A new Technique for Molten Metal Atomization

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A new molten metal atomisation technique has been developed which combines a swirl jet pressure nozzle to generate a thin liquid film in a first step and a gas nozzle to atomise this film in a second step. The swirl jet pressure nozzle needs a very small amount of gas to protect the molten metal and the particles from oxidation. But the mean particle sizes which can be achieved are quite large. Therefore the gas nozzle was added to generate smaller particle sizes. Different spray pattern - using laser sheet illumination – from water atomization were achieved by changing the geometry of the gas nozzle. When the water exits the swirl nozzle it builds up a liquid film which is shaped as a hollow cone. This film is atomised by the gas flow which hits the film or the ligaments app 20 mm from the nozzle exit. Finally, molten tin was atomized with the chosen gas nozzle and the powder was analysed. The powder was analysed by different methods (sieving and laser diffraction) which lead to different results in mean particle sizes and particle size distribution. Those results will be discussed in detail. As well, results on specific gas consumption, particle size distribution, correlation of spray parameter and median particle size will be presented.

1. Introduction

Challenges in the growing metal powder market are to improve conventional atomization techniques and to develop new atomization processes with better productivity and economy. Water atomization [1] is one of the economical processes but this technique is not suitable for the production of spherical and clean powders. For this purpose, different techniques such as Ultrasonic Capillary Wave Atomization [2], Rotating Disk Atomization [3] and Gas Atomization techniques like Close Coupled Atomization [4] are used. The close coupled gas atomization technique is characterized by its high mass flow rate and therefore it is frequently used for production of metal powders with mass median diameter ranging from 10 to 100 µm. However, the particle size distribution is quite wide and the specific gas consumption is high. In addition, generally, only a fraction of the raw metal powder can be used as a product. Larger and smaller particle size ranges must be separated which, in turn, reduces the productivity of the process. A wide size distribution of the raw metal powder means a low productivity.

Recent developmental attempts [5] try to overcome the problem of wide size distribution for the closed coupled atomization. It was observed that the melt film at the nozzle tip was not stable. Therefore, a slotted nozzle was introduced. The slots at the nozzle tip keeps the thin film of the melt more stable and leads to better results in the particle size distribution.

This paper presents a new atomization technique [6], which combines the Centrifugal Hydraulic Atomization (CHA) /7/ and gas atomization. Here, a conical molten metal film is atomized by high velocity gas jets. In absence of the atomizing gas the melt also will atomize, but the mean particle size would be quite large (d_{50}>200µm). Smaller mean particle sizes with a narrow size distribution and low specific gas consumption are the main goals of this technique. A comparison with other atomization techniques would be desirable, but this has to be done very carefully. Data from literature cannot be used for comparison because the raw powder has to be analyzed with the same equipment. This problem will be discussed in light of the results from this investigation.

2. Principle of the new atomization technique

The new atomization technique uses a conventional pressure nozzle [7]. A pressure difference of up to 1.0 MPa pushes the molten metal into a swirl chamber and the melt flows in the swirl chamber until it is pressurized to leave the pressure nozzle through a small cylindrical hole. At the exit, a conical thin film is generated due to centrifugal forces (fig. 1a). The film thickness decreases in flow direction. Figure 1b shows a photograph of the CHA process, when the melt leaves the pressure nozzle. After 10 to 15 mm travel at the cone surface, the continuous thin film starts breaking up forming small holes. This leads to detachment of large ligaments. The thin film gives an ideal surface for efficient disintegration into small droplets by high velocity gas jets. A schematic representation of this is shown in figure 1c.

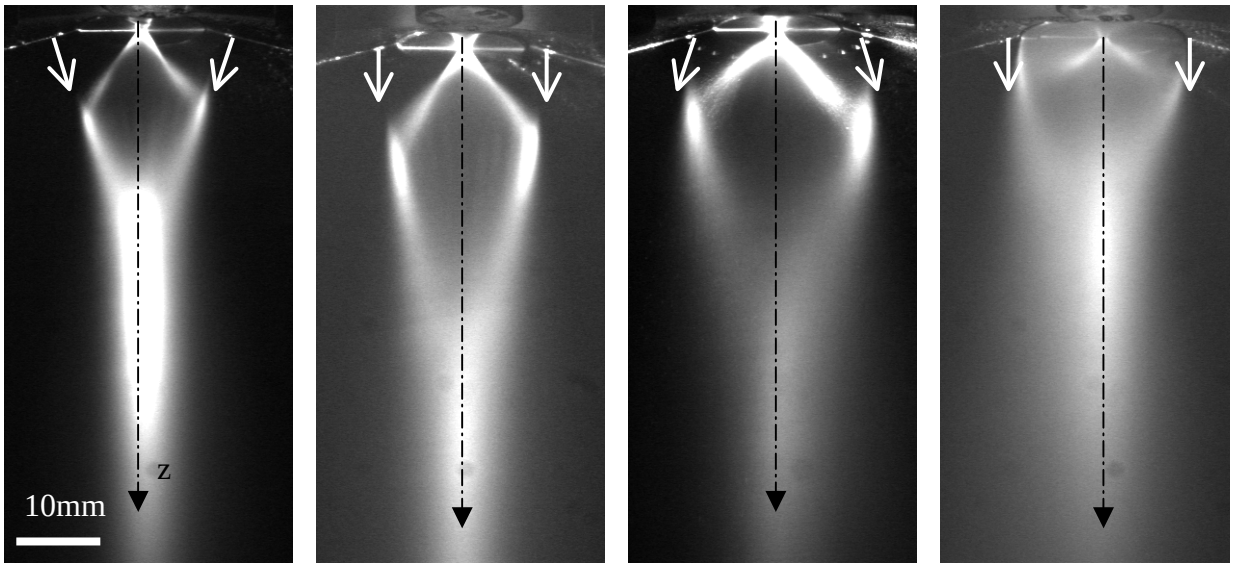
a) b) c)

Fig. 1: Schematic of CHA, cross section of the conical film (a), picture of CHA with tin alloy (b), schematic of new atomization technique (c)

The details of the atomization process are unknown and have not been investigated. To develop this process, initial experiments were carried out with water instead of molten metal and air instead of nitrogen. The goal of these experiments was to study the interaction between gas flow and liquid film. During this investigation, several parameters have been varied (water flow rate, gas flow rate or gas pressure in the gas nozzle and design of the gas nozzle). Figure 2 shows a few examples of the laser light sheet pictures. The bright zones mark the water film and the atomized particles. The water film leaves the nozzle at the top of the pictures and builds a conical film which is atomized by the gas stream (white arrows are used to mark the exit of the gas stream and the initial flow direction). Generally, discrete jet nozzles (axial array of cylindrical holes) were used at different axial impingement angles (-15, 0, +15°). It can be seen how different gas flow directions affect the cone angle of the conical water film. In addition, a ring slit nozzle was used. The laser light sheet picture shows

that the water film is even more influenced (Figure 1d). It was observed that many droplets hit the gas nozzle, which can cause serious problems in the case of molten metal.

Measurements of the static pressure (without water flow) give a better understanding of the gas flow. In figure 3, the static pressure relative to atmosphere (p_a) is plotted over the axial distance z from the atomizer. The pressure differences are very strong for the ring slit nozzle, which indicates a strong effect on the water film. There is a subpressure of about –10 kPa in the vicinity of the melt nozzle (z = 0 mm) and an overpressure of about 25 kPa at the stagnation point of the gas flow.



a) –15°, discrete b) 0, discrete c) +15°, discrete d) 0°, ring slit

Fig. 2: Laser light sheet pictures of water atomization with the new atomization technique, different gas nozzle designs, constant gas flow 45 kg/h, constant water flow app. 50 kg/h.

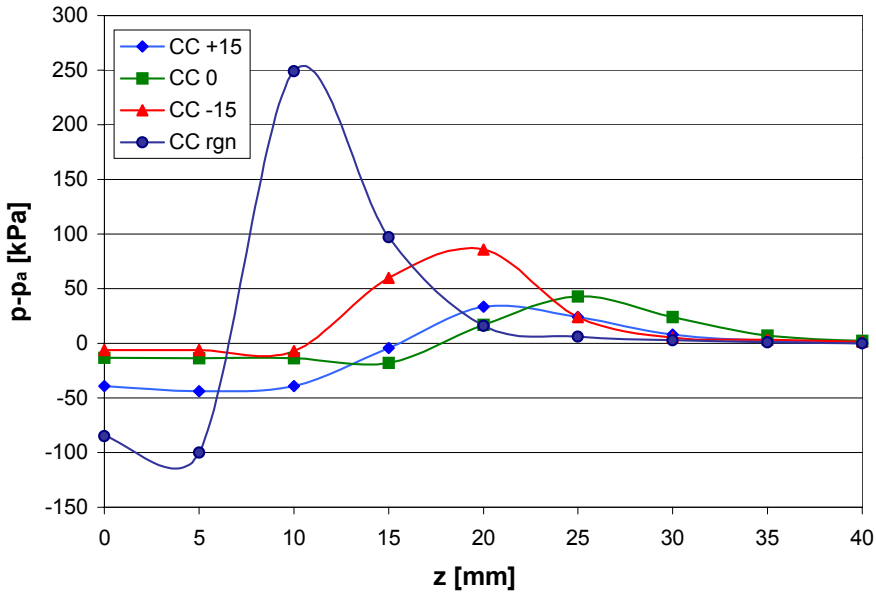


Fig. 3: Static pressure minus ambient pressure on the center line z of the atomizer, different gas nozzle design (compare to fig. 2)

3. Experimental Details

Table 1 summarizes the experimental parameters of the molten metal experiments with pure tin. Different alloys (Sn61Pb39; Sn62Pb36Ag2; CuSn15P5Ni4) were successfully atomized with this technique, but will not be analyzed in this paper. A static overpressure of 0.7 MPa on top of the melt forced the melt through the pressure nozzle and led to a melt flow of rate of 185 ±15 kg/h. Due to the investigations with water, the gas nozzle with an angle of 0° was chosen for all experiments. The atomization gas supply pressure, which directly varies with the gas flow, was the only parameter varied. The gas flow varied in the range from 21 to 98 m³/h. The spray chamber was evacuated (2 kPa) and refilled with nitrogen and, due to this process, the oxygen content was between 10 and 50 ppm before atomization started. Nitrogen was used as an atomizing medium. After atomization, the powder was separated in several samples and analyzed with different methods (dry sieving, laser diffraction with wet dispersion = BSM).

Table 1: Experimental Parameters and Results

no.	run time	pressure on top of the melt	mass flow melt	gas pressure	volume flow gas	gas to metal ratio	mass median (Sieving)	mass median (BSM)
	t	p_L	$\dot{M}_L$	p_g-p_a	$\dot{V}_G$	GMR	$d_{50,3}$	$d_{50,3}$
	[s]	[MPa]	[kg/h]	[MPa]	[m³/h]	[m³/kg]	[µm]	[µm]
5	181	0.7	199	0.2	27	0.14	150	-
6	187	0.7	193	0.4	45	0.23	98	85
3	188	0.7	191	0.6	62	0.33	61	47
8	209	0.7	172	0.6	62	0.36	67	49
7	192	0.7	188	0.8	80	0.43	54	39
2	206	0.7	175	1.0	97	0.56	-	23

4. Results

Table 1 shows strong dependence of median particle diameter on the gas flow rate. Figure 4 shows the particle size distribution (Cumulative mass Q_3) at different gas pressures. The powder was wet dispersed and the measurements were carried out with a laser light diffraction instrument (Coulter LS Particle Size Analyzer LS 130). Essentially, the particles are spherical, which is shown in the SEM pictures for two of the raw powders.

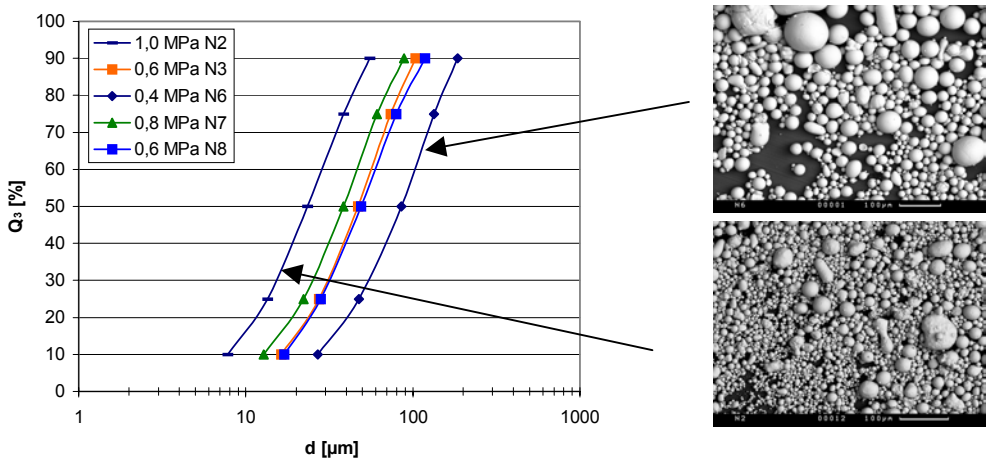


Fig. 4: Cumulative mass distribution Q_3 for different gas pressures (p_g – p_a varied between 0.4 and 1.0 MPa), SEM of the finest and coarsest raw powder, measurement by laser light diffraction (wet dispersion)

However, there was a big difference between the mass median diameter measured by the laser light diffraction and the sieving analysis of the dry powder. Figure 5 compares both measurements and shows that the mass median diameter obtained from the sieving analysis is approximately 15 µm larger in size. Further measurements are necessary to find the reason for this difference in the mass median diameter. Nevertheless, a correlation between the mass median and the gas to metal ratio (GMR) can be found, using the following function:

$$d_{50,3} = C_1 \, GMR^{-C_2} \tag{1}$$

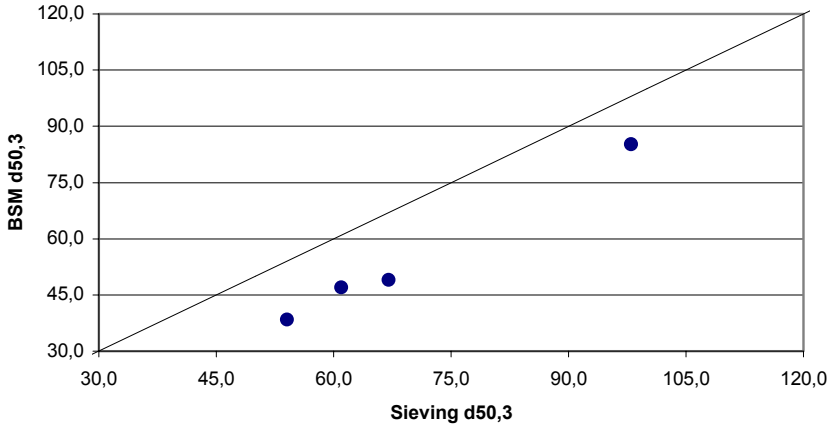


Fig.. 5: Comparison of different size measuring techniques, laser light diffraction (BSM) versus sieving

Figure 6 shows variation of mass median diameter with gas to metal ratio for both measurements (Laser Light Diffraction and sieving). Two correlation functions are shown as well, using the same exponent C_2 = 1.0. This indicates that the exponent does not depend on the measuring method. Further experiments with increased gas flows will show how much the particle size can be reduced.

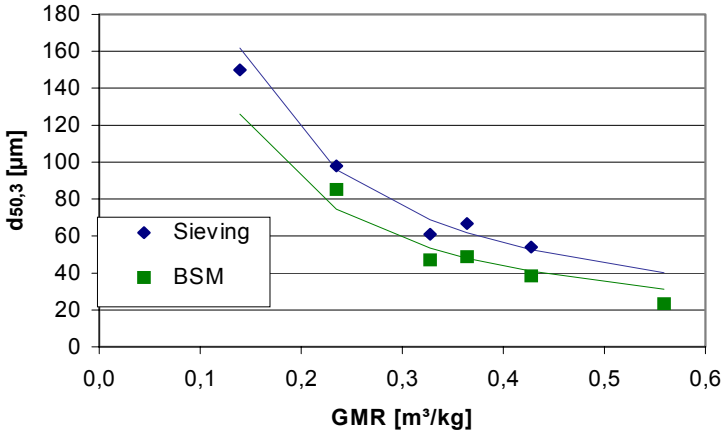


Fig. 6: Mass median diameter versus GMR, different measuring techniques (Sieving, BSM = Laser Light Diffraction)

Even more important than the mass median diameter is the standard deviation of the particle size distribution. In this investigation, the diameter ratio between d_{84} and d_{50} is used to characterize the width of the particle size distribution. Again the result of both measuring

techniques are presented versus the gas to metal ratio (figure 7). A correlation between the diameter ratio and the gas to metal ratio can be neglected for the sieving and diffraction measurement as well. But the mean values are quite different. While the diameter ratio for the laser diffraction measurement (BSM) is app. 2.0, the sieving analysis gives a value of app. 1.6. A narrow size distribution is interesting because this also means high productivity. Due to reasonable differences in the results (sieving and laser diffraction) a comparison with results achieved by other atomization methods (from the literature) is not useful. A comparison will only be useful if the powders were analyzed by the same technique.

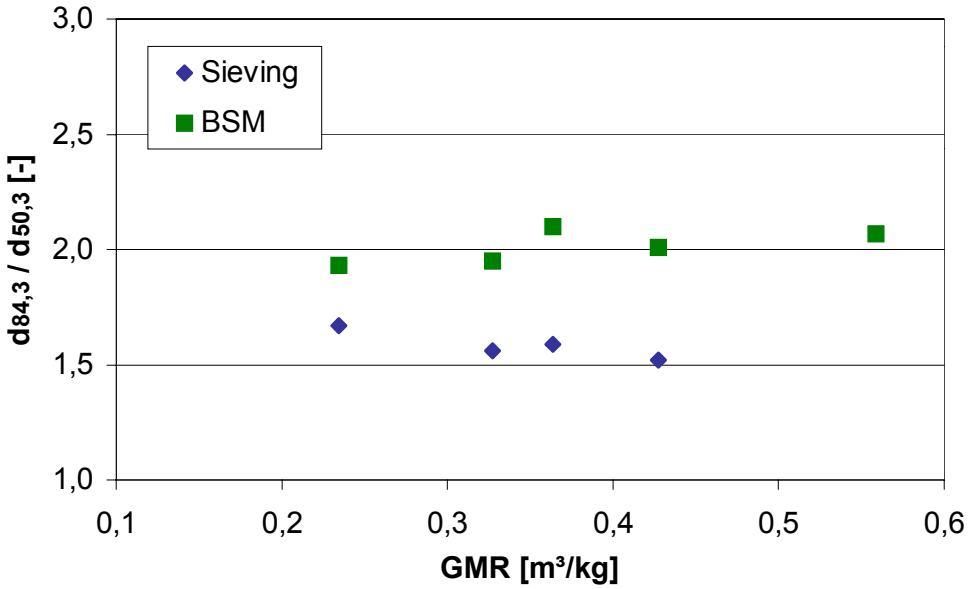


Fig. 7: Diameter ratio d_{84}/d_{50} versus gas to metal ratio GMR, sieving analysis and laser light diffraction method (BSM)

4.1 Effect of mass median diameter and geometric standard deviation on the productivity

The relation between productivity, mass median diameter and geometric standard deviation can be calculated. For this calculation a log-normal particle size distribution is assumed, which means that d84/d50 = d50/d16.

The calculation has been done for a size range of 25 to 45 µm, which is typical for „type 3“ solder powders. Figure 8 shows the yield versus the mass median diameter with different geometric standard deviations (σ). Each curve shows an optimum when the median diameter is app. 35 µm. This optimum is not very distinct for high standard deviations, however, it becomes more important when the standard deviation decreases. Low standard deviations are very important for the yield. Standard deviations of 2.25 are typical for gas atomization. The yield will increase if it is possible to achieve a standard deviation below 1.75.

Exactly similar curves are obtained for the relative productivity. Starting with a standard deviation of 2.25 the productivity will increase app. 40% or 80% if the standard deviation can be reduced to 1.75 or 1.5. Again this example shows how important it is to find atomization techniques with high production rates and narrow particle size distributions.

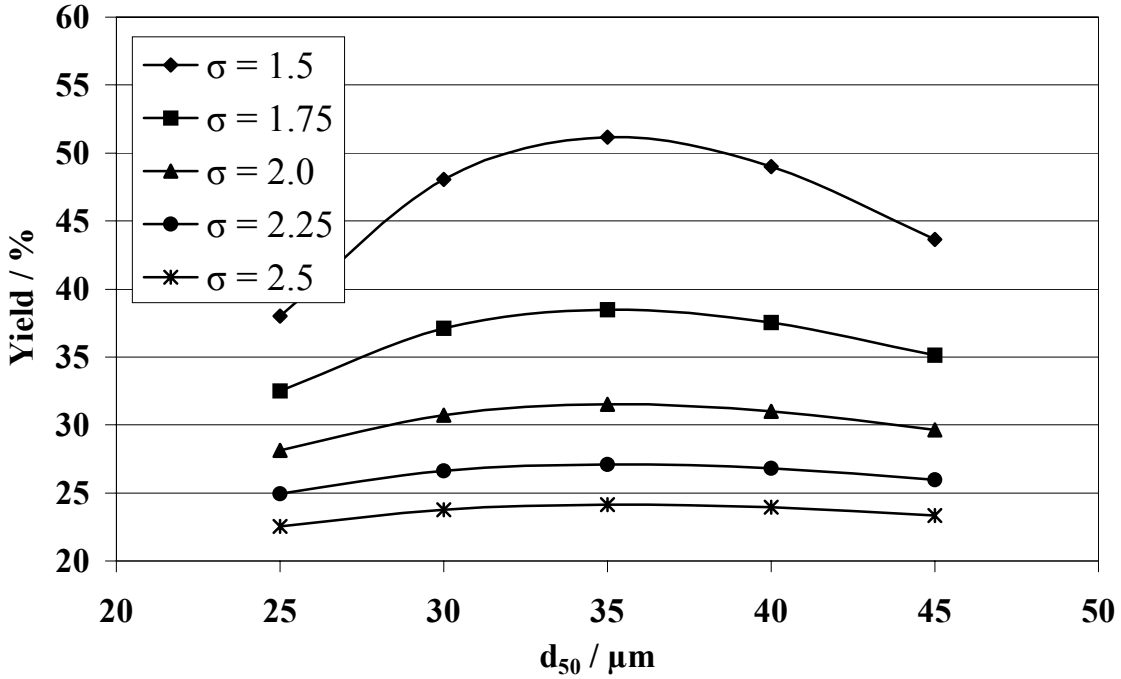


Fig. 8: Yield of the size range 25-45 µm versus mass median diameter, different geometric standard deviations (σ)

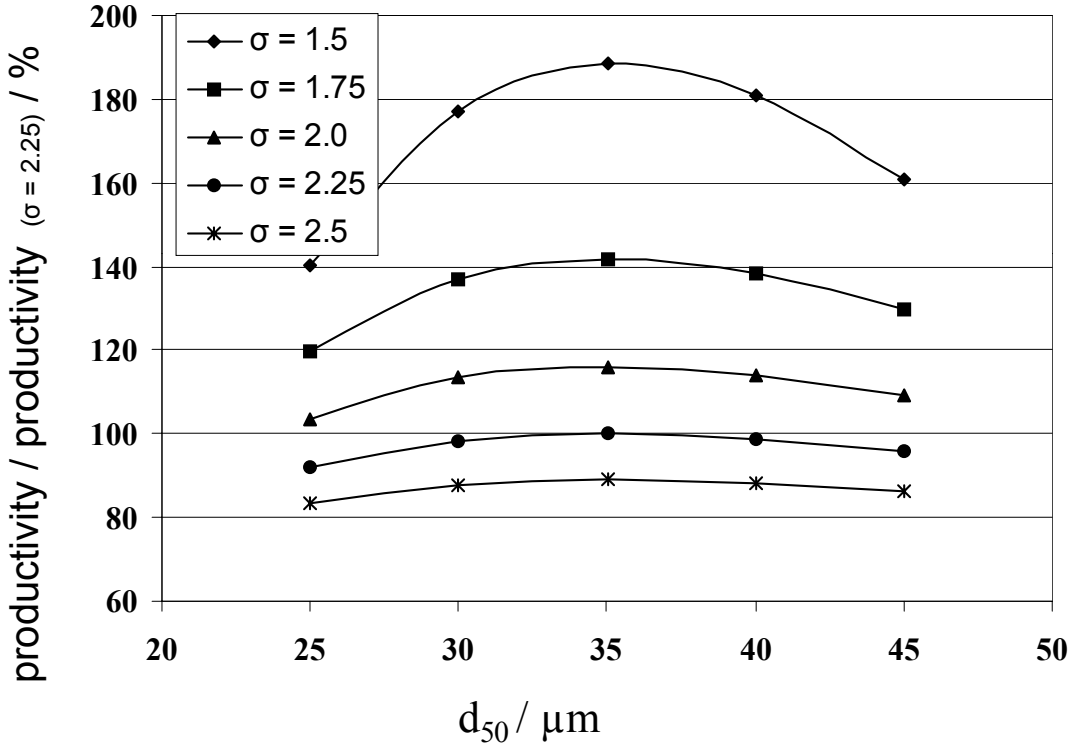


Fig. 9: Rel. productivity versus mass median diameter, different geometric standard deviations (σ)

5. Summary

A new atomization technique combining a pressure nozzle (centrifugal hydraulic atomization) and a gas nozzle was developed. This technique was refined by observations of flow patterns obtained from laser light illumination and measurement of the static pressure in the vicinity of the melt exit. For the first time, molten metals (pure tin and some alloys) were atomized successfully with this new method using different gas flow rates. The tin powder was analyzed by sieving and laser light diffraction. Both measuring techniques deliver different results for the mass median diameter (d_{50}) and the characteristic size distribution (d_{84}/d_{50}), as well. The decrease in median particle size with increasing gas flow can be described by an exponential law. For comparison with other atomization techniques it is necessary to use the same methods to analyze the metal powder. The sieving analysis shows larger median particle diameter (app. 15 µm larger than laser light diffraction) and much better results in diameter ratio (d_{84}/d_{50}) of about 1.6. A narrow particle size distribution is the key parameter for high yield and productivity. These first results on the new atomization technique are encouraging but several developmental steps are necessary for the future.

6. References

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