Inert Gas Metal Atomization Using a Laval Nozzle, Preheated Gas and Radial Melt Injection

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Abstract
A new strategy for the inert gas atomization of melts is presented. The location of the melt and the gas are inverted, i.e. the gas flows centrally through a Laval nozzle and the melt is inserted radially inward through an annular slit at the throat of the Laval nozzle. The gas has to be preheated to avoid freezing of the melt to a temperature that compensates the cooling due to the gas expansion to sonic conditions. Using tin as a metal of low melting point first results are shown. They show markedly smaller sizes of particles in the range of 10µm. However, another peak is present at much larger sizes that still has to be reduced. There are extremely fine particles (<1µm) in the distribution that show the potential of the technique.

Keywords
melt atomization, gas preheating, Laval nozzle, radial melt inflow.

Introduction
Vacuum Inert Gas Atomization (VIGA) is a method of producing metal particles by letting high-speed inert gas coming from an annular nozzle (or a circular array of nozzles) impinge onto the stream of molten metal issuing from a central nozzle. The typical diameter of the melt nozzle is in the order of a few millimetres. Depending on the distance between the exits of the gas and melt nozzles they are called either free fall or closed coupled atomizers. Within the free fall atomizers there have been explicit use of Laval nozzles for acceleration of the gas flow, [2]. In the case of closed coupled atomizers, the idea is that the melt flows along the tip of the nozzle to the annular nozzle due to the effect of a recirculating gas region at the tip of the nozzle. This is more or less pre-filming. The inert gas is accelerated within the annular nozzle to sonic conditions at the nozzle exit (convergent nozzle) and within the first Mach cell region if the jet is under-expanded. In all cases the result is that the gas (e.g. argon) has a temperature of about -50°C when it exits the annular gas nozzle or the array of nozzles. Due to the very large temperature difference between the atomizing gas and the melt, it would solidify on contact with the gas. This is avoided by superheating the melt, i.e. heating it to temperatures above the melting point. However, if one compares the specific heat of metal melts (iron) with that of argon they all are only about 30% higher. This means that the superheat has to be considerable if it is to avoid premature solidification of the melt, since basically, inert gas atomization is a twin fluid atomization applied to a melt. In some cases, the gas has been preheated, see [3,4] showing the beneficial effect of preheating. A mass flow ratio in the order one is typical for this type of atomizer. The fundamental mode of operation of these atomizers are the aerodynamic and shear forces of the gas acting on the liquid surface. These depend on the relative velocity between the phases. However, not only the aerodynamic forces depend on the relative velocity but also the heat transfer between the phases. Cold gas will not only atomize but will also effectively cool the melt. At the later stages of the atomization process, i.e. when secondary atomization is completed cooling is mandatory to solidify the drops otherwise, they can coalesce or impact and solidify on surfaces. Therefore, before primary and secondary atomization are completed, cooling hinders atomization. In the worst case a thin shell of solidified metal on the melt drops would stop atomization altogether and this although most of the drop is still molten. The work hypothesis of the present paper is that the atomizing gas has to be preheated to a temperature T_0 high enough that after expansion to let's say sonic conditions the gas has a temperature T_m at the nozzle exit at least as high of that of the melt:

$$T_0 > T_m \left(1 + \frac{k-1}{2}\right), \tag{1}$$

where k is the adiabatic exponent of the gas. Of course, if the expansion of the gas is extended to supersonic conditions ($Ma>1$) when it comes into contact with the melt an even higher initial temperature is needed.

$$T_0 > T_m(1 + \frac{k-1}{2} Ma^2) \tag{2}$$

A further problem of VIGA atomizers and of twin fluid atomizers too is the flow topology. All have in common that the melt or liquid flows in the central part and the gas flow surrounds it. This may be more adequate in the case of metallurgical temperatures, where only refractory materials can contain the melt. In the case of titanium, it would

react with the refractory material. However, if you take a look at the geometry of continuous casting of steel (Fig. 1) you find that in some cases argon is introduced centrally through the submerged entry nozzle at very low volume fluxes not for atomization but for reduction of clogging and to help reduce inclusions of alumina in the steel. Therefore, in principle it is possible to switch the location of the liquid or melt to be atomized to an external annular and thin layer on the wall of a Laval nozzle with sonic or supersonic central and axial gas flow. To the authors knowledge this has not been done before.

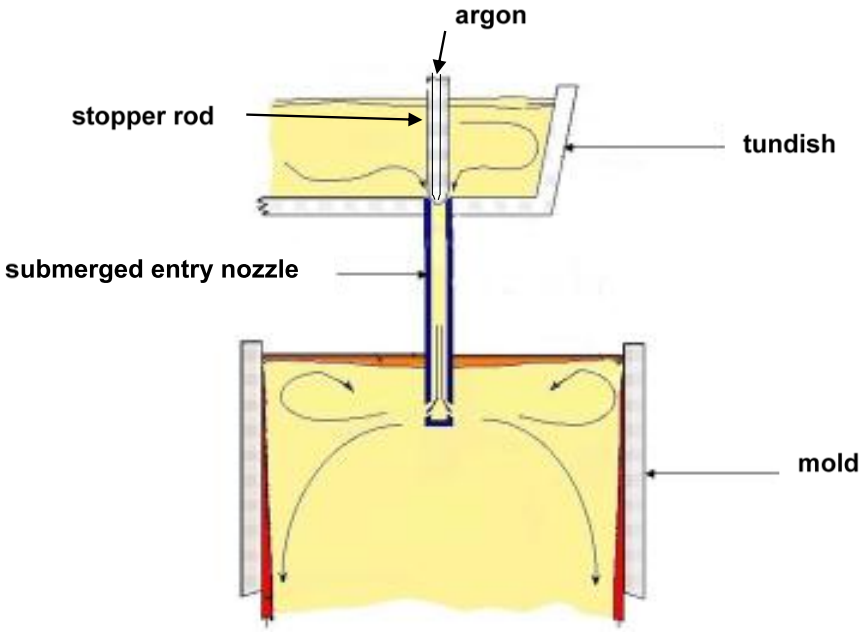

Figure 1. Basic arrangement for continuous casting of steel: argon is fed through the stopper rod.

Following the arrangement for continuous casting of steel a new atomizer geometry was developed. Figure 2 shows schematically the difference to existing atomizing strategies of a closed coupled atomizer, see also Fritsching and Uhlenwinkel [1]. In the new strategy the central gas flow is through a laval nozzle and the melt flow is through an annular gap between the stopper rod and the crucible at or close to the throat of the Laval nozzle. The convergent part of the nozzle is part of the stopper rod and the divergent part is built into the crucible at the seat of the stopper rod. This rod has the function of a valve to open the annular flow channel for the melt. In the case of continuous casting it is used to regulate the flow into the mold. Here it limits the mass flow rate of the melt and leaves only a thin annular slit open for the melt. The melt flows radially inward into the Laval nozzle. The stopper rod is shown in an open position. Due to the very high velocity of the gas, the melt is forced to follow the wall of the laval nozzle or it is immediately atomized. In the further course, shear forces on the melt will complete atomization. Of course, the prerequisite for this strategy to function is that the gas has to have at least the temperature of the melt. The present paper shows first results of the application of this new strategy to the atomization of tin. The advantage being of course, the low melting temperature that allows to use stainless steel for the crucible and the stopper rod.

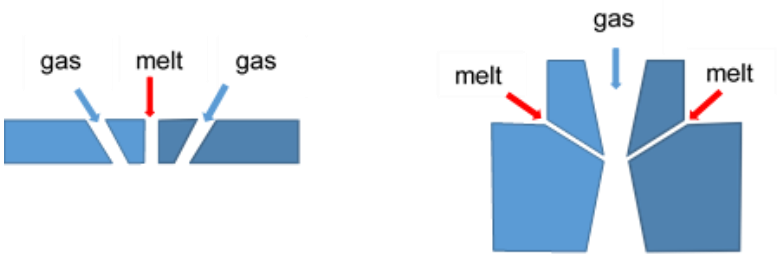

Figure 2. Basic difference between closed coupled atomizers on the left and the new strategy.

Material and methods

Figure 3 shows schematically the experimental set-up. For tin an electric heater is sufficient to heat the gas, about 450 °C. In the case of steel melt a different heater has to be used: a plasma arc can deliver the necessary temperatures, 3000 °C.

The temperatures of the melt, of the gas prior to entry into the stopper rod and at the exit of the Laval nozzle are monitored as well as the inlet gas pressure and the pressure in the crucible. The melt droplets and the gas form

the spray that expands into the plenum where the droplets can solidify. A further cold gas annular nozzle is mounted around the exit of the Laval nozzle for cooling, but it was not used. With careful control of the temperatures melt drop coalescence can be avoided. The solidified particles are separated from the gas in a cyclone and remaining fines below 1 µm are filtered before the gas exhausts. After a run, particles that are deposited on all inner walls of the set-up are collected and weighted. About 85 % of the mass placed initially in the crucible is found in a sieved fraction below 125µm. The rest are thin flakes. Apparently, the atomization of the thin layer of tin on the wall of the Laval nozzle is not complete when it exits the nozzle. This will need a longer nozzle in future experiments.

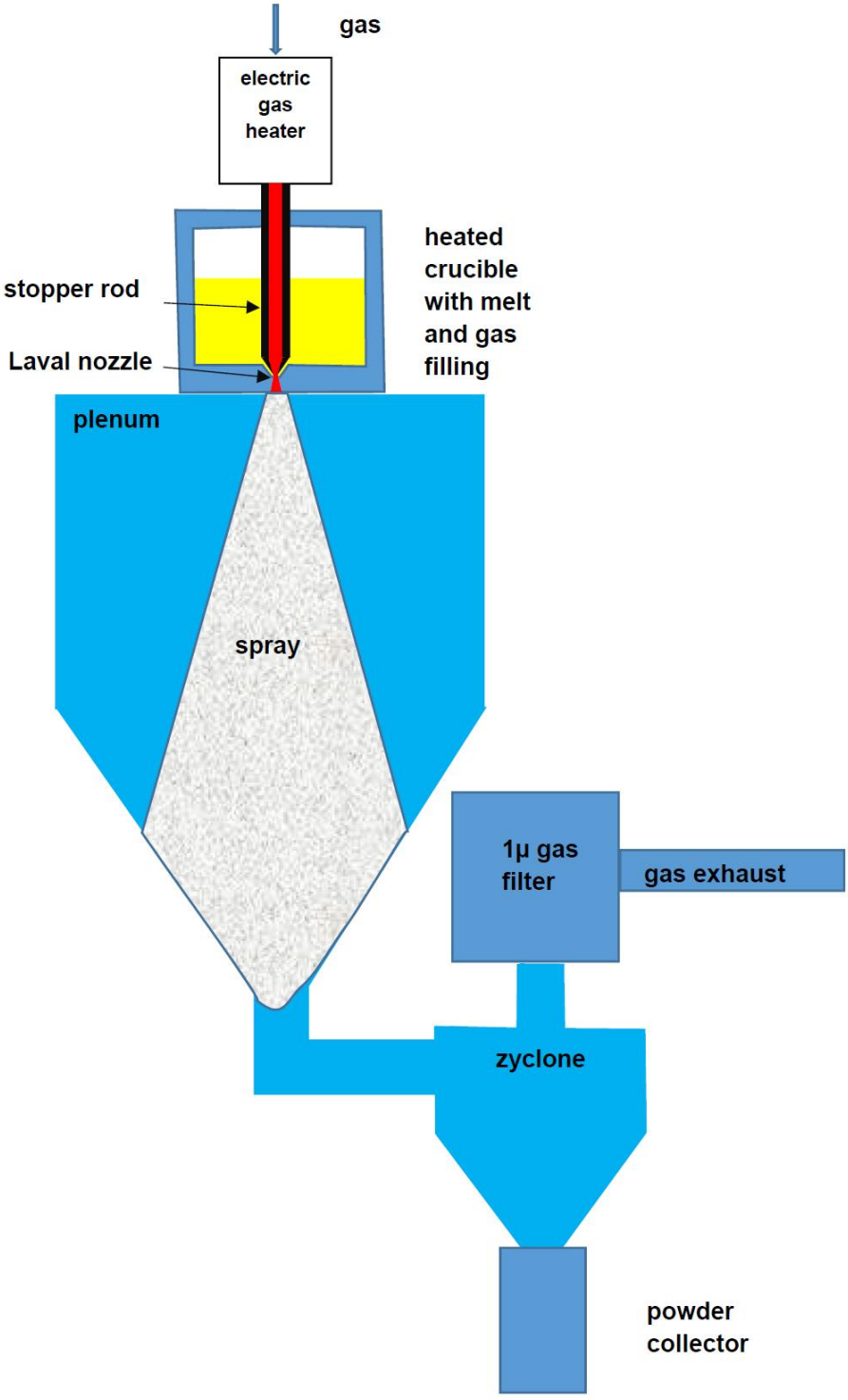

Figure 3. Schematic drawing of the experimental set-up.

Results and discussion

The Laval nozzle has a diameter of 3 mm at the throat and expands to 3.3 mm at its exit. This gives an exit Mach number of 1.4. The nozzle has to be cleaned after each run, since tin will not normally wet stainless steel but some deposits are still formed.

Figure 4 shows the particle size distribution obtained in the most successful run. The distribution shows that 80% of the particles are below 20 µm and the peak is at 10 µm. The peak of the distribution varies a little from run to run, the maximum value found for this lower peak was 15 µm. The shape is log normal. However, a second peak of low amplitude is present at about 100 µm. This is likely the result of sieving. There are obviously larger particles in the distribution, some of which can be seen on Figure 5, a REM image of the tin particles. An image with increased resolution, Figure 6 shows that there are very fine particles in the powder that are much smaller than 1 µm. This poses an unexpected problem since the exhaust filter was dimensioned for 1µm. Furthermore, there is some amount of agglomeration occurring during the flight of the particles. Obviously, there is still some work to be done to obtain consistenly a smaller particle sizes, spherical particles and less agglomerates but these first results show that the strategy can produce very fine particles, much finer than with the state of the art techniques.

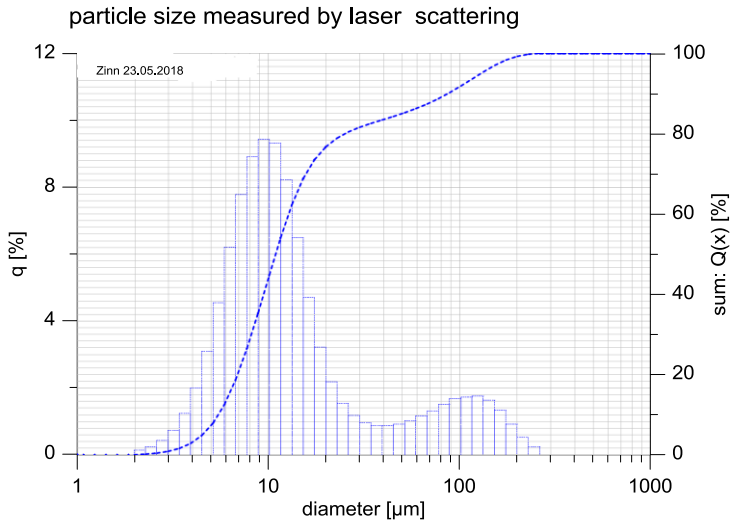

Figure 4. Particle size distribution measured by laser scattering.

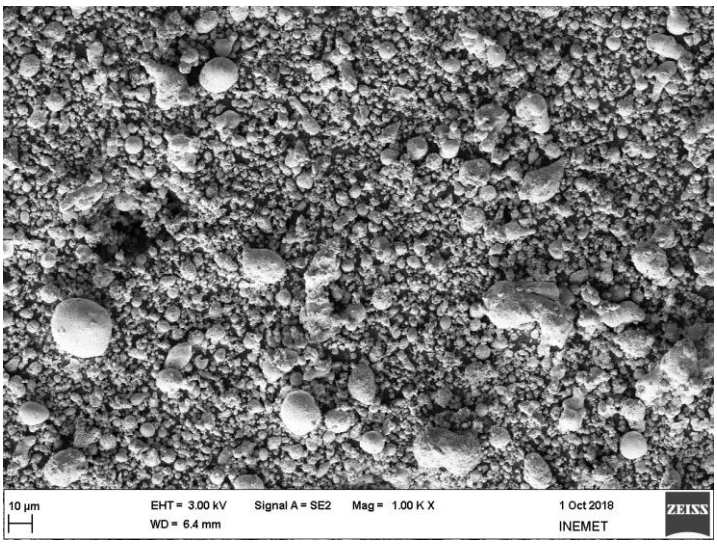

Figure 5. REM image of the tin powder.

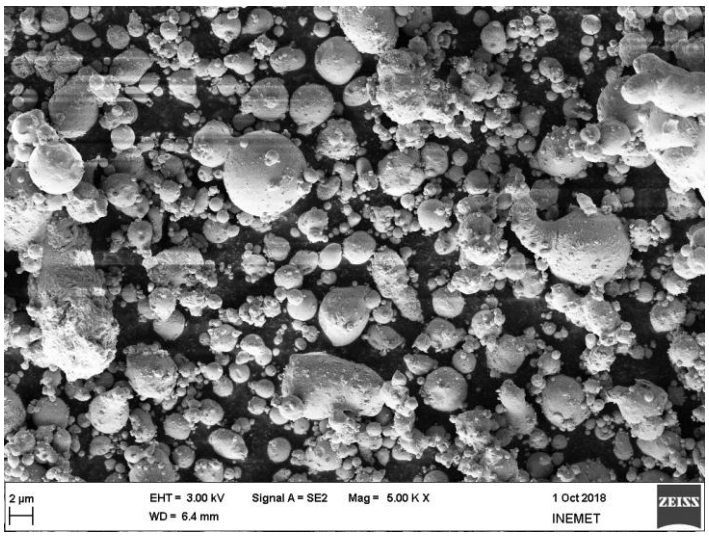

Figure 6. REM image of the tin powder, higher resolution.

Conclusions

The strategy of preheating the gas for inert gas atomization and inverting the position of atomizing gas and melt shows promising results. Even in the first experiments the particle sizes are significantly lower than that those found elsewhere. There appears to be two different physical mechanisms of melt drop production leading to a pronounced peak at very small sizes but also a peak at much larger sizes. The mass contained in this peak can be considerable. Therefore, future work will concentrate on improving the geometry of the set-up. The small size of the particles open up the possibility of reducing the roughness of parts produced by laser sintering methods or the mixture of the metal powder with ceramics for matrix composites that are not possible with large metal particles.

Nomenclature

T	Temperature [K]
k	adiabatic exponent [-]
m	of the melt
0	stagnation condition for the gas

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