Particle Size Distribution of Tin Powder Produced by Centrifugal Atomisation Using Rotating Cups

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Abstract

Centrifugal atomisation is a low-cost technology for producing metal powders, but its wide applications are hampered by its limited capability of producing fine powders and there is insufficient research on the particle size distributions of centrifugally produced powders. This paper studies the effects of the atomiser geometry and the key process parameters on the particle size distribution of tin powders produced by centrifugal atomisation. The results showed that the particle sizes of the as-produced powders follow lognormal distribution. The median particle size for all atomisers decreases with increasing atomiser rotation speed and with decreasing melt flow rate, due to reduced film thickness of the melt before disintegration. The cup atomiser with a wall angle of 67.5° produced the finest powders, because of a significant improvement of dynamic wetting between the melt and the atomiser. The particle size distributions of all the powder samples have a similar lognormal bell shape with the geometric standard deviations between 1.6 and 2.5. Narrow particle size distributions can be achieved by reducing the variability of any of the processing parameters affecting the particle size.

Key words: centrifugal atomisation, powder, particle size distribution, atomiser.

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Introduction

Centrifugal atomisation has been used for producing metal powders for several decades [1]. Its main advantages as an industrial scale technology are low production cost and high productivity for producing powders of relatively large particles. Recent applications include uranium fuel powders [2, 3] and solder powders [4, 5]. However, centrifugal atomisation is not as widely used in industry as the gas and water atomisation techniques, because of its limited capability of processing high-melting-point metals and producing fine powders. Several technical challenges in the design of centrifugal atomisers need to be overcome [6]. Firstly, a very high rotation speed is required to generate centrifugal forces great enough to break up the liquid metal into droplets in the desirable size range of 20 – 50 μm. This high rotation speed is often practically difficult to achieve due to the lack of robust high speed motors free of vibration problems. Secondly, the atomiser must be able to withstand the elevated temperature of the liquid metal for long periods of time and therefore needs to be water-cooled for most liquid metals. From the design and operational point of view, water cooling of a rapidly moving part is undesirable. Thirdly, a bulky chamber is required for the solidification of the high-velocity liquid metal droplets. Tian *et al* [7] have recently designed and built a centrifugal atomiser to address these issues. They used an externally mounted router motor that was magnetically coupled to the atomiser to provide a rotational speed up to 24,000 rpm and used a rotating quench bath filled with oil to provide efficient cooling of the liquid droplets. However, further technical development and scientific understanding of the centrifugal atomisation process are still necessary before it becomes a more competitive metal production process.

Hinze and Milborn [8] studied atomisation of liquids from a rotating cup and identified three modes of liquid disintegration: direct droplet formation (DDF), ligament disintegration (LD), and film disintegration (FD). In the DDF regime, the droplet sizes of some metals may be estimated theoretically from the atomising conditions [9]. The droplet size distributions become increasingly poly-disperse as the disintegration mechanism moves to the LD and FD regimes. Under the atomisation conditions normally used in the metal powder production, the disintegration mechanism is either LD or a combination of DDF, LD and FD [10]. Zhao [11] attributed the occurrence of the three atomisation modes to incomplete wetting between the liquid metal and the atomiser and developed an analytical model to illustrate the effects of process conditions on the transition from one disintegration regime to another. Better wettability between the liquid metal and the atomiser leads to a thinner continuous film before its disintegration into ligaments and subsequently droplets, and therefore results in finer droplets.

Several quantitative models have been developed to calculate the liquid velocity and film thickness on centrifugal atomisers [12, 13] and the droplet size formed in the DDF [14], LD [15] and FD [16] regimes. These models assume simple atomiser geometries and perfect wetting conditions and often require numerical solutions. Although they offer some valuable insights into the atomisation process, their applications are limited. This is mainly because the flow conditions on a practical centrifugal atomiser are complicated by hydraulic jump [12, 17], skull formation [10, 12, 18] and coating material [19, 20]. The particle size distribution of centrifugally atomised powders has to be determined experimentally.

The effects of process conditions on the particle size of centrifugal atomised powders are well documented in the literature. The particle sizes normally follow a lognormal distribution, and lower melt flow rate, higher atomiser rotation speed and larger atomiser diameter result in smaller powder particles [10, 21]. Various types of atomisers, including flat discs, vaned wheels, inverted bowls and cups, have been used [22]. Although atomisers with complex design and shape offered no significant benefits under certain atomising conditions compared with a simple flat disc [23], cup shaped atomisers often produced finer powders than flat discs [5, 10, 24, 25]. Cup atomisers can improve the wetting between the liquid and the atomiser. They also make it easier to obtain a symmetrical feed of melt, especially at high melt flow rates. For many applications, cup atomisers are the best choice. However, the research on atomiser design is still inadequate and inconclusive, largely because it is difficult to isolate the effect of atomiser design from so many other factors that may affect the performance of atomisation. The effect of cup designs, e.g., cup wall angle, on the characteristics of the particle size distribution, including median and spread, is not fully understood.

This paper carries out an experimental study on the particle size distribution of centrifugally atomised tin powders using cup-shaped atomisers with different cup wall slope angles. The effects of the key process parameters, i.e., atomiser rotation speed and melt flow rate, on the particle size distribution are studied. The spread of the particle size ranges are characterised and the key parameter determining the particle size is discussed.

Experimental

The centrifugal atomisation apparatus and the operational procedures used for the experiments was described in detail in the previous paper [10]. The atomisers used in the study were cylindrical steel cups with a diameter of 8 cm. Each cup had a flat bottom and a sloped conical wall at the circumferential edge. A series of cups with different wall slope angles of 45°, 67.5° and 90°, as shown schematically in Fig. 1, were used. They were all pre-tinned in order to maximise the wetting with the melt during atomisation. In each experiment, commercially pure tin was first melted in a furnace at a temperature of 550° to ensure a high superheat. The melt was then poured into a tundish with an exchangeable bottom nozzle. The melt flow rate was determined by measuring the mass of the melt in the tundish and the time required for its discharge through the nozzle under gravity. The cylindrical nozzles had diameters of 2, 3 and 4 mm, corresponding to melt flow rates of approximately 65, 150 or 220 kg/h, respectively. The melt flowed through the nozzle under gravity onto the centre of the rapidly rotating cup, positioned 1 cm below the nozzle, and was broken up by the cup into a horizontal spray of droplets. A part of the spray was allowed to fly into a powder collection chamber, which was long enough to ensure that the vast majority of the droplets solidified during flight to form solid particles. The as-produced powder sample (Fig. 2) was finally collected and subsequently analysed by a series of sieves.

Results and Discussion

Figs. 3-5 show the lognormal plots of the accumulative weight percentage of the particles under a specific particle size versus the particle size for the powder samples produced using 45°, 67.5° and 90° atomising cups, respectively, with different atomiser rotation speeds and melt flow rates. All the plots are linear in the lognormal scale, with the coefficients of determination $R^2 > 0.94$.

The results show that the particle sizes of the powders produced by centrifugal atomisation using cups follow the lognormal distribution. As flat disc atomisers also produced powders with lognormal distributions [10], it is demonstrated that lognormal distribution applies to all centrifugally atomised powders, regardless of the atomiser used. A log normal distribution results if the variable is the product of a large number of independent variables. This is clearly true in centrifugal atomisation, where the particle size is affected by many independent variables or processing parameters, including the physical properties and flow rate of the melt, the geometry, surface condition and rotation speed of the atomiser. Each of these variables can fluctuate due to changes in the processing conditions, e.g., melt temperature, atomiser vibration and other operating variations.

Fig. 6 shows how the median (or geometric mean) particle size of the powder samples produced using a flat disc [10] and cups varies with atomiser rotation speed and melt flow rate. The values of the median particle size, D_{50}, which corresponds to the accumulative weight percentage of 50%, were obtained from the fitted lines in Figs. 3-5. Some clear trends can be observed from the results. For all atomisers, the median particle size decreases with atomiser rotation speed for any given melt flow rate. The median particle size generally increases with melt flow rate, but the magnitude of the effect of melt flow rate depends on the atomiser rotation speed. At a low rotation speed of 6000 rpm, an increase in melt flow rate results in a significant increase in the median particle size. At higher rotation speeds, increasing melt flow rate produces a less significant effect on the particle size. For each rotation speed, there seems to be a critical melt flow rate below which particle size does not decrease with decreasing flow rate. In other words, there exists a minimum particle size achievable for each rotation speed. Overall, the particle sizes obtained in the present work are comparable with those reported in the literature for low-melting-point metals. The median particle sizes in the present work are in the range of 50-250 μm, while those for a tin-lead alloy (61% Sn + 39% Pb) [4], tin-silver-copper alloy (96.5% Sn + 3% Ag + 0.5% Cu) [5] and commercially pure zinc (100% Zn) [25] were in the range of 36-115 μm, 50-150 μm and 140-300 μm, respectively.

The design of the atomiser can have a considerable effect on the median particle size, as shown in Fig. 6. The effect of the angle of the atomising cup wall on particle size of the as-produced powder can be examined by comparing Figs. 6(a), (b), (c) and (d). The 45° cup has a similar performance as the flat disc. The median particle sizes of the powders produced by the 45° cup are similar to those produced by the flat disc, for all the combinations of atomiser rotation speed and melt flow rate. The performance of the 90° cup at the low flow rate of 65 kg/h is similar to the flat disc and the 45° cup. At the higher flow rate of 220 kg/h, however, the median particle sizes of the powder samples produced by the 90° cup are 20% to 30% lower than those produced by the flat disc and the 45° cup under the same processing conditions. In comparison, the 67.5° cup has a far better performance for all conditions, producing powder particles 40% to 60% smaller than those produced by the flat disc and the 45° cup. Among the conditions studied in this paper, the finest powder was produced by the 67.5° cup at a rotation speed of 12000 rpm, having a median particle size of 47 μm.

Liu *et al* [15] developed a numerical model to calculate the droplet size in centrifugal atomisation, based on an analysis of the liquid disintegration using the wave theory. They showed that the droplet size is directly related to the thickness of the liquid film at the edge of the atomiser, predicted by the analytical model developed by Zhao [13]. Mantripragada and Sarkar [26] calculated the thickness of the liquid film at the edge of a rotating disc using CFD modelling and also showed that the drop size has a linear relation with the liquid film thickness. The effects of atomiser rotation speed and melt flow rate on the particle size can be explained by their effects on the liquid film thickness.

Fig. 7 correlates the median particle size with the thickness of the tin melt film off the edge of the atomiser calculated by Zhao's model [13]. The physical properties and process parameters used in the film thickness calculations were: melt density 6970 kg/m^3, viscosity 0.00197 kg/m/s [1], atomiser radius 0.04 m, melt flow rates 65, 150 and 220 kg/h (2.6×10^{-6}, 6.0×10^{-6} and

8.8×10^{-6} m^3/s, respectively), atomiser rotation speeds 6000, 9000, 12000 and 15000 rpm (628.3, 942.5, 1256.6 and 1570.8 radian/s, respectively).
Fig. 7 shows that there exists a linear relationship between the median particle size and the theoretical film thickness. The predicted film thickness decreases with decreasing melt flow rate and with increasing atomiser rotation speed [13]. The variations of the median particle size with melt flow rate and atomiser rotation speed are therefore follow the same trends. It should be pointed out that the measured median particle sizes are greater than the corresponding predicted film thicknesses by one to two orders of magnitude. This is because the melt film has very likely disintegrated on the atomiser well before reaching the atomiser edge [11] under the process conditions studied in this paper.

The effect of atomiser design on particle size cannot be explained by its effect on film thickness alone. The thickness of the liquid film at the edge of the atomising cup predicted by the model in [13], assuming no premature film disintegration, is not sensitive to cup wall angle under the atomisation conditions studied in this work. The predicted film thickness increases only slightly with cup wall angle, especially at low (65 kg/h) and medium (150 kg/h) melt flow rates. If the melt remains a complete liquid film on the atomiser, the droplet size would be expected to be proportional to the thickness of the film at the edge of the atomiser [15, 26] and as such cup atomisers with different wall angles would produce similar droplet sizes as a flat disc atomiser. The experimental findings made by Ahmed and Youssef [23] supported this argument. They studied atomisation of water, which has a good wettability with most solids, and found that varying cup wall angle had little effect on the droplet size.

The effect of a cup atomiser on droplet size is likely to result from its effect on the liquid disintegration mechanism. A cup shaped atomiser can increase spreading of the melt on the atomiser surface, which is especially important for liquids with poor wettability such as metals. With a sloped cup wall, the centrifugal force exerts a pressure in the liquid film towards the cup wall, promoting dynamic wetting between the melt and the atomiser [6]. This will delay the disintegration of the film into ligaments and further breakup into drops [11]. The radius at which the uniform melt film on the cup starts to disintegrate into ligaments is very sensitive to cup wall angle [11]. In other words, the mechanism by which the liquid disintegrates can change if the cup wall slope angle is changed. Reliable data for dynamic wetting angle is not available for most liquid metals, because it is extremely sensitive to the physical properties and surface conditions of the solid to be wetted as well as to the environment of the measurement. It is therefore unrealistic to predict and difficult to measure the disintegration radius. However, the link between wettability and disintegration mechanism can provide a useful insight into the effect of cup wall angle.

The centrifugal force, F, generated by a rotating cup with a wall angle of θ, can be resolved into a component in the slope direction, $F\cos\theta$, and a component in the normal direction to the cup wall. $F\sin\theta$. The slope direction component provides a driving force for the ejection of the liquid from the atomiser. The normal direction component generates a force pressing the liquid towards the cup wall. On a cup with a steep slope or large wall angle (e.g., $\theta = 67.5°$), the liquid is subjected to a large force towards the cup wall and a small but sufficient force for its ejection. The large pressure against the cup wall results in a good spreading of the liquid film and delays the disintegration of the film into ligaments. The film thickness at the point of disintegration is effectively reduced and, as a consequence, finer droplets are formed. A cylindrical cup with a vertical wall ($\theta = 90°$) can maximise liquid wetting but the liquid experiences no centrifugal force in the slope direction. The ejection of the liquid is caused by the internal pressure alone. The film thickness at the cup edge can fluctuate and can also be significantly larger than a sloped cup. As a result, a vertical-walled cup often has worse performance than a steep-sloped cup, i.e., produces coarser droplets. For a flat disc or a cup with a relatively small wall angle (e.g., θ = 45°), either there is no force or the force is too small to press the liquid against the atomiser. The low-wettability liquid film will break up at a small radius, well before reaching the edge of the atomiser. The liquid film thickness at the point of disintegration is still large, leading to coarse droplets. The experimental work by Ahmed and Youssef [23] on the slip between the liquid and the rotating surface of the atomiser shed some light on the effect of liquid spreading from a different perspective. They showed that the effect of cup wall angle on liquid slippage depends on the cup diameter and, for the same diameter, cups with small and intermediate wall angles have similar or even larger slip ratios than a flat disc, while cups with large wall angles generally have reduced slip ratios. Overall, for the atomisation of low-wettability liquid, there is an optimum wall angle at which the finest droplets can be produced due to good liquid spreading and a low liquid film thickness prior to disintegration.

While the median particle size is a key parameter for a powder, the shape and spread of the particle size distribution are as important in describing the characteristics of the powder. Table 1 lists the geometric standard deviation and coefficient of variation (in brackets) data for particle size distributions of the powders produced with different atomisers, atomiser rotation speeds and melt flow rates. In lognormal distribution, the geometric standard deviation, σ^*, describes the dispersion or spread of data and 68% of the data values fall in the range [D_{50}/σ^*, $D_{50}\cdot\sigma^*$]. σ^* is the ratio between the median particle size, D_{50}, and the particle size corresponding to the accumulative weight percentage of 16%, which can be easily obtained from the fitted lines in Figs. 3-5. The coefficient of variation (CV), i.e., the ratio of the standard deviation to the arithmetic mean, is an alternative measure of spread. In lognormal distribution, it can be obtained from the geometric standard deviation by

$$CV = \sqrt{\left(e^{[ln(\sigma^*)]^2} - 1\right)}$$

Geometric standard deviation and coefficient of variation describe the amounts of variability relative to the arithmetic and geometric means, respectively. Although the coefficient of variation is helpful for data interpretation, the geometric standard deviation provides more useful information in terms of the dispersion of the lognormal particle size distribution.

The values of the geometric standard deviation fall between 1.6 and 2.5 (CV 0.5-1.1), with most in the range 1.8 – 2.3 (CV 0.7-1). This indicates that the particle size distributions of all the powder samples have a similar lognormal bell shape. They are similar to many log-normal distributions from various branches of science, where most values of geometric standard deviation vary in the range of 1.4 to 3 [27]. The effects of the atomiser rotation speed, melt flow rate and cup wall slope angle on the values of geometric standard deviation and coefficient of variation are not substantial. However, a combination of a low atomiser rotation speed of 6000 rpm and a low melt flow rate of 65 kg/h appears to give relatively lower values of geometric standard deviation and coefficient of variation, i.e., narrower particle size distributions.

In lognormal distribution, the data are skewed with a long tail towards large values, and the greater the geometric standard deviation the longer the tail. This means that centrifugally atomised powders always contain considerable amounts of large particles. Assuming D_{50} = 100 μm and σ^* = 2, for example, 68.3 wt% of the particles would fall in the range 50-200 μm and 95.5 wt% in the range 25-400 μm. In other words, nearly 16 wt% of the particles would be greater than 200 μm and more than 2 wt% greater than 400 μm.

A small geometric standard deviation is desirable to reduce the dispersion or increase the uniformity of the particle size distribution. The multiplicative nature of variation of lognormal distribution means that reductions in the variability of any processing parameter that affects the particle size can result in significant improvements in particle size uniformity. This provides a useful guidance for process optimisation. For instance, the flow rate of the melt may fluctuate when it is poured from the tundish onto the atomiser. Reducing the distance between the tundish and atomiser may achieve a more steady flow rate and therefore potentially a narrower particle size distribution.

Conclusions

1) The particle sizes of the powders produced in centrifugal atomisation using flat disc and cup atomisers follow lognormal distribution.
2) The median particle size for all atomisers decreases with increasing atomiser rotation speed and with decreasing melt flow rate, largely due to the reduced film thickness of the melt on the atomiser before disintegration.
3) The cup atomiser with a wall angle of 67.5° produced the finest powders under the same melt flow rate and atomiser rotation speed, mainly because of improved dynamic wetting between the melt and the atomiser.
4) The particle size distributions of all the powder samples have a similar lognormal bell shape with the geometric standard deviation values falling between 1.6 and 2.5. A combination of a low atomiser rotation speed and a low melt flow rate resulted in narrower particle size distributions.

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Table 1 Geometric standard deviation and coefficient of variation (in brackets) data for particle size distributions of the powders produced with different atomisers, atomiser rotation speeds and melt flow rates

Rotation speed (rpm)	Melt flow rate (kg/h)									
	Flat cup#			45° cup			67.5° cup		90° cup	
	65	150	220	65	150	220	65	220	65	220
6000	1.61 (0.50)	1.72 (0.58)	2.19 (0.92)	1.65 (0.53)	2.16 (0.90)	2.47 (1.12)	1.88 (0.70)	1.96 (0.76)	1.89 (0.71)	1.96 0.75
9000	1.80 (0.64)	2.06 (0.83)	2.19 (0.92)	2.12 (0.87)	2.32 (1.02)	2.29 (0.99)	- -	- -	- -	- -
12000	1.79 (0.64)	1.98 (0.77)	2.14 (0.88)	2.22 (0.94)	2.27 (0.98)	2.24 (0.96)	2.47 (1.13)	2.15 (0.89)	2.27 (0.98)	2.10 (0.86)
15000	2.32 (1.02)	2.02 (0.80)	2.14 (0.89)	- -	- -	- -	- -	- -	- -	- -

Values extracted from the data in [10]

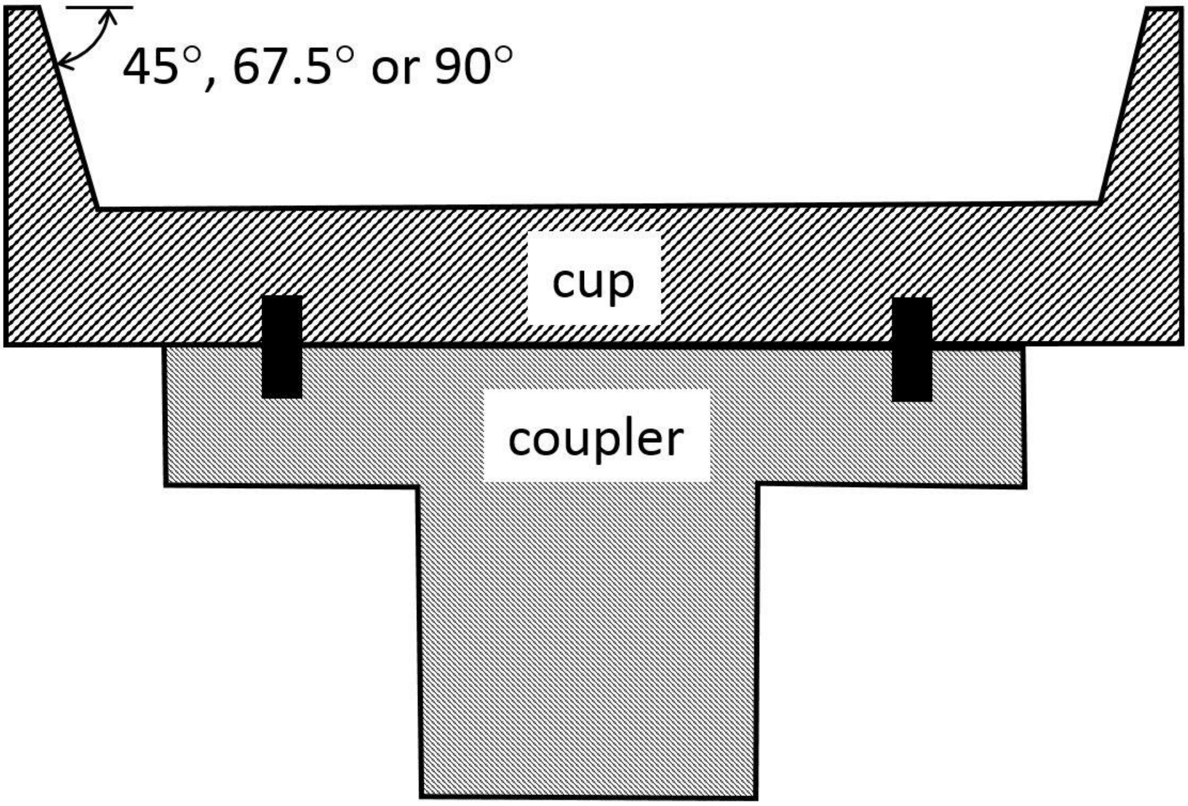

Fig. 1 Schematic diagram of the atomising cups.

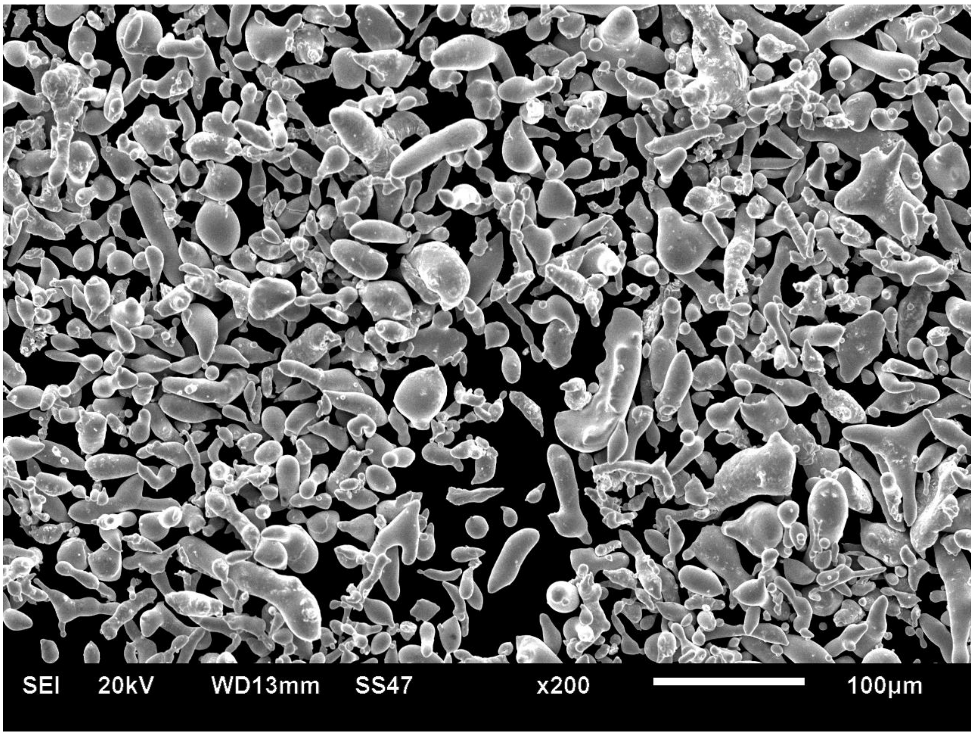

Fig. 2 SEM photograph of a typical tin powder, produced using the 67.5° cup with a rotation speed of 12000 rpm and a melt flow rate of 65 kg/h.

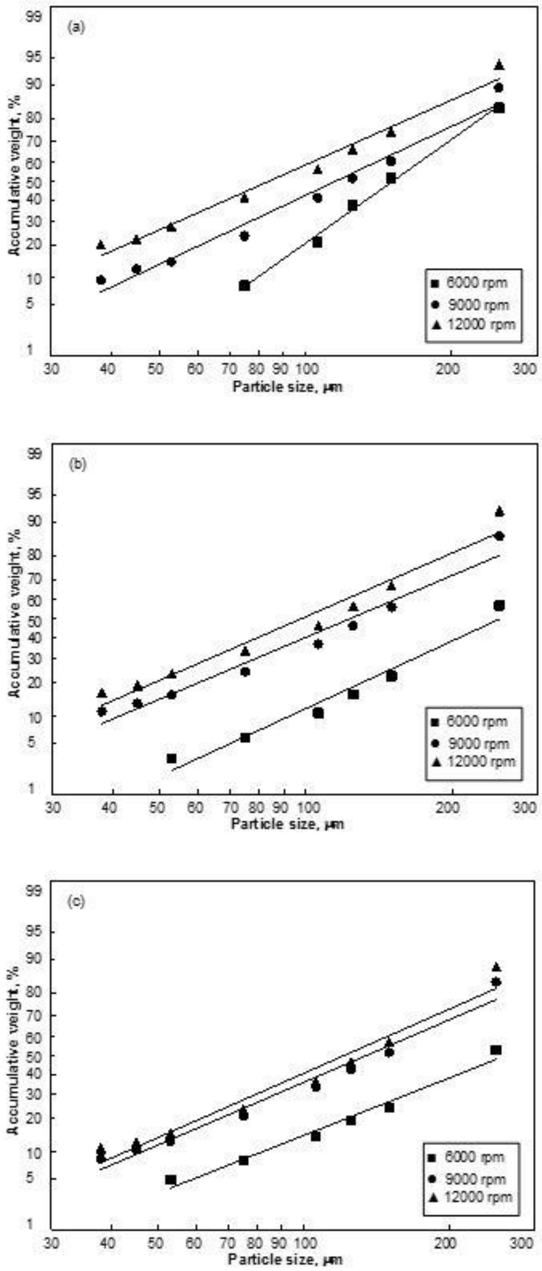

Fig. 3 Particle size distributions of the powder samples produced using a 45° cup at different rotation speeds for different melt flow rates: (a) 65, (b) 150 and (c) 220 kg/h.

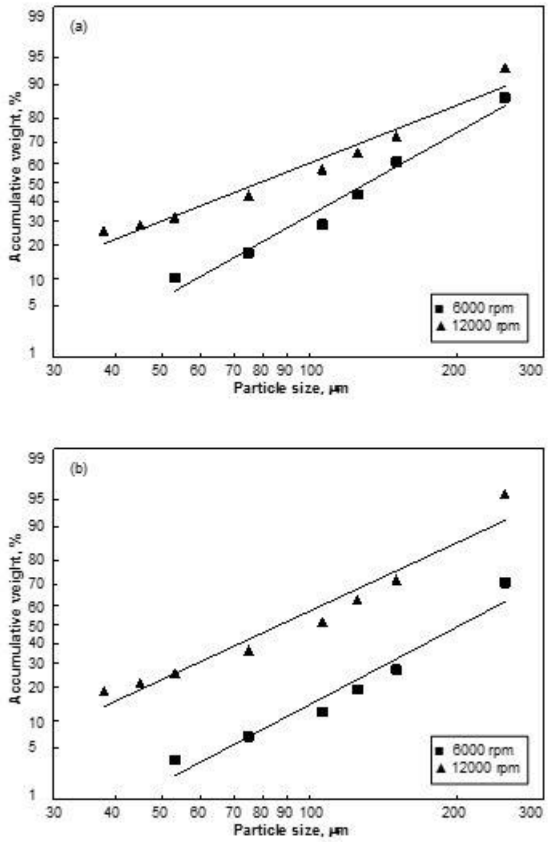

Fig. 4 Particle size distributions of the powder samples produced using a 67.5° cup at different rotation speeds for different melt flow rates: (a) 65 and (b) 220 kg/h.

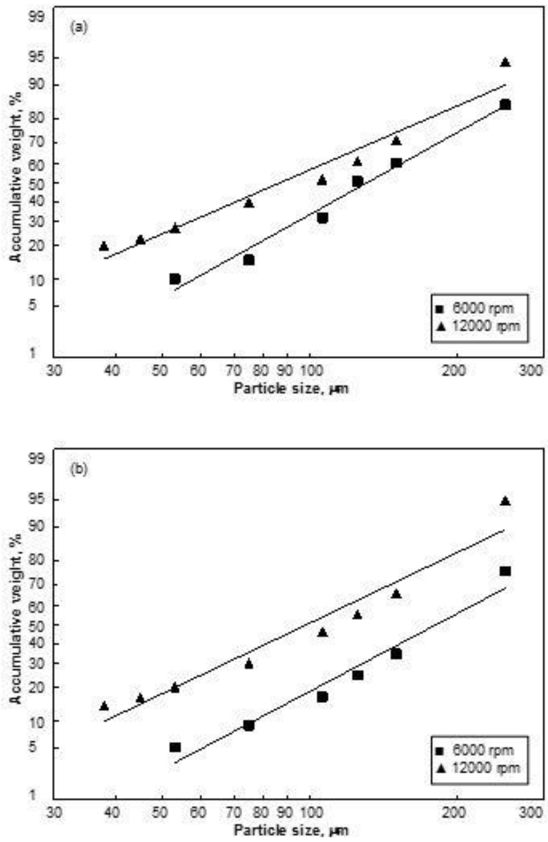

Fig. 5 Particle size distributions of the powder samples produced using a 90° cup at different rotation speeds for different melt flow rates: (a) 65 and (b) 220 kg/h.

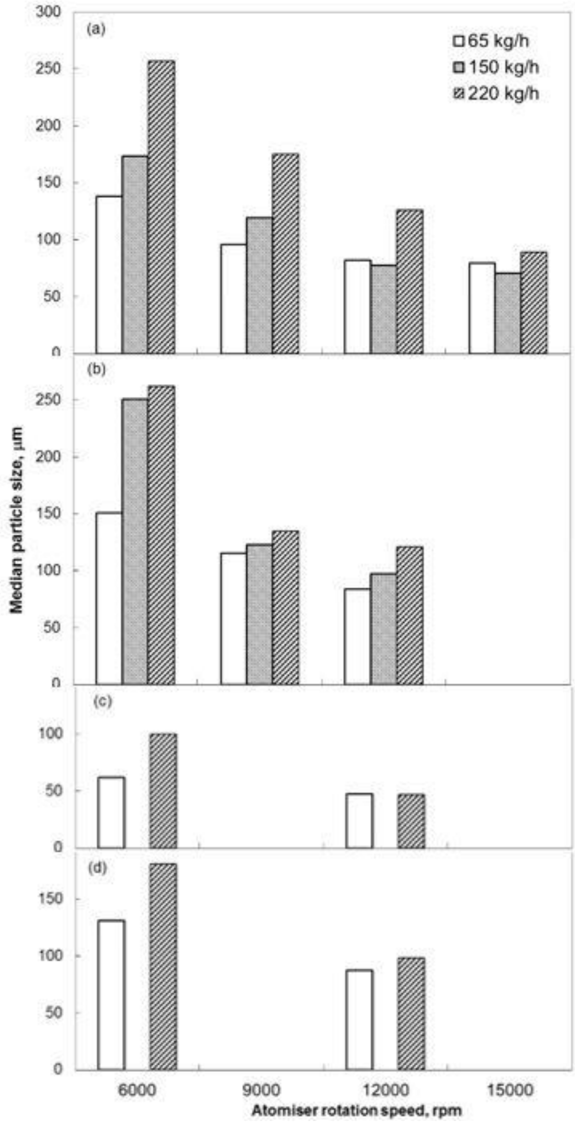

Fig. 6 Median particle sizes of the powder samples produced at different atomiser rotation speeds and melt flow rates using (a) a flat disc [10], (b) a 45° cup, (c) a 67.5° cup and (d) a 90° cup.

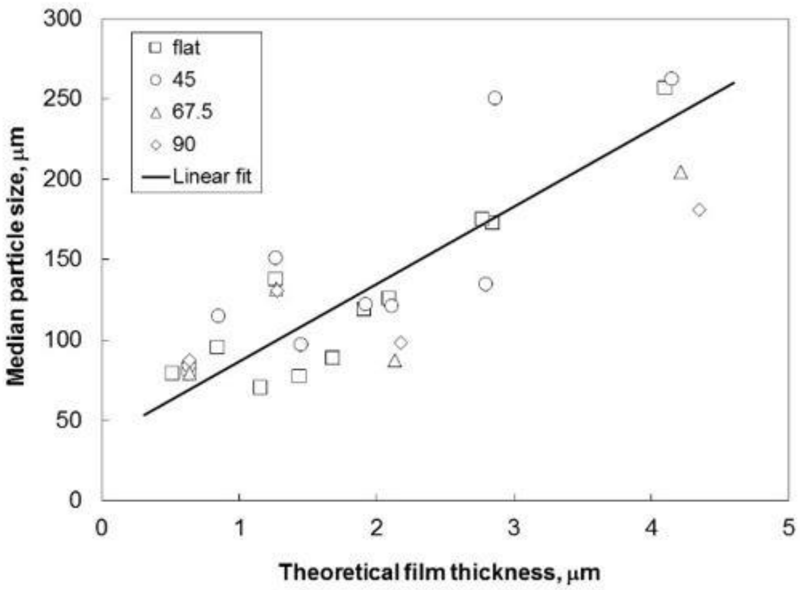

Fig. 7 Correlation between median particle size and predicted film thickness at the atomiser edge for different atomisers, rotation speeds and melt flow rates.