# Intelligent Sensors for Atomization Processing of Molten Metals and Alloys

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## Abstract

This paper briefly introduces the principles of atomization processing of molten metals and alloys. It is shown that particle size and velocity are considered the two most important variables to monitor and control the atomization process. Potentially applicable instrumentation are discussed, and opportunities for developing intelligent sensors for monitoring and control of the process are proposed. Several on-line sensors suitable for atomization of molten metals and alloys are compared, including the laser diffraction particle analyzer (LDPA), particle counting, sizing and velocity measurement (PCSV) instruments, the phase/Doppler particle analyzer (P/DPA), the electric principle based monitor for particulates (EPMP), various imaging techniques for particle sizing (ITPS), and infrared image analysis (IIA) instruments. The isokinetic probe is introduced as a direct method to sample powders at the desired point in the atomized spray. Prospects and problems of integrating instruments based on these principles into *intelligent processing of materials (IPM)* scenarios are discussed.

## The Atomization Process

Atomization is the preferred method for producing rapidly solidified powders of metals and alloys. These powders have outstanding or specialized properties which cannot be obtained by conventional processing routes. The benefits of rapidly solidified powders of metals and alloys are a result of the higher cooling rates achieved in the process. Mainly these are decreased segregation, increased solid solubility, elimination of segregation phases, and a finer microstructure of powder and of consolidated product. These specific microstructures mean good chemical homogeneity, high corrosion resistance and advanced mechanical properties, particularly hot and cold workabilities[1,2,3] and improved creep properties. Rapidly solidified powders of metals and alloys have found wide application in the production of engine discs in advanced jet aircraft, gas turbine, near net shape manufacturing and other fields.

Unfortunately the atomization process is difficult to control: metallurgical properties are not always predictable, and finished product rejection rates are high. These problems arise due to the difficulty in controlling the process which results in variable grain size, broad particle size distributions, and in higher operating costs (eg. higher atomizing gas consumption). Tedious and costly powder classification is always carried out subsequent to the atomization process to ensure that the powder used in downstream processing has the desired characteristics. Thus, there is indeed a new challenge for the development and implementation of intelligent sensors to measure and monitor rapid solidification, and eventually to be integrated into intelligent processing of materials scenarios.

The atomizing process parameters can be divided into three types; control, dependent and product variables. Examples of control parameters are the temperature of molten metal, the pressure of the atomizing medium, the head of molten metal in the tundish, and the flow rates of the molten metal and the atomizing medium. Dependent parameters include the velocity of the molten metal droplets and of the atomizing medium, size and size distribution of droplets and cooling rate. Finally some of the product parameters are size and size distribution, microstructure, chemistry and shape of metal powders. Present practice is to measure and monitor some of the control parameters on-line by using conventional sensors. This paper will not deal with these instrumentation needs as they are readily available and are widely in use in the metals industry. The powder is then characterized off line upon completion of the atomization process. The control system which is mostly manual regulates the atomizing process at the desired set points of the control parameters. This approach has not produced commercial powders of the desired characteristics and of sufficiently high yield. Thus, the atomization of molten metals is an expensive process which remains more of an art than a science. Furthermore, the development of fine powders for novel applications has been an illusive commercial goal due to the lack of understanding and control of the process. Therefore, the challenge is to develop intelligent sensors for the accurate, reproducible, reliable and consistent measurement and prediction of powder characteristics (i.e. powder size, distribution, shape and microstructure). These sensors would be useful in research for generating a greater understanding of the atomization process and in production for on-line process control. The issue of powder quality (i.e foreign particles) while important is nevertheless outside the scope of the present discussion.

The ideal approach for atomization processing is to develop intelligent sensor(s) for in-situ monitoring, measurement and interpretation of powder size, shape and microstructure. However, sensors of the solidification microstructure have only been evaluated recently in continuous casting[4]. Ultrasonic reflection or attenuation in slabs have been investigated to locate the solid/liquid interface and predict the

microstructure in these castings. Clearly, this sensing approach is not relevant to atomization processing. On the other hand, other sensors do exist which have the potential for in-situ measurement of powder size and shape in small regions of the droplet spray cone. Control of these characteristics is critical to the improvement of powder yields[2] and powder microstructure Powder size distribution and shape is critical for press and sinter operations and powder shape is an important feature for good powder flowability applications. Thus, in the near future, powder characteristics must be determined indirectly or inferred from measurement of control and dependent parameters in regions of the spray and from a fundamental and heuristic understanding of the process - powder characteristics - property relationship for the molten metal atomization process. This approach has been referred to in general as Intelligent Sensors, Intelligent Processing of Materials or Integrated Processing of Materials.

To gain insight into the role and importance of sensors to atomization processing, a brief review follows of atomization processing of molten metals. The atomization process can be divided into twin fluid atomization, centrifugal atomization, and mechanical atomization.[3] The centrifugal atomization can be subdivided into several variants: rotating electrode, rapid rotating disc, and rotating perforated cup processes. All of them involve the supply of molten metal to a rotating part by gravity feed from a crucible or tundish or by melting the end of a rotating metal bar. Disintegration of liquid metal occurs at the edge of a rotating disc or bar and the liquid metal drops are thrown off by the centrifugal force. The droplets of molten metal spheroidize and solidify in flight or can be gas-quenched as they leave the rotating carrier. There is a wide variety of techniques to get small droplets by mechanical means: twin roll atomization, vibrating electrode atomization and Duwer gun technique.

Figure ATOMIZATION shows schematically the twin fluid atomization process. The metal stream falls freely down through the die, and is broken by atomizing fluids which is usually helium, argon or water. Typical operating parameters are given in Table ATOMI.

In atomization processing the sequence of events which culminate in the formation of powder with given characteristics are neihter well understood nor fully defined at present. However, the following phenomena play an important role in the atomization of molten metals and alloys: Atomization of liquid metal involves the break-up of the liquid stream into ligands and droplets, spheroidization of the droplets, nucleation of solid within the droplets, growth of the solid nuclei and removal of the heat of fusion from the powder to avoid recalescence. Ideally, these steps should occur in sequence while producing very fine powder of desired shape and narrow size distribution. In practice, however, this does not occur. In fact, there is a great deal of overlap in the occurrence of these phenomena. Research conducted to define and model these phenomena will be briefly reviewed in the following section.

## Process Fundamentals

**Atomization and Spherodization:** The mechanism of the molten stream break-up is common to all molten metal atomization schemes and thus will be presented *vis a vis* the gas atomization process. See *et al*[6,7] and Seaton et al[8] observing high speed movies of atomization considered that the droplet formation in gas atomization occurred in three stages: primary and secondary disintegration of the molten metal, and solidification.

Figure STAGE shows the droplet formation process for gas atomization. During the primary stage, the liquid stream of molten metal enters into the region of high speed gas and gets some kinetic energy and

momentum from the gas. The ligaments of liquid will separate as the dynamic pressure increases and may be subjected to additional pressure force and undergo further disintegration which is the second stage of atomization. Spherodization and solidification occur in the third stage. The droplets under the influence of the surface tension force revert to a sphere. If solidification of the droplets is complete before the spherodization, the resulting powders will have the shape of ligaments. On the other hand, if spherodization is complete before solidification, the resultant powder shapes will be spherical. During the second stage of atomization, powder droplet collisions can also occur. These may result in the presence of satellites (the design of the atomization chamber will affect the extent of this occurence). Let us discuss the parameters which control the particle size, the spherodization and solidification times of the molten droplets.

Lubanska *et al*[θ] introduced an empirical correlation for gas atomization in that the droplet size of a molten metal stream $x_m$ is related to stream diameter $D$ by:[1]

$$\frac{x_m}{D}=K\left[\frac{\nu_m}{\nu_g W}\left(1+\frac{M}{A}\right)\right]^{1/2} \tag{1}$$

W is Weber Number, defined as

$$W=\rho\frac{v^2 D}{\gamma} \tag{2}$$

Lubanska's model has been modified by Mehrotra,[10,11] and Bradley[12,13] has developed a model for molten metal atomization based on the first principles of Raleigh's instability. These models basically involve similar parameters to that in Lubanska's Equation (1). None of these models though deal with sonic and super-sonic flow conditions around the atomizing nozzle.

The state of current knowledge on the atomization of molten metals from visualization and mathematical modelling efforts can be summarized as follows. In modelling, atomization of molten metals the viscosity, density and interfacial energy of molten metal are all functions of the temperature of molten metal and the given alloy. So the droplet size of a given alloy is determined basically by the thermal history of the droplets, gas property, the flow ratio of molten metal and gas, and gas velocity. These last two variables have a very significant effect on atomization with the other variables playing a secondary role. Since quantitative data of the molten metal properties is very scarce, their dependence with temperature can only be crudely estimated at best. Thus, they are considered as invariant with temperature in analyzing the atomization process. The velocity field of the atomizing gas is determined by the delivery gas pressure and by the nozzle geometry. The high- velocity gas increases the velocity of the free-falling molten metal overcoming the shear resistance and leading to atomized powders. The velocity field of the atomizing medium is a key factor in controling the size of the final powders[1]. It is obvious that particle size decreases with increasing gas delivery pressure and density of the molten metal. However, recent work by Anderson[14] has shown that increases in delivery pressure can result in a minimum of powder size. This minimum is dependent on the interaction of the gas shock wave with the molten metal stream. This again shows the importance of having knowledge about the gas velocity field below the atomizing nozzle. Powder size increases with stream diameter, ratio of molten metal rate to atomizing gas rate and viscosity of the molten metal. It is very difficult to get fine powders of aluminum alloys because of their high interfacial energy.

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[1]Variables are defined in the Nomenclature at the end of the paper.

The relationship between the particle shape and process variables has not been fully quantified. It has been proposed that the spherodization time ($t_{sph}$) can be evaluated according following equation :

$$t_{sph}=\frac{3\pi^2\mu_m}{V\sigma_m}(R^4-r^4) \quad (3)$$

Superheat, spray angle of molten metal and cooling rate influence the shape of powders to a certain degree. If the molten metal is superheated, the shape of the final powders may be spherical[2]. A smaller spray angle results in more rounded powders, while a larger one produces irregular shapes. Iron and copper powders tend to be spherical; but atomizing cast iron can yield both spherical ($d_p$<45μm) and acicular shapes ($d_p$>45μm).[8] In general, it may be stated that if a ligament requires more time to lose its superheat and solidify than to spherodize, the resulting powder shape will be spherical. Conversely, acicular or ligand shaped powders result when spheroidizing times are long than cooling and solid times. Thus powder shape is dependent on droplet size and on the primary and secondary atomization stages. Hence, for a given nozzle, metal, gas system, the operating conditions and the spray field of gas and metal play a dominant role in determining powder shape. Particle collisions in flight result in particles with satellites or other irregularities in shape. If the cooling rate is increased, the occurrence of satellites would be diminished. When helium is used as an atomizing medium, the atomized powders are rounded without satellites[1]. Particle shape may also be affected by the atomising gas composition. When atomizing lead-tin alloys, mostly acicular powder results when atomizing in air; while spherical powder has been observed when atomizing with an inert gas[15]

**Solidification:** The solidification time consists of three parts. The first is the time for cooling the superheated metal to melting point. The second is the time associated with undercooling, and the third is the time required for the particle to solidify and for the heat of fusion to be removed.

Naida and Chiporenko[16] considered the total solidification time to be:

$$t_{sol}=\frac{d_m\rho_m}{6h_c}\left[(C_p)_m \ln\left(\frac{T_i-T_g}{T_m-T_g}\right)+\left(\frac{\Delta H_m}{T_m-T_g}\right)\right] \quad (4)$$

$$h_c=\frac{k_g}{d_m}(2.0+0.6R_e^{0.5}P_r^{0.33}) \quad (5)$$

The surface heat transfer coefficient is correlated to the type of atomizing gas and the slip velocity and is very sensitive to the diameter of droplet. The small powders have high surface heat transfer coefficient. The value of interface heat transfer coefficient of helium is one order of magnitude higher than that of argon[17]. More recent work in rapid solidification has shown that in gas atomization droplets of molten metals and alloys experience significant undercooling. Thus the total solidification time must be calculated on the basis of the undercooling temperature rather than the melting point. Unfortunately, there is yet to be available a quantitative model to predict the degree of undercooling or to measure it under gas atomization conditions.

From equation SPH, SOL and COEF it is seen that the droplet size is the key factor to determine cooling rate. The solidification time is roughly proportional to the square of the droplet diameter. Powders of the same size would be expected to have experienced the same cooling rates.

Undercooling is also an important factor to be considered in refining and controlling the microstructure of powders. A high degree of melt undercooling would get high crystal growth velocity.[17] Prevention of

crystal nucleation during cooling is an effective way to get high undercooling. In the atomization process, the smaller the droplet size, the smaller the number of nucleants in each droplet. It is believed that the majority of melt droplets do not contain any nucleant when the droplet size is less than 10 μm. Thus the degree of undercooling is related to the droplet size.

Numerous experimental results have shown that for many alloys, powder microstructure depends on the powder particle radius. Powder microstructure can be characterized by dendritic arm spacing of solidified powder, cell size or eutectic inter-cellular spacing. FigureDENDR shows that the relationship of dendrite arm spacing and particle diameters. Tewari *et el*[18] considered that secondary dendritic spacing (d) varies with local solidification time $t_s$ in the following manner: $d\alpha(t_s)^m$ where m are experimentally determined from 0.3 to 0.5. Thus the dendritic arm spacing (d) depends on the powder size in this manner, $d\alpha(R)^m$, for given atomizing conditions. This implies that the same size powders are expected to get same dendritic arm spacing because they will experience the same cooling rates. Hence, for given atomization conditions and alloy system powder size infers the microstructure of powder product. Finer powder microstructure in turn will yield a finer microstructure of the consolidated product with concominant improvement in mechanical properties such as yield and creep strength.

**Summary:** In summary the atomization process involves several basic phenomena: primary and secondary atomization, spherodization, heat transfer and solidification. Primary and secondary atomization involve complex two phase momentum transfer between the molten metal and the atomizing fluid which influences to a great degree the resultant droplet size. Spherodization strongly depends on the size of ligands and the interfacial tension and viscosity of molten metal. For a given atomizing system, and under known control parameters, the heat transfer and solidification characteristics will vary with powder size for many alloy systems. There still remains to be developed a comprehensive mathematical model of the molten metal atomization. However, it appears that knowledge of the size of the droplets, of the slip velocity between droplets and the atomizing fluid and of the atomizing spray can be related to powder size, shape and microstructure. Clearly, therefore, the gas velocity field and the molten metal spray *vis a vis* their velocities and the size of the droplets are the most critical parameters which control all the basic phenomena comprising the atomization process. The challenge is therefore to monitor the gas and droplet velocity fields and to measure the droplet size. Such knowledge would be readily used to monitor and control the atomizing process to improve powder yield and, to control powder characteristics of size, shape and microstructure and to develop new knowledge about the atomization process.

## Needs for Intelligent Sensors

Figure 4 shows a block diagram of the automated materials processing approach. For molten metal atomization, the key process parameters to measure *in situ* have been identified in this report to be metal and gas flowrates, and powder size and velocity. Since there is readily available and well established instrumentation for measuring molten metal and gas flowrates under production environments, sensors for these parameters will not be discussed in this work. If these parameters (process variables) could be measured or monitored on-line by sensors, the measurements interpreted, and fed back to the control system, it would be possible to predict final powder characteristics. A list of potential sensors and their principle of operation is shown in Table 2. While none of these sensors have been used to control or monitor an atomization process, their principle of operation and their current state of technological development make them candidates for consideration.

Among the desired characteristics of the sensors are that they be non-intrusive and should work reliably in high ambient temperature, high ambient light, and high dust particle density environments. Usually non-intrusive sensors are based on electric, electromagnetic or optical principles. The optical based sensors are the most developed. Optical methods for particle sizing can be divided into imaging and nonimaging techniques. The imaging techniques include microphotography, holography and cinematography; while nonimaging technique can be considered as the ensemble or multi-particle technique and single particle counters.

The remainder of this paper will describe those sensors that are believed to be potentially suitable for atomization processing of molten metals. These will include the laser diffraction particle analyzer (LDPA), various particle counting instruments, sizing and velocity measurement (PCSV) instruments, phase/doppler particle analyzer (P/DPA) instruments, various imaging techniques for particle sizing (ITPS), and the closely related infrared image analysis (IIA) techniques. Two electric principle based monitors are discussed briefly.

## Laser Diffraction Particle Analyzing Technique (LDPAT)

The LDPAT is an ensemble particle technique. It has found widespread applications in the fields of spray, atomization and combustion[19,20,21,22,23,24]. The advantages of LDPAT include wide measurement range of size (1.2 to 1800 μm), independent measurement of location and velocity of particles and the ability to make *in situ* non-intrusive measurements. Figure DIFFRACTION schematically shows typical apparatus.

The illuminating source is a monochromatic, coherent laser. It is expanded to several millimeter diameter (9 mm for the Malvern 2600 instrument) and passed through the particle field. The undiffracted portion of light is focused onto a nominal point on the focal plane at the center of an array of concentric annular detectors. All the particles in the collimated beam cause diffraction of light. If the diameter of a particle is significantly smaller than the wavelength of the light used (632.8 nm for the He-Ne laser), the intensity of the light diffracted in the near forward direction is nearly independent of the refractive index and the shape of particles. The exact position of the particle within the beam and its velocity are not important and the light intensity distribution depends on the particle size distribution only. The intensity distribution is a series of concentric light and dark rings.

The angular light intensity of monodisperse of spherical powders described by Frauhofer diffraction theory is a function of particle size:

$$I(\theta) = I_0 \frac{\alpha^4 \lambda^2}{16\pi^2} \left( \frac{2J_1(\alpha\theta)}{\alpha\theta} \right)^2 \tag{6}$$

In a practical system, particles have their size distribution, all particles diffract light onto all detectors. The real angular light intensity is given by

$$I(\theta) = \int_0^\infty n(\alpha) I_0 \frac{\alpha^4 \lambda^2}{16\pi^2} \left( \frac{2J_1(\alpha\theta)}{\alpha\theta} \right)^2 d\alpha \tag{7}$$

The diffracted light is focused by the receiver lens, and converted into a radial distance at the detector plane, which consists of annular concentric ring diodes (31 diodes for Malvern products). The relationship between the diffraction angle (θ) and the radial distance (r) is $\theta = r/f$ where f is the receiver lens focal length. The smaller particle size corresponds to the larger diffraction angle (θ) and the larger radial

distance (r). From Equation (7) we see that to measure a wide range of particle sizes, the detector has to cover a very large range of intensities. It is difficult to produce a detector with adequate dynamic range. This is the reason that a detector consisting of 31 concentric annular rings is used. The areas of the rings increase with increasing mean radius. The ratio of the largest ring diode area to the smallest one is about 1900. The signals of all the detectors are thus of the same order-of-magnitude. Table RIN shows the ring diode dimension.

The measurement ranges of particle size are related to the focal lengths of the lenses used. It is impossible to cover all measurement ranges from 1.2 to 1800 μm using a single lens. The computer averages the output of adjacent detectors and produces in 15 paired-data. All powders in the laser beam diffract some amount of light onto all rings. The size distribution could be calculated according the intensities obtained from the detector rings and knowledge of inverse matrix. There is no unique relationship between size range and ring numbers. The Table LIMITS gives the 15 size classes of Malvern 2600 instrument for 300 mm lens.

The size range of atomized powders is from decades to five hundred micros. It is just covered by the 300 mm lens. Table LENS gives the particle size ranges for different lenses.

There are some limitations caused by the apparatus itself. The first one is poor resolution while measuring at upper size limits of lenses. The areas of the ring detectors are based on avoiding great intensity variations. so size limits are not even for rings. If good resolution for upper size limits is needed, a redesign of the detectors is required. The second is the limited particle concentration. Constant incident intensity, and photons scattering one at a time off single particles, are the premises of the theoretical model. Actually the incident light is obscured by particles, and the diffracted light can encounter other particles before it reaches the receiver lens. These multiple scatterings result in broadening of measurement size distribution, and in decreasing the reported average particle measurement size.

The Opacity (defined as DCL, where C is the particle concentration, L is the laser path length, D is the particle diameter) serves as a check on whether multiple scattering is significant. Reasonable opacities are bigger than 0.005 and less than 0.5. Choosing D = 50 μm and L = 1 m, reasonable concentrations are from $6.4 \times 10^{5}$ to $6.4 \times 10^{8}$ particles-$m^{-3}$. The indicated distribution will be broader in distribution and smaller in average size than the actual ones at high concentration because of the multiple scattering.

The third limitation is that the detector is sensitive to ambient light intensity. The molten metal radiation at the top of the atomization tower may overload the optical detector. The heat radiation from hot powders at the bottom of the atomization tower may contribute to errors in size measurements. Use of an argon laser or narrow band filtering are solutions to consider. Beam steering is a common problem for optical based methods with high temperature processes. Thermal gradients in the airpath result in spurious signals, especially in the big particle ranges. Dodge *et al*[25?] extended the applicability of diffraction-based size instrumentation by using a chopped laser beam and a narrow band interference filter. These modifications make measurements possible at temperatures and pressures as high as 700 K and 586 KPa.

## Particle Counting, Sizing and Velocity Measurement (PCSV)

This single particle counting technique relies on a robust theory for scattering in the near-forward direction. The illumination source is a focused laser beam whose (1/e)-diameter is about 20 to 30 μm. Figure SING shows it in block diagram.

The measured particles pass through the measurement volume perpendicular to the beam. The scattering signals observed by the detector have a Gaussian distribution in time, as shown as Figure GAUSSIAN.

Particle size and velocity are related to $A_j$ and $(t_p\text{-}t_s)$ separately. Holve[26] considered that the scattering amplitude $A_j$ is a function of gain constant $B_1$, the local illumination intensity in the sample volume I(X, Z) and the absolute response function $F(d_j, m)$:

$$A_j = \beta_j I(X,Z) \bullet F(d_j,m) \tag{8}$$

The optical system is designed so that a monotonically increasing signal amplitude with particle size, and minimum sensitivity to complex refractive index and shape of particles, are obtainable. The diffraction component of scattered light depends only on the projected surface area of the scatter, and the light collection system is designed for the near-forward mode (scattering angle < $1^o$). Hence it is fairly insensitive to material properties such as refractive index and shape. If the particle properties and shape are known and invariant, the use of backward scattering geometries is possible.[27] The backward scattering configuration is preferred to forward mode in industry sometimes, because of its advantages: simplicity of configuration (all of optical components are fixed to a single, stable breadboard ) and small misalignment (both the incident and scattered light pass through the same paths and experience the same change of the average refractive index ). The disadvantages of backward mode are lower intensity of backscattered light and greater sensitivity to the complex refractive index of particles. If the scattering properties are not well defined, it would be better to use a diffractive forward-scatter configuration to avoid sizing ambiguities.

We can see from Equation SINGLE that the amplitude of scattered light is a function of particle size, and particle trajectory through the measurement volume. The same result is obtainable when a big particle grazes the measurement volume as when a small particle passes through the center of the measurement volume. To overcome the ambiguity, the trajectory dependence inversion technique is suggested by Holve[26,28,29]. An equal probability of passing through any element of the cross section of the sample volume, and equal mean velocity, are assumed for all particles in the measurement volume. Equation NUM shows the relationship among $N_j$ concentration of particles in a size range $\Delta d_j$, $C_i$ signal count rate in an amplitude range $\Delta A_i$, U mean velocity of particles and $\Delta \underline{S}_{ij}$ cross sectional area of the measurement volume normal to the flow direction, which yields signal peak amplitudes in the range $\Delta A_i$ for particles in the size range $\Delta d_i$

$$= U \tag{9}$$

$$\underline{\mathbf{N}} = (U\underline{\Delta S})^{-1}\underline{\mathbf{C}} \tag{10}$$

Equation (10) is written symbolically for Equation NUM.

If we make a monodisperse particle distribution of known diameter and concentration, passing through the measurement volume at known mean velocity, the $\Delta\underline{S}$ matrix will be determined according the count rate distribution $\underline{C}$, which is given by the pulse height analyzer. Then the $\underline{N}$ can be solved from $\underline{C}$ for unknown particle size distribution. This technique is based on absolute measurement, the signal amplitude is subject to electronic drift and to the obscuration of the window by atomization powders. This technique is usually thought of as giving absolute data, numbers-cm$^{-3}$ for number density, gm-cm$^{-3}$ for mass density and m-sec$^{-3}$ for velocity. The basic advantage of single counting technique over the ensemble are capability of giving spatial resolution and simple optical configuration (single beam) which is important for high temperature environment to prevent beam steering. Because there is no inherent spatial averaging with this technique, if it is to be used in industry for real time monitoring, the spatial sampling point must be carefully chosen.

## Phase/Doppler Particle Analyzer (P/DPA)

This is a single particle counting technique which gives the information on size distribution, velocity of powders at a specific point. Figure PHAS shows a typical dual-beam optical configuration.

It consists of transmitter, receiver, and signal processor. The illuminating source is usually continuous gas laser (helium-neon or argon ion). The highly collimated light beam is divided into two parallel beams of equal power by a beam splitter. They are focused by a good quality lens. The region of intersection is usually called the measurement volume or sample volume. The receiver includes the collection optics, aperture, one or more photomultiplier tubes, and corresponding signal preamplifiers. The signal processor mainly consists of an input conditioner and a timer. Let's consider the measurement volume in more detail. The intersection of two coherent beams is an ellipsoid whose size depends on the laser power, the illuminating beam focusing, the scattering properties of the particles, and photodetector sensitivity.

When a particle passes through the measurement volume, it is illuminated by two beams and scatters light from both beams to produce a heterodyne signal. The scattered light is collected by the receiver lens, and focused through the aperture onto the photodetectors. The two scattered light waves are mixed by the photodetector and the Doppler shift signal is detected by using the square-law property of the detector. Figure DOPP shows the Doppler shift signals.

The Doppler shift frequency is a function of particle velocity, laser wavelength, and intersection angle:

$$f_d = 2U_x SinK/\lambda \tag{11}$$

The velocity component perpendicular to the bisector of the two beams is determined by Equation (11). When multiple photodetectors are used, they detect multiple Doppler shift signals which have same shapes but different phases. Figure DOPP shows high-pass filtered Doppler burst signals whose pedestal has been removed. Phase shift is determined by measuring the time between the zero crossings of the signals from detectors 1 and 2 and dividing by the measured Doppler period.

$$\Phi_{1-2} = \frac{\tau_{1-2}}{\tau_D} x 360^0 \tag{12}$$

By using the computed linear relationship shown in figure F-D, the powder size related to the phase shift is given. The DELTA in Figure F-D is the fringe spacing which is a function of the light wavelength and the laser beam intersection angle. The slopes of the linear relationship are changed, while changing the optical parameters, including the laser beam intersection angle, collection angle and laser wavelength.

Three photodetectors are required to ensure an unambiguous measurement when phase shift is bigger than $360^0$. The third detector provides a logical test identify and eliminate the ambiguity of measurements. As long as the scattering light is dominated by reflection the phase shift is linearly dependent on diameter, and is independent of the particle of index refraction.

This technique is based on Doppler shift frequency and phase difference rather than intensity of scattering light. It thus has high immunity to interference. It also offers reasonably good spatial resolution, and is capable of tracing very high frequency velocity fluctuations. The significant advantage is the relative insensitivity to the scattered light intensity which can cause uncertainties in the scattered-light-intensity mode. The linear relationship between measured phase and the dimensionless powder size creates a uniform size sensitivity and simplifies the implementation of this method. Backward mode based on reflected light is suitable for opaque particles. Its repeatability and accuracy are good enough for velocity measurement, but if the particles have a wide size distribution, the measured velocity distribution will be anomalously broad. Calibration is not needed for this technique but other techniques or instruments are needed to set the instrument parameter properly.

Another approach is to relate the visibility to particle size. The visibility is defined as the ratio of the amplitude of Doppler signal to the amplitude of pedestal

$$Visibility = a\text{–}c\ magnitude/pedestal \tag{13}$$

Famer[??] concluded that the visibility in forward or back ward scattering is given by

$$\overline{V} = \frac{|J_1(\pi a/d_f)|}{\pi a/d_f} \tag{14}$$

Figure VISB gives Doppler signals and V vs $a/d_f$.

Some work has been done to measure particle size by using the relationship between visibility and particle size, but a few limitations should be pointed out. First, the size dynamic range is limited. When the visibility exceeds 0.95 the curve is quite flat. This means the size resolution for small particle size is poor. On the other hand, the measurement ambiguity occurs at lower visibility. Secondly, the visibility is

related to intensities of beams. When a large particle passes through the individual beam, the variation of beam intensity is expected, it will contribute to big error.

The gas velocity and turbulence intensity are inferred from seed particle measurements. As a seed particle the most important property is small diameter and narrow size distribution to get narrow PDFs and satisfactory data which are not sensitive to the instrument parameters, such as gains, bandwidth of filters. The signal amplitude is a function of particle size, refractive index (reflective index for backward mode) and the trajectory through the sample volume. A conventional LDV cannot discriminate signals from small and large particles which have different scattering properties.

## Imaging Technique for Particle Sizing (ITPS)

Imaging techniques have long been used to determine size of particles. Automatic analysis of images using computers have already been used to study combustion and atomization sprays. Oberdier[30] developed an instrument system to automate the analysis of fuel spray imaging using computer vision. Figure 12 shows a block diagram of this system.

The iluminating source is a pulsed laser whose duration is 10 to 20 nsec. The particles with high velocity seem to be 'frozen,' because of the extreme shortness of the laser pulse. A second pulse follows after a fixed time (typically 10 μs) to make a double exposure for velocity measurement. On the opposite side a television camera receives the images, which are a mixture of in-focus and out-of-focus particle images. The thousands of images are stored on a surveillance type recorder for re-analyzing later. Deciding which particles are in-focus and measuring their diameters, pairing multiple powder images and determining velocity are the steps of image analyzing.

It is said that months of effort are required for manual analysis. Thus great effort is being expended on developing automatic image analysis systems. A common goal is to analyze an image every 10 seconds. This system has several functions: image storage, image processing and particle statistic computation. The particles having considerable shading and diffraction ring structure are classified as in-focus.

Image processing includes thresholding image restoration, segmentation, feature extraction and classification. The key to this technique is automatic image processing. Most errors come from failure to correctly decide which particles are in focus. Schafer *et al*[31] developed a semi-automatic evaluation system to analyze double pulse holograph for local measurement of particle concentration, size, and velocity in the sheet of a flat spray nozzle. He concluded that holography was rather troublesome and time consuming, to be used only in the absence of other alternatives.

## Infrared Image Analysis (IIA)

The goal in IIA is not to concentrate on a single point in the process, but rather to analyze the characteristics of the entire spray. This is in contrast to optical techniques which would monitor the atomization process by measuring particle size and velocity of a specific point in the spray. In the IIA approach, the shape and color of the atomization spary of liquid metal is correlated to the powder temperature for a specific metal. The relationship between cooling rate and particle size can be estimated from a data table of results. Small particles have high cooling rates, and consequently attain low temperature quickly. The operators of atomization processes can regulate the process according to

the properties of the spray cone of atomization. Of course the sensitivities of the human eye are different for different operators and the personal experiences are limited. An infrared camera is therefore expected to be an alternative to the human eye and an expert system would combine the operator experience and expert knowledge. As is well known, infrared radiation is extremely sensitive to the temperature. The Stephan-Boltzmann Law states the radiation from a blackbody as:

$$W_{tot} = \sigma T^4 \tag{15}$$

Equipment similar to that used for imaging (infrared television cameras, recoreders, etc.) can be used to record and analyze the infrared images.

By sampling and analyzing the metal powders from the process, and then comparing the stored infrared images with the analyzed data, the inferred relationship between the characteristics of the cone (shape, color, and angle) and the final properties of the powdercould be found for a specific condition. An expert system mau be built to predict the properties of metal powders. This method is suitable for the process which is not understood exactly or difficult to model using mathematical analysis. The knowledge base of this expert system may be enlarged and modified with further research which allows a better understanding of the process. The most important step for understanding more about the atomization process is studying and accumulating the information or relationship between the cone properties and parameters of the final products.

## Electric Principle Based Monitor for Particulates (EPMP)

The disadvantages of optical principle based sensors, such as complex alignment of optical system, high cost and potential of contamination of lenses, limit their application in industry. While these sensors which could give parameters of metal powders with high accuracy, there is room for sensors which simply monitor the process and which provide an indication of changes in specific parameters such as concentration and average diameters. Following are some electic-principle based monitors. We discuss first the surface ionization monitor for particulates, then the electirc noise method.

When an atom or molecule whose ionization potential is comparable with the work function of a metal comes into contact with the heated metal surface, the atom or molecule may leave the surface as a positive ion. In the SIMP instrument, ions produced by this process are collected by an ion collection electrode. The burst of ions accompanying impact of a particle containing surface ionization impurities produce a sizable electrical pulse. Sodium and potasium, which are the sixth and seventh most abundant elements on earth, are easy to surface-ionize. Besides sodium and potasium other atoms, ranging from aluminum through zirconium (Al, Ba, Ca, Eu, In, Ca, Pr, Th and U) have this property. The hot surface is a wire filament (usually tungsten) and resistivly heated, that maintains a constant resistance and therefore approximately constant temperature. An electrical potential difference between the filament and an adjacent ion collection electrode collects the ion bursts.

The powders produced by atomization usually, contain enough impurities with low ionization potentials that we would expect them to be sensitively deltected.

Although this technique is not non-intrusive, the filament and collection electrode are not large enough to seriously interfere with the flow. The advantages of this technique are high sensitivity ( 0.01μm in diameter is detectable) and simplicity. File *et al.*[32] points out that this detector had selective behavior.

While sensitivity detecting particles containing alkali and other surface ionizable constituents, it responds only very weakly to other particles such as combustion products, water mist and photochemical smog. Pulse height analysis gives a particle size distribution, although calibration may be difficult and the relationship between pulse height and particle diameter is both non-linear and not independent of material characteristics.

A similar technique is called electric noise method[33]. Solid powders moving in a turbulent gas stream always acquire some electrostatic charges because of triboelectrification. When a metal probe is inserted into the spary, the collected signals depend on the type, concentration, and size of the powder. The signal current is strongly sensitive to the concentration, but not strongly sensitive to the size. A sample device is used to draw samples from the spray. By using a cyclone seperator the sample stream is divided into two streams: overflow with fine powders and underflow with coarse pwders. The relative mass flowrates of powders in the two streams are determined by the size distribution of the powder. The size measurment is converted to concentration measurement. The ratio of concentrations is calibrated against average size for specific powders. The sampled powders could be sent back to the spary with the gas stream, otherwise taken away for on-line or off-line analysis if necessary. Similarly, the height analyzer used in the SIMP method could be available to analyze the electric noise signals.

## Isokinetic Sampling Probe

It is very difficult or nearly impossible presently to measure some powder characteristics on line such as shape, microstructure and chemical compositions of metal powders. Furthermore, there is also a need to check the measurements obtained using the sensors described above. One approach is to withdraw particles from a known location of the spray in the atomization chamber, and to characterize the sample off line. The key point of this sampling approach is isokinetic sampling conditions. This means the velocity in the sample probe must be equal to the average velocity at the measuring point, otherwise the volume to be withdrawn will not be representative of that point in the stream.

Fig. 13 shows an automated system to maintain the isokinetic conditions. The sampling probe consists of two parallel tubes placed into the direction of the stream, both containing radially located thermistors. The resistance of the thermistor is sensitive to both temperature and flow rate. The resistance difference of two thermistors is picked out by a bridge. If the sampling condition is isokinetic, the output of the bridge should be zero. The differential measurement could not provide for changes in velocity during sampling. However, a measurement of resistance of the thermistor in the open tube can be taken periodically, a comparison of the readout will indicate the fluctuations of flow rate and temperature at the sampling point. Samples of powders can be removed from the cyclone and the concentration can be calculated from the flow meter in the sampling tube.

## Summary

It can be shown that control of the size of droplets produced in atomization will provide control of powder size, shape and microstructure. All of the important basic phenomena of atomization processing are a strong function of the droplet size. Although the atomization has not yet been fully described by a mathematical model, a combination of the best available models and a heuristic understanding of atomization can be used with sensors to develop intelligent sensors or an intelligent processing approach

to atomizing metals and alloys.

The sensors available for measurement and monitoring of atomization processing of molten metals and alloys have been discussed. Table 5 summarizes the behaviors of these sensors to allow a quick comparison of their advantages, limitations and capabilities. Every sensor is based on a different principle and has its inherent limitations: no one is considered to be "the best." The comparison between methods based on different principles is very difficult, because the measurement volume varies from a single point to a large volume and the sampling periods are not the same. Single particle counting, sizing and velocity measurement seems to be suitable for research to study the process of atomization. In this approach, particle size distribution, velocity and concentration are given in absolute units simultaneously. The electric principle based monitor for particulates and infrared radiation analyzing may be worth developing as an industrial monitoring technique because of their relative simplicity and ruggedness, and the sanguine prospects for integrating them into intelligent materials processing systems. However, these instruments will require a great deal of calibration to standardize their signals and to relate their output to specific processing parameters and conditions.

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# Nomenclature

| | |
|---|---|
| A | mass flow rate of gas |
| a | diameter of the particle |
| C | constant |
| $(C_p)_m$ | heat capacity of metal |
| D | diameter of molten metal stream |
| $d_f$ | fringe space |
| $d_m$ | mass median particle diameter |
| $f_d$ | Doppler shift frequency |
| $\Delta H_m$ | latent heat of fusion of metal |
| $h_c$ | convective heat transfer coefficient |
| $I_0$ | incident light intensity (assumed constant) |
| $J_1$ | first order Bessel function of first kind |
| K | constant |
| k | intersection angle of beams |
| M | mass flow rate of molten metal |
| m | complex refractive index, $n_1 + in_2$ |
| Pr | Prandtl number for gas |
| R | particle radius after spherodization |
| Re | Reynolds number for particle |
| r | particle size before spherodization |
| T | absolute temperature of the body |
| $T_g$ | temperature of gas |
| $T_i$ | initial temperature of particle |
| $T_m$ | melting point of metal |
| $u_x$ | velocity of particles(which is perpendicular to the bisector of two illuminating beams in the same plane) |
| V | volume of particle. |
| v | velocity of atomizing gas |
| W | total radiation per unit area and solid angle |
| $x_m$ | mass median particle diameter |

| | |
|---|---|
| $\alpha$ | dimensionless size parameter. $\alpha = \pi D/\lambda$ |
| $n(\alpha)d\alpha$ | is the numbers of particles with sizes between $\alpha$ and $\alpha+d\alpha$ |
| $\Phi_{1\text{-}2}$ | phase shift of Doppler signals |
| $\gamma$ | surface tension of molten metal |
| $\lambda$ | wavelength of incident light |
| $\lambda$ | wavelength of laser ***** change ***** |
| $\mu_m$ | viscosity of molten metal |
| $\nu_m$ | kinematic viscosity of molten metal |
| $\nu_g$ | kinematic viscosity of gas |
| $\theta$ | diffraction angle between incident and diffracted light |
| $\rho_m$ | density of molten metal |
| $\sigma$ | surface tension of liquid metal |
| $\sigma$ | Stephan-Boltzman constant, $5.672 \times 10^{-12}$ W-cm$^{-2}$-K$^{-4}$ |
| $\tau_{1\text{-}2}$ | time difference of of signals from detectors 1 and 2 |
| $\tau_D$ | period of Doppler signal |