) of nozzles as given in Table I were fabricated.

In order to determine the flight distance for complete solidification i.e. solidification distances of spherical shape particles, six conical trays numbered from 1 to 6 were arranged on a rotating rod at different vertical distances

TABLE I. Details of atomizers

| Atomizer | N | D (mm) | $\alpha$ (°) | F (mm) |
|---|---|---|---|---|
| a27 | 4 | 2 | 20 | 70 |
| a45 | 4 | 2 | 40 | 50 |
| a47 | 4 | 2 | 40 | 70 |
| a49 | 4 | 2 | 40 | 90 |
| a67 | 4 | 2 | 60 | 70 |
| A27 | 4 | 3 | 20 | 70 |
| A212 | 4 | 3 | 20 | 120 |
| A45 | 4 | 3 | 40 | 50 |
| A47 | 4 | 3 | 40 | 70 |
| A49 | 4 | 3 | 40 | 90 |
| A412 | 4 | 3 | 40 | 120 |
| A62 | 4 | 3 | 60 | 20 |
| A65 | 4 | 3 | 60 | 50 |
| A67 | 4 | 3 | 60 | 70 |
| A69 | 4 | 3 | 60 | 90 |
| A612 | 4 | 3 | 60 | 120 |
| b412 | 6 | 2 | 40 | 120 |
| B45 | 6 | 3 | 40 | 50 |
| B47 | 6 | 3 | 40 | 70 |
| B49 | 6 | 3 | 40 | 90 |
| B67 | 6 | 3 | 60 | 70 |

A, B: Atomizers with $N = 4$ and 6, respectively and $D = 3$ mm.
a b: Same as above but $D = 2$ mm.
Ist digit after capital or small letter* 10 = apex angle (degree) and following later digit(s) * 10 = focal length (mm).
Example: *A62:* Atomizer having $N = 4$, $D = 3$ mm, $a = 6 \times 10 = 60°$ $F = 2 \times 10 = 20$ mm.

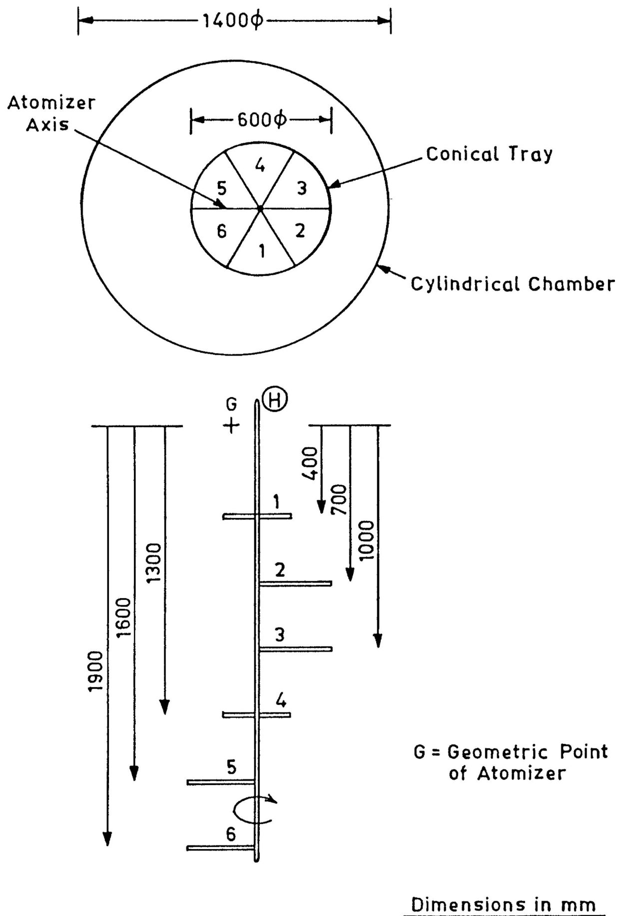



*Figure 2* Arrangement of trays to determine solidification distances of droplets moving in the gas stream.

measured from the point G (geometric point) such that each successive tray does not interfere with the other (see Fig. 2 for values of different distances of trays numbered from 1 to 6). This is achieved by positioning the trays at equal circumferential distances around the rod. The radial dimension of each tray was 300 mm. This arrangement was hinged vertically on the circular plate (Fig. 1) at a distance equal to half the radial dimension of the tray. This distance was selected to ensure the passing of the center of each tray through the axis of the atomizer during rotation of the rod.

## 2.2. Procedure

Aluminum, lead, zinc and tin were atomized in the set-up given in Fig. 1. For this purpose 500 gm of the respective metal was charged into the crucible. The liquid metal delivery tube was closed with stopper rod and the metal was heated to the required temperature above its melting point. The air was opened to the atomizer and the stopper rod was lifted to atomize the liquid metal stream. In this way a number of atomization runs were carried out at different plenum pressures (400 to 1900 kPa), different delivery tube diameters (3.5 to 12.0 mm) and different superheats of liquid metal (70 to 190 K) using different atomizers

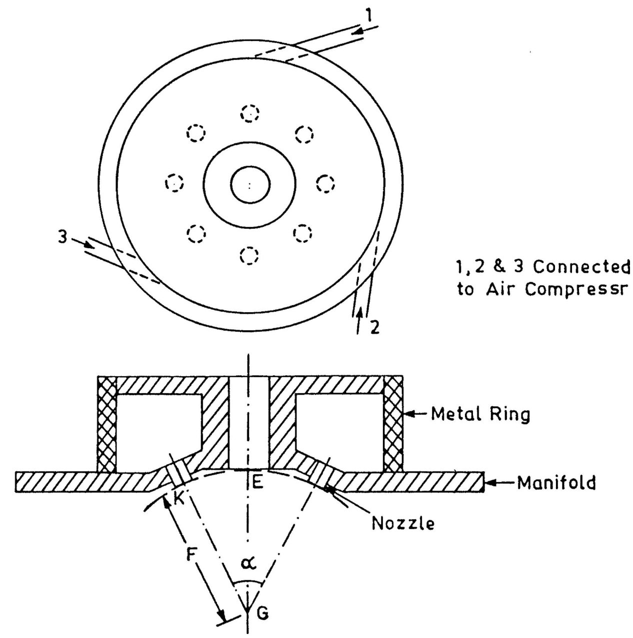



*Figure 3* Schematic diagram of the design of a free fall type atomizer.

reported in Table I. The surface morphology of powder particles was studied by scanning electron microscope (SEM) JEOL JXA 840A.

To determine the solidification distances of maximum size spherical shape droplets the atomization runs were carried out as described above but after fixing the aforementioned six conical trays in the atomization unit. Since size of spherical shape particles can be determined exactly as compared to other shapes, therefore experimental parameters were selected such that the atomization could produce a large fraction of spherical powder. SEM observations were made on the powder sample collected from each tray to determine the size of the spherical powder particles in each tray.

# 3. Results

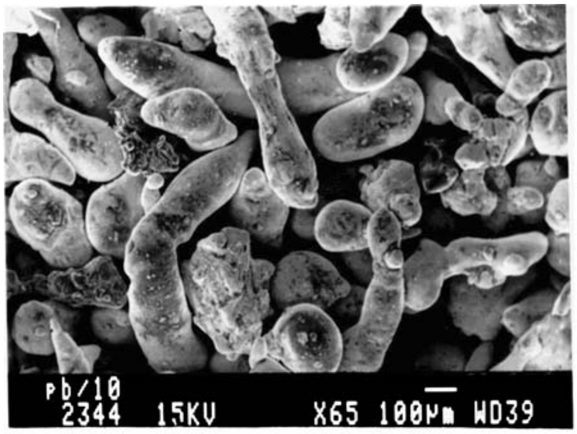
The surface morphology of produced powder was studied as a function of plenum pressure, apex angle, focal length, number of nozzles, diameter of delivery tube, superheat of liquid metal and type of metal.

## 3.1. Shape of particles

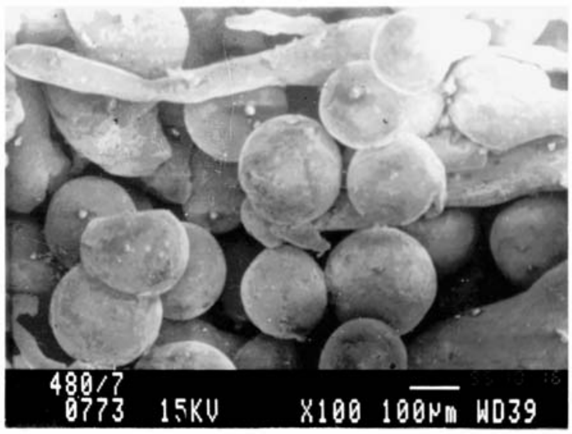
Each collective of powder was found to contain particles of mixed shape. Some collectives contain mixture of spherical and rounded particles, whereas others contain particles of spherical, rounded and irregular shape.

### *3.1.1. Effect of apex angle*

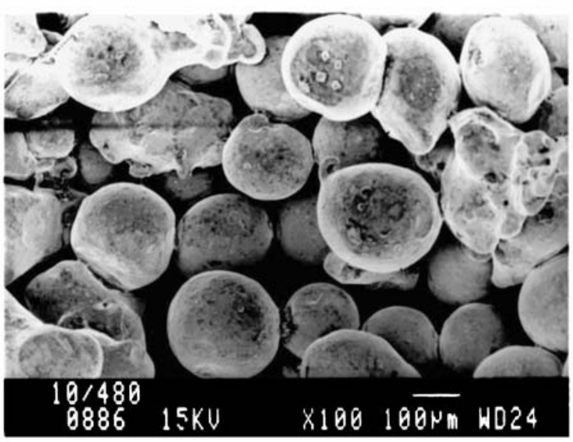
Fig. 4a, b & c shows micrographs of lead powder particles within the size range 76–211 $\mu$m produced by atomizers A 212, A 412, A 612 having different apex angles 20°, 40° and 60°. The liquid metal was superheated to 150 K. It can be seen that the proportion of spherical powder particles




(a)




(b)



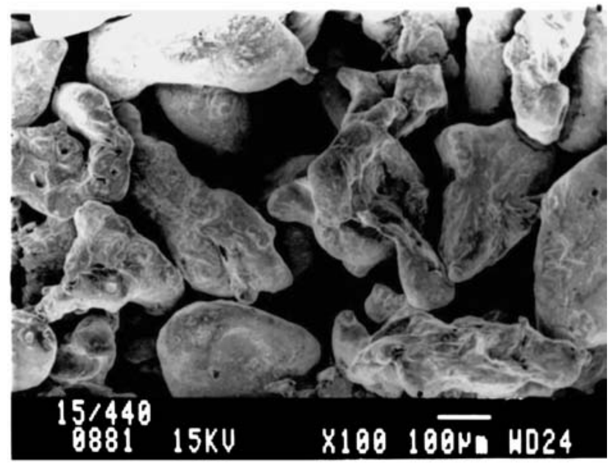

(c)

*Figure 4* SEM micrographs of the particles within the size range 76–211 $\mu$m produced by atomizers of different apex angles (a) 20° (b) 40° and (c) 60°.

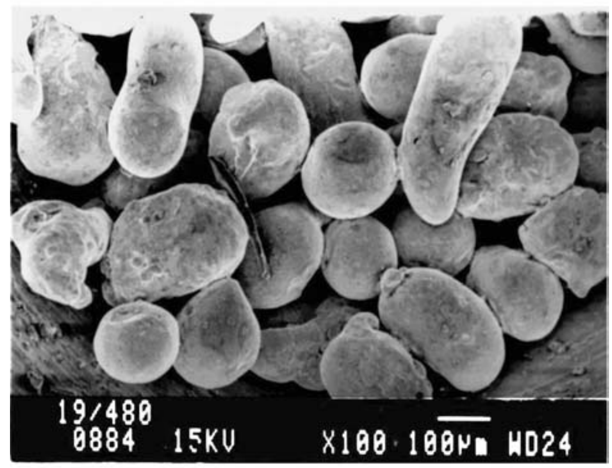
in the Figs b and c is more as compared to other shapes. Whereas proportion of rounded and irregular shape is more in Fig a, which is for atomizer of apex angle 20°.

### 3.1.2. *Effect of superheat of liquid metal*

Fig. 5a, b & c shows micrographs of lead powder particles (76–211 ($\mu m$) produced by an atomizer A 612 at




(a)

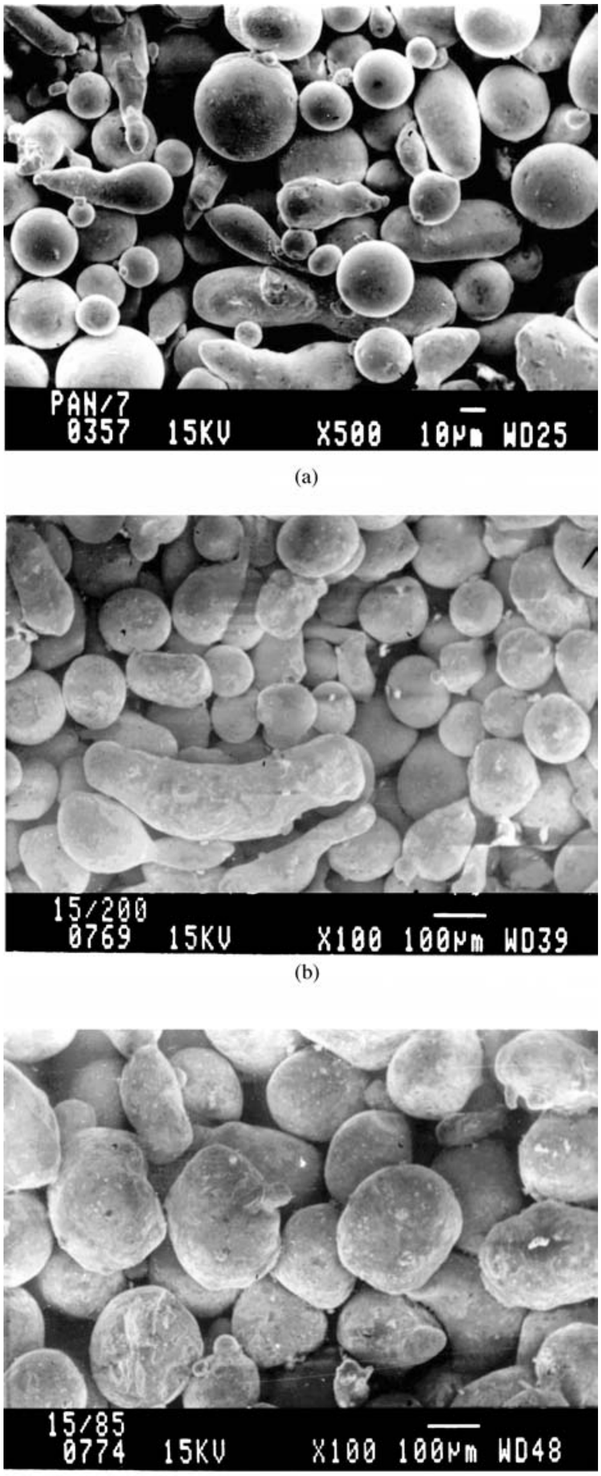



(b)


(c)

*Figure 5* SEM micrographs of the particles within the size range 76–211 $\mu$m produced at different superheats (a) 70 K (b) 150 K and (c)190 K.

different superheat of liquid metal. It can be observed that except at superheat of 150 K particles are rounded and irregular shape. Only at a superheat of 150 K large proportion of particles are spherical and remaining ones are rounded.



(a)

(b)

(c)

*Figure 6* SEM micrographs of the particles lying in different size range of a collective (a) < 38 $\mu$m (b) 105–153 $\mu$m (c) 211–300 $\mu$m.

### 3.1.3. Effect of particle size range

Fig. 6a, b & c shows SEM micrographs of lead powder particles in different size ranges viz. <38 $\mu$m, 105–152 $\mu$m and 211–300 $\mu$m. It can be seen in Fig. 6a that large proportion of powder particles in the size range < 38 $\mu$m is spherical. In the 105–152 $\mu$m range about equal proportion of spherical and rounded particles can

*Figure 7* SEM micrograph of the particles within the size range 76–211 $\mu$m of tin powder.

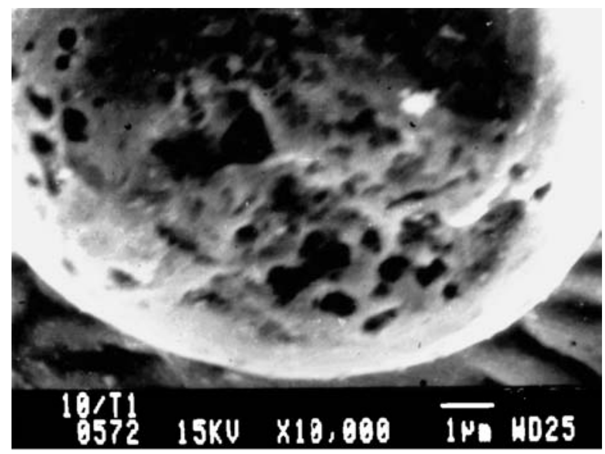


*Figure 8* SEM micrograph of the surface of a lead particle showing some black spots.

be seen in Fig 6b. As size range increased to 211–300 $\mu$m, Fig. 6c shows that few particles are nearly spherical whereas others rounded shape. Hence, the proportion of spherical particle in a size range is decreasing by increasing the particle size.

### 3.1.4. Effect of type of metal

Fig. 7 show micrograph of tin powder produced at superheat of 150 K by an atomizer A212. Most of the particles can be seen to be irregular and rounded. Similar results were observed for powders of aluminum and zinc.

All other parameters like plenum pressure, diameter of delivery tube, number of nozzles, apex angle etc were not observed to show any effect on particle shape of produced powder. All collectives were found to have mixed shape particles viz. very few spherical and remaining rounded and irregular shape.

## 3.2. Surface porosity and shrinkage

Fig. 8 shows SEM micrograph of the surface of a lead particle. It can be observed that there are some dark black spots on the micrograph. These spots show the presence

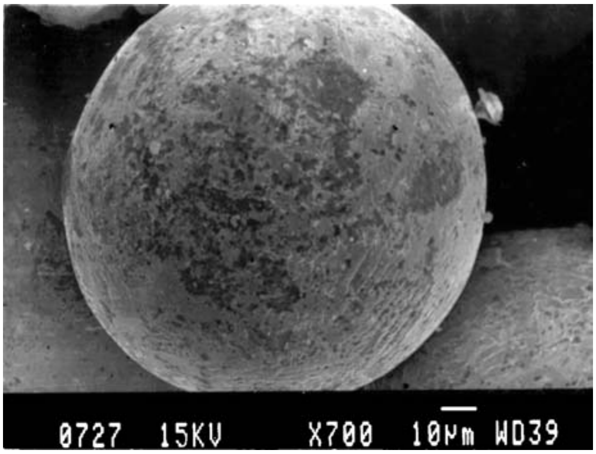



*Figure 9* SEM micrograph of the surface of a lead particle showing some waviness.

of porosity on powder particle surface. Similar type of porosity was observed on powder particles produced at different atomization parameters.

Fig. 9 shows the surface of a particle which is rough and has some waviness. This type of surface may be due to the solidification shrinkage of lead which indicate that the liquid metal existing at the surface solidify at the end of solidification process. The large size particles (>300 $\mu$m) were not observed to have waviness at the surface and these also were not spherical.

### 3.3. Solidification distance of spherical shape droplets

Solidification distances of spherical shape droplets of various sizes were determined by observing the powder particles collected on six conical trays (Fig. 2) by SEM and using the following analogy:-

According to observations reported for Fig. 9, it appears that particles are solidifying from center to surface. This mode of solidification gives enough time for droplets to spherodize before the completion of solidification. Since the droplet is solidified and spherodized, surface of the droplet should be free from cracks. On the other hand, the presence of any crack on the surface of the spherical shape particle indicate that the particle is partially solidified and the crack is formed due to hitting of such particles on the surface of the powder collection tray. Thus, if a powder collective from a tray is observed under SEM, then by observing the spherical particles without and with surface cracks, one can find the spherical particle of maximum size which has been solidified up to that tray under the chosen experimental conditions. The solidification distance for this size of particle will be equal to its flight distance up to its collection tray.

Fig. 10a is a SEM micrograph of the maximum size spherical shape particle (109 $\mu$m) without any crack and Fig. 10b of the particle with a surface crack as found on a tray positioned at a distance of 400 mm from the geometric point. This powder was produced at 1000 kPa using A412 atomizer. The spherical shape of this particle

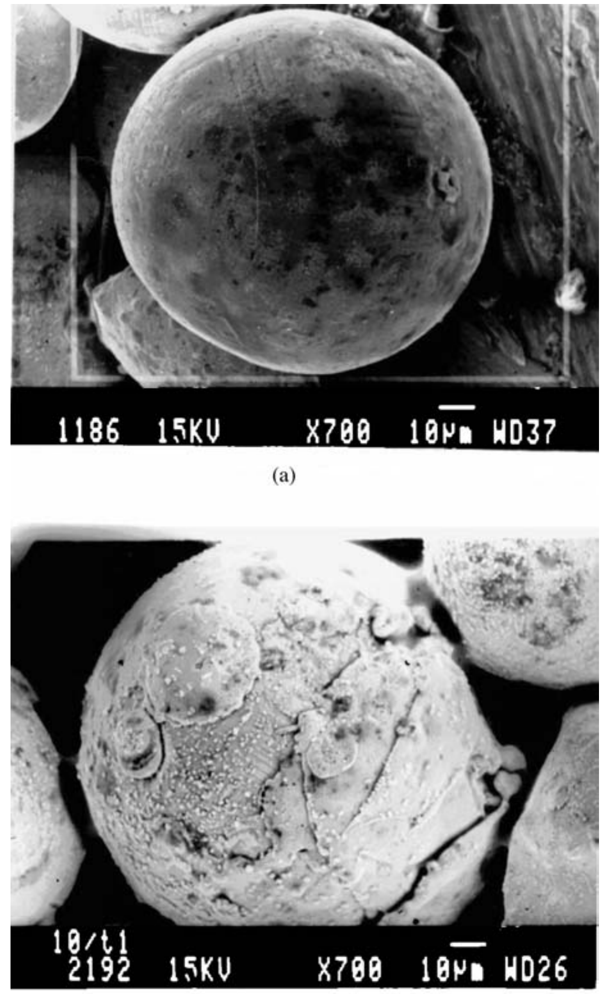



*Figure 10* SEM micrographs of particle found on first tray (a) Spherical particle of maximum size (b) Particle showing crack.

appears to be distorted on hitting the tray, which is an evidence of incomplete solidification. The average size of the particle is 115 $\mu$m. All the particles greater than 109 $\mu$m were found to be like Fig. 10b.

Similarly, the sizes of maximum size spherical particle collected on six conical trays were determined for the powder produced at 1000, 1500 and 1900 kPa using an atomizer A412. The size of aforementioned particles produced at 1000 and 1500 kPa plenum pressure using atomizer A612 and collected on six conical trays were also determined. The solidification distance of these particles was determined as follows: -

Six trays intercepted the liquid metal droplets, moving with the gas stream, along the horizontal plane $N_1Q_1$, $N_2Q_2$, $N_3Q_3$, $N_4Q_4$, $N_5Q_5$ and $N_6Q_6$ as shown in Fig. 11. Let us take the example of the first tray. The solidification distance for the maximum size spherical particle obtained on the tray would be in between the minimum distance $GM_1$ and the maximum inclined distance $GN_1$. Since motion of particle is not known, hence a mean value of $GN_1$ and $GM_1$ was taken as the solidification distance. Similar procedure was followed for other trays.

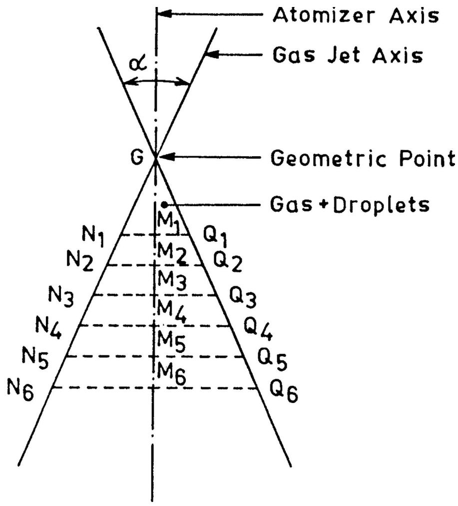



*Figure 11* Schematic sketch of the scheme of calculation to determine solidification distances of metal droplets moving in the gas stream.

## 4. Discussion

Results of present study show that the shape of particles of a powder collective produced by a free fall atomizer depends on superheat of liquid metal, apex angle of the atomizer and type of metal. Shape was found to be independent on other process parameters. Large proportion of lead particles were found spherical below the size of 211 $\mu$m of different powder collectives produced by atomizers of either 40° or 60° apex angle at superheat of 150 K. Other then 150 K superheat of liquid metal i.e. 70 K, 110 K and 190 K the large proportion of particles of powder collectives were found to be rounded and irregular in shape in all size ranges. Whereas large proportion of particles of powder collectives in all size ranges produced by atomizer of 20° apex angle were observed to be rounded and irregular in all size ranges at all superheats. The observation of the spherical shape of the particle in the powder collectives produced at 150 K superheat is explained by considering the time for spherodization and solidification. It is known that the shape of particle is controlled by the solidification and spherodization time of the droplet [12]. If spherodization time is less than that of solidification time, the resulting particle will be spherical in shape. Nichiporenko and Naida [14] have proposed the following equation to calculate the time of spherodization '$t_{\text{sph}}$' of the ligament into spherical shape droplet: -

$$t_{\text{sph}} = \frac{3\pi_m^2}{4V\sigma_m}\left(R_p^4 - r_p^4\right) \tag{1}$$

The $r_p$ is the radius of the ligament prior to and $R_p$ is the radius after spherodization. $V$ is the volume of the droplet which is equal to $\frac{4}{3}\pi\ R_p^3$. The $\mu_p$ and $\sigma_p$ are viscosity and surface tension of liquid metal, respectively. Assuming the value [15 ] of $r_p = 0.1\,R_p$, Eq. (1) reduces to

$$t_{\text{sph}} = 0.883\frac{m}{\sigma_m}d_p \tag{2}$$

TABLE II. Spherodization and solidification time of particles produced at 1500 Kpa plenum pressure

| Particle Size ($\mu$m) | Spherodization time ($\mu$s) | Solidification time ($\mu$s) |
|---|---|---|
| 95 | 0.47 | 11.1 |
| 133 | 0.66 | 20.7 |
| 160 | 0.80 | 29.7 |
| 233 | 1.16 | 61.8 |
| 243 | 1.21 | 66.9 |
| 252 | 1.26 | 71.7 |

Where, $d_p$ ($=2\,R_p$) is the diameter of the droplet. According to Eq. (2), time for spherodization increases with the decrease in surface tension but with the increase in viscosity and droplet diameter. Thus $t_{\text{sph}}$ for a given diameter of droplet can be determined from the physical properties of lead. The calculated values of $t_{\text{sph}}$ are given in Table II.

The time required for the droplet to reach collection tray of known distance was calculated by analyzing equation of motion of the droplet as given elsewhere [16, 17]. Since the droplet was solidified at that distances, hence this time is the solidification time. The so determined time of solidification ($t_{\text{sol}}$) is also given in Table II for powder produced at plenum pressure of 1500 kPa. It can be seen that solidification time (or cooling rate) heavily depends on particle size [18, 19] and $t_{\text{sph}} < t_{\text{sol}}$, which suggested that large proportion, if not all, of the particles should be spherical.

For aluminum, zinc and tin and at all other combinations of superheats for lead i.e. 70, 110 and 190 K and apex angles i.e. 20°, 40° and 60°, the shape of powder particles was found to be irregular. It suggests that there are also other factors which affect the shape of powder particles. The first factor is the initial shape of ligament after primary break up and the second factor could be the oxidation of metals. Nichiporenko and Naida [14] indicated that an increase in gas velocity relative to the droplet velocity would tend to elongate the initially formed cylindrical droplet. More initial elongation of droplet means it would take more time to spherodize and hence more chance to have non-spherical particles. In previous study [1] it has been observed that apex angle has no effect on gas velocity. Therefore, apex angle should not have effect on the shape of powder particles. But, in present study powder produced by atomizer of 40° and 60° apex angle was found to have more spherical particles (Fig. 4b & c). It indicates that apex angle also has effect on initially formed cylindrical droplets (formed after primary break up). The cylindrical droplets initially formed by the atomizer of 20° could be more elongated [20]. More elongated droplets shall take more time to spherodize and hence more particles will be non-spherical produced by this atomizer. Other metals (aluminum, zinc and tin) are more prone to oxidation during atomization, which will increase the spherodization time of droplets [21–23] and hence particles will be non-spherical.

In case of lead more spherical particles were observed at superheat 150 K as compared to 70, 110 and 190 K. As superheat decreases, the viscosity of liquid metal increases and hence the spherodization time of droplet increases (Eq. 1). Therefore, at superheats below 150 K, particles were found to be having irregular shape. Also, by increasing the superheat, the oxidation of metal increases which hinders the process of spherodization of droplets. Hence, at higher superheats (190 K) particles were found irregular.

Thus it appears that superheat and apex angle determines the dynamics of solidification of the droplets in order to obtain the final powder shape. Large size particles were found to have irregular shape whatever be the atomization parameters. Thus, both the mechanism of solidification of the droplets i.e. from surface to center and center to surface are important in order to obtain the desired powder shape. The former mechanism appears to produce irregular shaped powder (as observed for large size particles) since surface of the droplet is already solidified, and the later mechanism produces spherical powder since enough time is available for spherodization.

## 5. Conclusions

Shape of the powder particle was observed to depend on superheat of liquid metal, apex angle of atomizer, type of metal and size of particle for all plenum pressures of gas. At 70, 110 and 190 K superheats, and 20°, 40° and 60° apex angles, the shape of most of the lead particles was found to be irregular, rounded etc, whereas at 150 K superheat and 40° and 60° apex angles, a large fraction of the particles in the powder collectives was found to be spherical in shape. Tin, zinc and aluminum powder particles were found non spherical for all process parameters. The porosity and surface shrinkage were observed at the surface of almost all particles except that of large size range particles which were not observed to have solidification shrinkage at the surface.

## References


1. D. SINGH, S. C. KORIA and R. K. DUBE, *Powder Metall.* **42** (1) (1999) 79.
2. S. A. MOIR and H. JONES, *Mater. Sci. Eng.* **A 173** (1993) 161.
3. S. A. MOIR, H. JONES and S. B. M. BECK, *Powder Metall.***39** (4) (1996) 271.
4. D. SINGH, S. C. KORIA and R. K. DUBE, *ISIJ Int.* **43** (12) (2003) 2067.
5. G. HELMERSSON and KLAAS BURGDORF, *Scandinavian, J. Met.* **25** (1996) 51.
6. Y. LIU, K. MINAGAWA, H. KAKISAWA and K. HALADA, *Int. J. Powder Metall.* **39** (2) (2003) 29.
7. S. OZBILEN, A. UNAL and T. SHEPPARD, *Powder Metall.* **34** (1) (1991), 53.
8. G. HELMERSSON, A. HEDE, T. JOHANNESSON, B. BERGMAN and H. HALLEN, *Scandinavian, J. Met.* **26** (1997) 93.
9. A. UNAL, *Metall. Trans.B.* **20B** (2) (1989) 61.
10. A. UNAL, *Powder Metall.* **33** (1) (1990) 53.
11. S. OZBILEN, A. UNAL and T. SHEPPARD, *Powder Metall.* **39** (1) (1996) 44.
12. J. B. SEE and G. H. JOHNSTON, *Powder Technol.* **21** (1978) 119.
13. A. LAWLEY, in "Atomization: The production of metal powders" (MPIF, Princeton, NJ, 1992) p. 83.
14. O. S. NICHIPORENKO and YU. NAIDA, *Sov. Powder Metall. Met. Ceram.* **7** (67) (1968) 509.
15. E. KLAR, in Metals Handbook, vol 7 (Powder Metallurgy, 1984) p. 75.
16. E.G. MIRAVETE and E. J. LAVERNIA, in Physical Chemistry of Powder Metals Production and Processing edited by W. MURRAY SMALL, (Minerals metals and Materials Society, 1989) p. 175.
17. D. CHENG, Q. XU, G. TRAPAGA and E. J. LAVERAIA, *Metall. Mater. Trans. B.* **32B** (6) (2001) 525.
18. Z. LIU, *Powder Metall.* **45** (2) (2002) 134.
19. N. H. PRYDS and A. S. PEDERSEN, *Metall. Mater. Trans. A.* **33A** (12) (2002) 3755.
20. D. SINGH, S. C. KORIA and R. K. DUBE, *Powder Metall.* **44**, (2) (2001) 177.
21. S. OZBILEN, *Powder Metall.* **43** (2) (2000) 173.
22. S. OZBILEN, *Powder Technol.***102** (1998) 109.
23. S. OZBISLEN, *Powder Metall.* **42** (1) (1999) 70.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