

# Powder generation from melts with high viscosity

**C. Czisch, H. Lohner, U. Fritsching and K. Bauckhage**

University of Bremen, Dept. of Chemical Engineering  
Badgasteiner Str. 3, D-28359 Bremen, Germany  
email: czisch@uni-bremen.de

Atomizing melts with high viscosity and a comparably low surface tension generally result not in spherical particles but in a great amount of fibres. The solution of this problem to be described here to prevent fibre formation and to generate spherical powder particles is to increase the particle spheroidization time during atomization by increasing the gas temperature in the process. A pilot scale atomization plant with a spray tower equipped with a twin fluid atomizer has been designed and set to work which uses hot gas or hot steam to atomize this molten materials. With this device it is possible to adjust an atomizing gas temperature from 300K to 1273K combined with a gas pressure up to 6 bar and gas and melt mass flow rates up to 500 kg/h respectively.

Experimental results show that for constant atomization pressures the powder to fibre ratio increases with increasing gas temperature (thus at decreasing gas mass flow rates or gas to melt mass flow rate GMR). Also, for constant gas temperature the mass median diameter of the powder particles decreases with increasing gas pressure.

Atomization experiments using different gas temperatures, gas pressures and melt temperatures have been performed. The results demonstrate, that an almost fibre free product with powder mass fractions up to 97% can be obtained by hot gas atomization of viscous melts.

In addition, numerical simulations for different atomizer nozzle designs and several operating conditions have been realized to assist the physical interpretation of the experimental results.

## 1. Introduction

Typical mineral melts have a high viscosity and a low surface tension compared to other melts, such as metal melts. When conventionally atomized under cold conditions, these material characteristics lead to a fibre product or at least to a product with a high fibre content instead of a particle product.

In general in gas atomization the atomization process starts, when the atomization gas comes in contact with the liquid stream to be atomized. When using a twin fluid atomizer with external mixing, the atomization gas for disintegration of the liquid stream typically is supplied by discrete nozzles which are arranged concentric to the melt stream. In a free fall atomizer normally each nozzle is directed with an angle towards the melt stream so that the atomization gas comes in contact with the liquid stream in an atomization spot, which is

located a few centimeters below the atomizer. A sketch of a typical twin fluid atomizer with external mixing is shown in fig. 1. The atomization process itself can be separated in two sequential fragmentation steps. The first step starts with small surface perturbations leading to instabilities which grow due to the shear stress caused by the high velocity difference between the atomization gas and the liquid stream. On the surface these instabilities appear as growing waves. The waves will be amplified until a critical level is reached, and the melt stream disintegrates into ligaments. These ligaments may be further disintegrated due to surface tension.

In the second step of fragmentation the generated ligaments are deformed. Like in the first step of fragmentation the deformation depend on the velocity difference between the melt and the atomization gas. When the elongated ligaments reach a critical length to diameter ratio the ligament disintegrates into smaller parts and may form spherical particles due to surface tension.

The problem in atomizing mineral melts is that the viscosity drastically increases with decreasing temperature within the atomization process. Thus it is possible to disintegrate the melt stream into ligaments, because of the high temperature and the low viscosity at the beginning of the atomization process, but as the temperature of the ligaments decrease and the viscosity increases rapidly so that the ligaments can not be disintegrated further into particles in the second atomization step. Therefore the ligaments remain in a fibre state.

To prevent the termination of the atomization after the first fragmentation step and to disintegrate also the ligaments into powder, it is necessary to increase the atomizing gas temperature. The lower temperature difference between the hot gas and the melt results in a lower heat transfer. Thus the melt stays longer in a liquid state with low viscosity and the atomization process can finish in globular particles. A few approaches cited in literature are known using hot gas to atomize melts [1], [2], [3], [5].

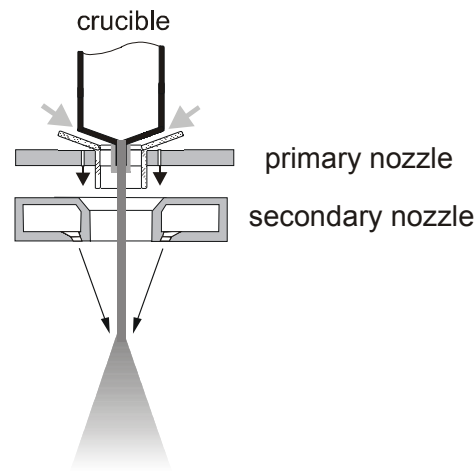
To investigate the influence of increasing gas temperatures on the atomization process a pilot plant for hot gas atomization of viscous melts is used. Different melt types have been atomized by hot gas as well as hot steam using a twin fluid atomizer. The aim is to produce a powder containing a high amount of spherical particles.

## **2. Pilot plant**

Main part of the pilot plant is a spray tower app. 5.5m in height. On top of the spray tower the material is molten by an induction heating device. The melt flows out of the crucible due to gravity (melt-mass flow rates up to 500kg/h) and is atomized in a free fall-twin fluid atomizer by heated gases (air, nitrogen or watersteam). The setup of the pilot plant is shown in fig. 2 and the used gas atomizer is shown and fig. 1.

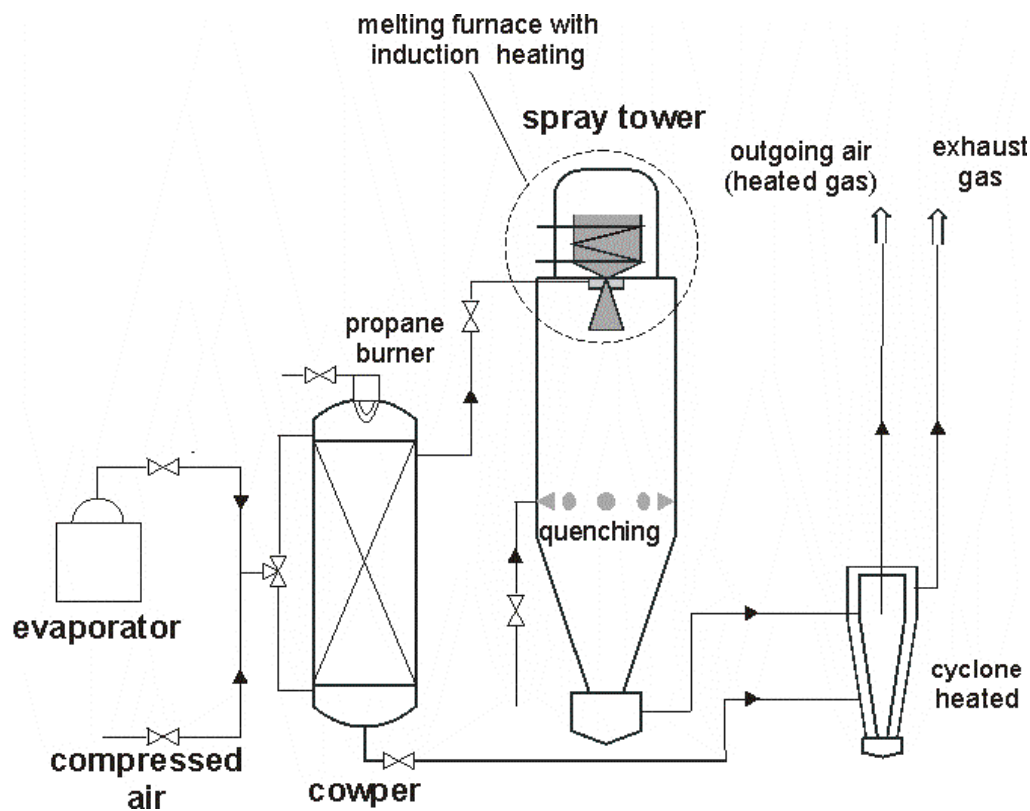
The melt jet is initially covered by a primary gas flow to suppress recirculation of melt droplets in the nozzle vicinity and prevent clogging of the crucible or the gas nozzles. Atomization of the melt jet is realized by the secondary gas flow. Gas temperatures up to 1273K and gas pressures up to 0.6MPa can be realized. The resulting melt droplets in the spray may be quenched and solidified about 2m below the atomization nozzle. The resulting

powder is collected at the bottom of the spray tower, the fine powder is deposited in a heated cyclone.



**Fig. 1** Atomization nozzle.

The hot gas for atomization is produced by a discontinuous heat exchanger (cowper). A ceramic bulk material inside the cowper is heated by a propane burner up to app. 1670K. After this heating process, compressed gas or steam is blown into the cowper. The preset value of the atomization gas temperature is obtained by mixing the heated gas with gas at room temperature at the top of the cowper before entering the atomizer.



**Fig. 2** Experimental setup of the pilot plant.

### 3. Experimental Results

Atomization experiments for different types of melts and atomizing gases, as well as different gas temperatures, gas pressures and melt temperatures have been performed [4], results will be discussed based on main physical influences.

#### Temperature influence on the particle to fibre ratio and on the mass median particle size

Under cold atomization conditions (20 °C atomization gas temperature) the atomization of high viscous melts tend to produce fibres. With increasing temperature it is assumed to increasingly produce particles. Thus the particle to fibre ratio PFR is an important aspect within the investigation of the temperature influence on the atomization process. Experimental results for atomization of a ceramic melt by heated gas (air) or steam are shown for the PFR in fig. 3, thereby the focus is on the particle fraction. It can be seen that the fraction of particles increases with increasing gas temperature.

Experimental results considering the temperature influence on the mass median particle size of the powder fraction are shown in fig.4. The mass median particle size decreases with increasing temperature. Strauss and Dunkley [7] estimated that the temperature influence on the decreasing median particle size when the pressure is constant is given by an exponential factor of  $-0.25$ . Our experiments are best correlated by an exponential factor of  $-0.39$ . The experiments were done with steam as well as with hot gas at constant gas pressures ( $p_1 = 0.3\text{MPa}$  and  $p_2 = 0.45\text{MPa}$ ). In fig.5 an optical microscope image of a sample powder fraction with a diameter  $d < 112\mu\text{m}$  is shown. It can be seen that most of the powder is spherical.

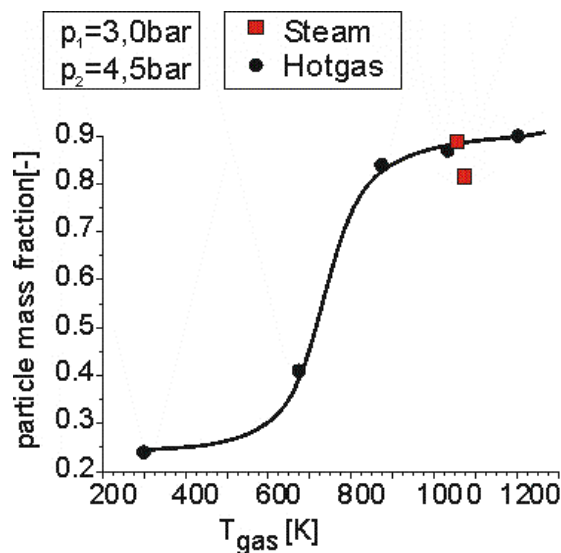


Fig. 3 Influence of temperature on the particle mass fraction.

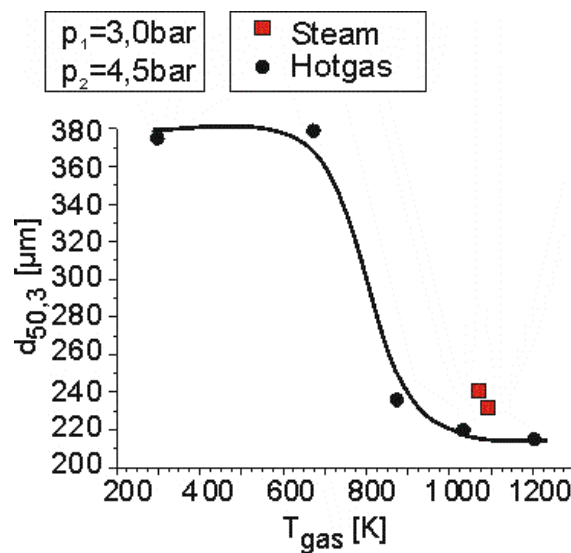
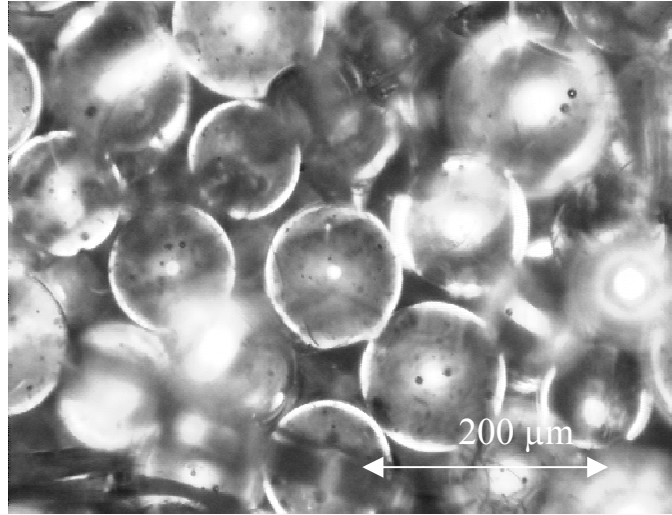


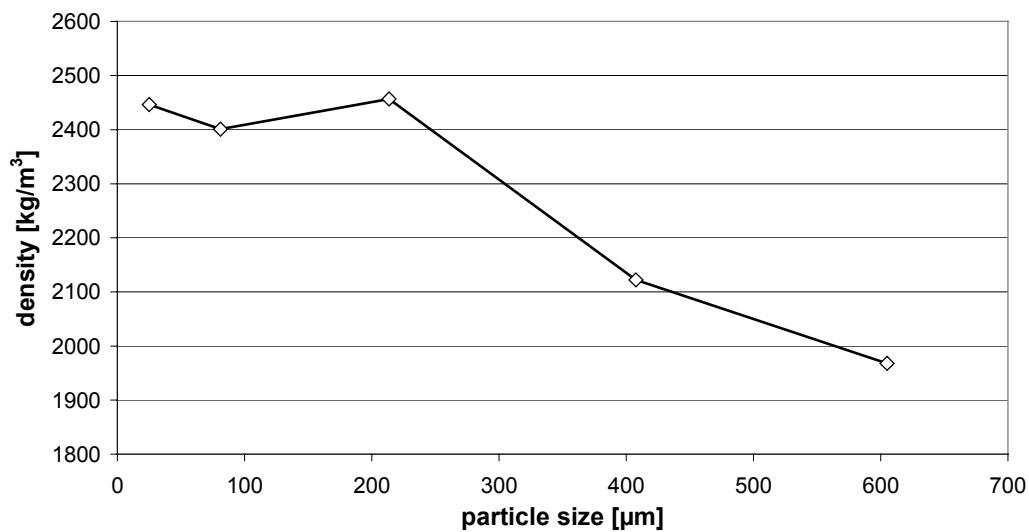
Fig. 4 Influence of temperature on the mass median particle size



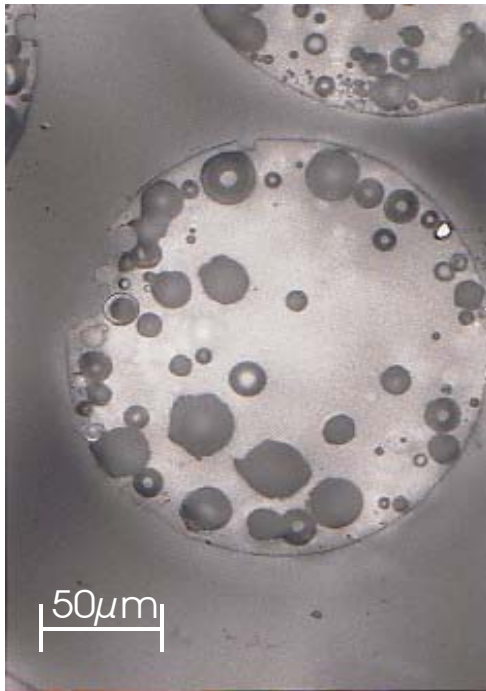
**Fig. 5** Optical microscope image of hot gas atomized powder particles.

#### 4. Gas/melt interactions

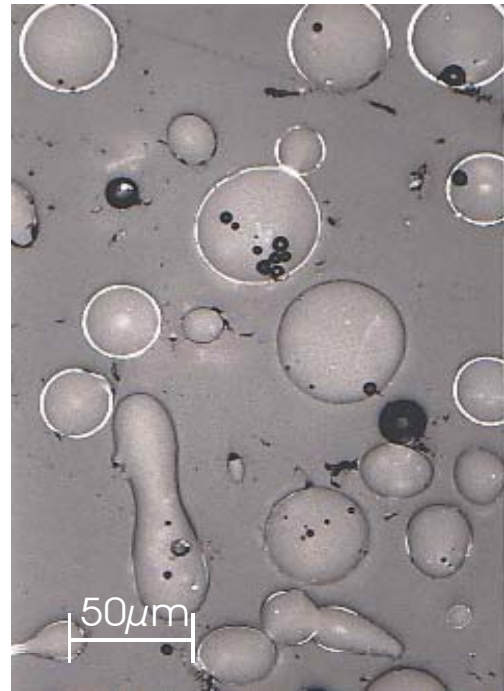
Besides mechanical interactions of the atomizing gas and the liquid melt also a species interaction is observed within the atomization investigations. Ceramic powder generated with hot steam show a certain amount of porosity increasing with particle size. Fig.6 shows the density characteristics of the atomized particles depending on particle size. The density decreases by app. 20% for the larger particles. Fig.7 and fig.8 present some images of hot steam generated particles taken by a optical microscope. Here the powder fraction has been embedded into plastics and micro grinded for preparation. The pore distribution shows that most pores are to be found immediately beneath the surface of the particle. Therefore it is assumed that a solution / diffusion process causes the pore formation. The pores predominantly are found in the larger particles.



**Fig. 6** Density of particles generated by hot steam (minimal density = 1970 kg/m<sup>3</sup>; original material density = 2600 kg/m<sup>3</sup>)



**Fig. 7** Fraction 315-500 $\mu\text{m}$



**Fig. 8** Fraction 112-315 $\mu\text{m}$

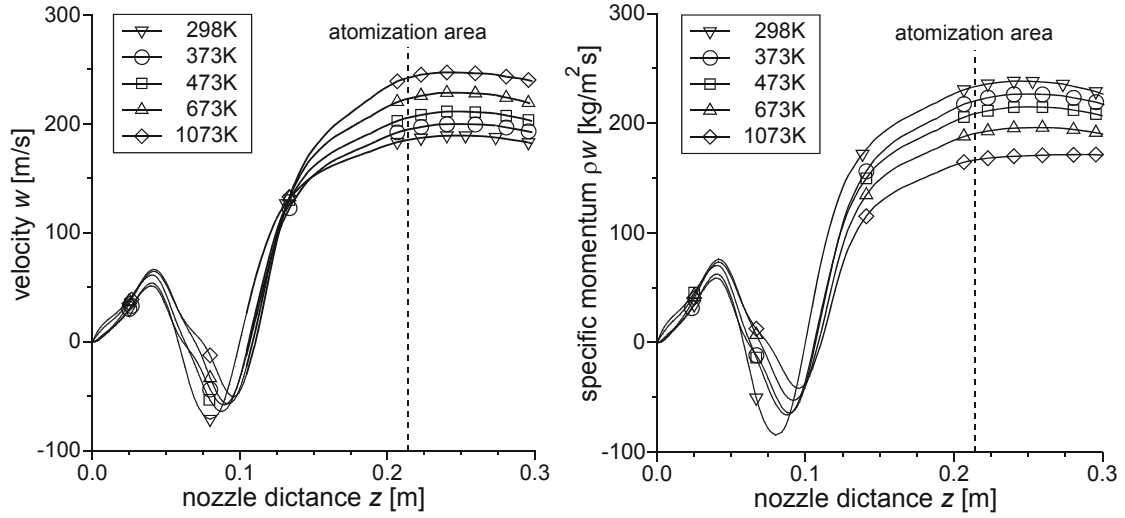
## 5. Maximum viscosity

Various types of melts has been hot gas atomized. Depending on experimental results for different melt types with different viscosities the maximum viscosity which can be successfully atomized within the actual pilot plant setup is limited between  $>1$  and  $<10$  Pa\*s, where the melt surface tension was app. 0.2 to 0.3 N/m.

## 6. Simulations

CFD-simulations support the proper description of the temperature and flow conditions during the atomization process. Numerical simulations for several operating conditions have been realized.

In fig.10 the local gas velocity  $w$  (left) and the simulated specific momentum  $\rho w$  (right) on the center line of the atomizer are shown. It can be seen that the gas velocity increases and the specific gas momentum decreases with increasing gas temperature. Both simulations have been performed at constant gas pressures (atomization pressure  $p_2 = 0.55\text{MPa}$ , primary pressure  $p_1 = 0.18\text{MPa}$ ).



**Fig. 10** Influence of gas temperature  $T_{\text{gas}}$  on gas velocity  $w$  (left) and specific gas momentum  $\rho w$  (right) on the center line at constant gas pressure.

To take into account that the experimental results show a decreasing mass median particle size with increasing atomization gas temperature, the slip velocity between the atomization gas and melt stream respectively a shear process can be assumed to be the main driving force of the atomization process and not the momentum exchange between gas flow and melt jet.

## 7. Summary and Outlook

The investigation point out that it is possible to atomize high viscous melts such as glass or ceramic melts and generate an almost fibre free product by hot gas atomization. The particles which were produced show a spherical shape and the mass median particle size depends on the adjusted process parameters. The mean diameter of the particles decreases for constant atomization pressure with increasing gas temperature and decreasing gas mass flow rate resp. decreasing specific gas momentum in the atomization area. Thus the main driving force of the atomization process is assumed to be the shear stress between gas and melt.

A maximum viscosity limit between 1 and 10 Pa\*s is observed for the basic pilot plant setup. The melt surface tension in this case was approximately 0.2 to 0.3 N/m. This limit result from the temperature gradient between the molten melt and the atomizing gas which is still too high, so that melts with a higher viscosity cannot be transformed into powder. To enhance this limit to higher values the atomizing gas temperature needs to be increased further.

For future investigations an additional gas heater will be attached to the basic plant setup to extend the viscosity limit up to higher values. A new atomizer design based on a prefilming of the viscous melt prior to gas atomization is developed to increase the atomization efficiency.

## 8. Acknowledgment

This investigation was partially supported by the DFG (Deutsche Forschungsgemeinschaft).

## 9. References

- [1] Dunkley, J.J., "The role of energy in gas atomization", PM2TEC 01, New Orleans, 13.-17.05.2001, pp. 2-29-2-35
- [2] Gerking, L., " Powder from metal and ceramic melts by laminar gas streams at supersonic speeds ", PMI 25, (1993), 2, pp. 59-65.
- [3] Lohner, H., Czisch, C, Fritsching, U., Bauckhage, K, "Granulation viskoser Mineralschmelzen mittels Heißgaszerstäubung", Chem. Ing. Tech. 74, (2002), 8/9
- [4] Lohner, H., Zerstäuben von Mineralschmelzen mit Heißgas, PhD-thesis University of Bremen, (2002)
- [5] Strauss, J.T., "Hotter gas increases atomization efficiency", MPR 11, (1999) 24-28
- [6] Watkinson, D., Hughes, R., Sims, G., Yule, A., "Gas atomization of polymeric materials", ILASS Europe, Zürich, 02.-06.09.2001
- [7] Strauss, J.T., Dunkley, J. J. "An Experimental and Empirical Study of Close-Coupled Gas Atomisation", Proc World PM Congress, Kyoto 2000