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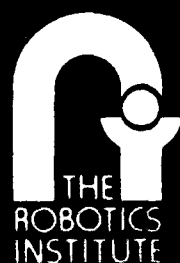
Intelligent Sensors for Atomization Processing of Molten Metals and Alloys

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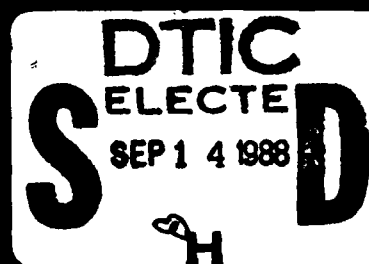
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Technical Report

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We briefly introduce the principles of atomization processing of molten metals and alloys, discuss potentially applicable instrumentation, and propose opportunities for developing intelligent sensors for monitoring and control of the process. Particle size and velocity are considered the two most important variables to monitor and control the atomization process. Several on-line sensors suitable for atomization of molten metals and alloys are compared: laser light scattering methods using small angle scattering analysis, moderate angle scattering analysis, and doppler analysis; electrical methods using surface ionization and triboelectric charging, and image analysis. The isokinetic sampling 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.

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Contents

Abstract

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Atomization Process and Sensor Overview

Atomization is the preferred method for producing rapidly solidified powders of metals and alloys. These powders have outstanding or specialized properties that cannot be obtained by conventional processing routes. The benefits of rapidly solidified powders of metals and alloys are mainly decreased segregation, controlled grain size and particle size distribution, increased solid solubility, and elimination of segregation phases. These specific microstructures mean good chemical homogeneity, high corrosion resistance and advanced mechanical properties, particularly hot and cold workabilities [11, 25, 18]. 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 product rejection rates are high. These problems have created challenges for sensors to monitor production and characterize the product. The complexity of the atomization process, and of the subsequent part manufacturing processes as well, dictate that suitable sensors will have to extract relevant measurements from complex data sources, relate them to process control parameters, and suggest suitable process adjustment, both upstream and downstream, in context of product application requirements. In the jargon of our day, atomization needs *intelligent sensors*.

Parameters

The parameters of the atomizing process can be divided into three types: *control, dependent*, and *product* variables. Control parameters include the temperature of molten metal, the pressure of the atomizing medium, 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. Product parameters include size and size distribution, and measures of 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, then to characterize the powder 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 failed to produce high yields of commercial powders of the desired characteristics. Thus atomization remains an expensive process, more an art than a science, and the development of fine powders customized for novel applications has been an illusive commercial goal. The challenge is a two step one: first, to develop intelligent sensor systems for the accurate and consistent measurement of relevant powder characteristics *e.g.*, powder size, distribution, shape and microstructure, and second, taking context into account, to automate measurement feedback for process control. In the first step, these sensors would be used in research to improve understanding of the atomization process, which would lead to the second step and its application in on-line production process control. Certain issues such as powder quality, *e.g.*, foreign particle content, are for the present deferred.

Processes

Atomization processes can be divided into twin fluid atomization, centrifugal atomization, and mechanical atomization [18]. In the best known twin fluid method a molten metal stream exits a die in the bottom of a reservoir under gravity alone, and atomization is effected by its downstream interaction with a high pressure stream of water, helium, argon, or hydrogen. Centrifugal atomization can be subdivided into rotating electrode, rapid rotating disc, and rotating perforated cup processes; since the droplets of molten metal spheroidize and solidify in flight, or can be gas-quenched as they

leave the rotating carrier, post-atomization measurement needs for centrifugal atomization have much in common with twin fluid atomization. Similarly, techniques to get small droplets by mechanical means, which include twin roll atomization, vibrating electrode atomization and the Duwer gun technique, involve the generation and solidification of metal droplets. Because of this commonality with respect to sensing needs, it is adequate to restrict the rest of our examples to the twin fluid method.

While details internal to the sequence of events that culminate in the formation of powder with given characteristics are neither well understood nor fully defined, the phenomena that play an important role are known to include atomization itself, *i.e.*, the break-up of the liquid metal 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 particles (to avoid recalescence). Ideally these steps would occur in neat sequence and yield very fine powder of controlled shape and size distribution. However in practice these process steps overlap.

Mechanism

The mechanism of the molten stream break-up is common to all molten metal atomization schemes, so will be described only for the gas atomization process. See and co-workers [30, 31] and Seaton and co-workers [29], observing high speed movies of atomization, concluded that the droplet formation in gas atomization occurred in three stages: primary and secondary disintegration of the molten metal, and solidification. During primary disintegration the stream enters the region of high speed gas from which it acquires additional kinetic energy and momentum. The stream breaks up into ligaments of liquid that separate and undergo further disintegration as the dynamic pressure increases. The third stage is solidification, preferably preceded by spheroidization, depending on the relative time constants for dissipation of internal mechanical energy *vs* thermal energy. If the latter time constant is long enough, the droplets under the influence of the surface tension force revert to spheres. However if solidification of the droplets is complete before the spheroidization, the powders will have the shape of ligaments. Higher order phenomena, *e.g.*, powder droplet collisions, must also be considered in a complete treatment. Details and empirical models of droplet size *vs* stream diameter material properties can be found in Lubanska [19].

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, the flow ratio of molten metal and gas, gas velocity, and gas thermal and transport properties. Since quantitative data on the molten metal properties are scarce their dependence on temperature are rarely known even crudely, and in most models this dependence is simply ignored. 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 thus a key factor in controlling the size of the final powders [25]. It seems obvious that particle size decreases with increasing gas delivery pressure and density of the molten metal; however recent work by Anderson [2] has shown that there is a minimum in the powder size *vs* gas pressure curve. The optimum pressure depends on details of 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 flow rate to atomizing gas flow rate, and viscosity of the molten metal. Various other factors may dominate in specific cases; for example, it is difficult to get fine powders of aluminum alloys because of their high interface energy.

Heuristics

Atomization studies have generated a wealth of empirical knowledge that constitutes a powerful database for heuristically justified automated control strategies based on marginally understood correlations between sensor data and powder characteristics. For example: superheat, spray angle of molten metal, cooling rate, and metal or alloy all influence the shape of powders. If the molten metal is superheated, the final powders are likely to be spherical [11]. Smaller spray angles result in rounded powders, while larger ones produce irregular shapes. Iron and copper powders tend to be spherical, while those of lead and tin are irregular. In atomized cast iron, finer particles are acicular in shape, while the under 45 μm fraction are spherical [29]. If a ligament requires more time to lose its superheat and solidify than to spheroidize, the powder shape will be spherical. Acicular or ligand shaped powders result when spheroidizing times are longer than cooling and solid times. Powder shape is dependent on droplet size and on the primary and secondary atomization stages. For a given nozzle, metal, and 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 are diminished. When helium is used as an atomizing medium, the atomized powders are rounded without satellites [25].

Summary

In summary, the atomization process involves several basic phenomena: primary and secondary atomization, spheroidization, heat transfer and solidification. Primary and secondary atomization involve complex two phase momentum transfer between the molten metal and the atomizing fluid that influences the resultant droplet size. Spheroidization 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 the gas and molten metal velocity fields and the size of the droplets are the most critical parameters that control all the basic phenomena comprising the atomization process. The first challenge is therefore to monitor the gas and droplet velocity fields and to measure the droplet size distribution. This knowledge, combined with heuristics relating the measurements to control strategies, constitutes an intelligent processing approach to improving powder yield by achieving the desired characteristics of size and size distribution, shape, and microstructure. Furthermore the structure of this program naturally assumes a form amenable to generating new knowledge about the atomization process, and automatically and continuously modifying the control system to take advantage of this new knowledge.

Intelligent Sensors for Atomization Process Control

For molten metal atomization, the key process parameters to measure are generally agreed to be powder size and velocity. In the intelligent processing scenario, these process variables would be measured on-line and the measurements interpreted and fed back to the control system. Intelligent sensors report their measurements in terms familiar to the people who are receiving them, and thus exactly in the terms expected by a control system based on rules derived from human experience (expert systems). Thus the velocity sensor should report the mean velocity, velocity distribution, and a measure of turbulence in terms appropriate to the context or listener, whether operator, engineer, or computer. Similarly the size sensor should report the size and size distribution according to

appropriate conventional measures (rosin rambler, normal, log normal, etc.), and similarly for powder concentration.

The generally perceived most urgent need for intelligent atomization processing is the on-line *in situ* sensing of powder size and velocity. The principles of operation and current analogous applications of several sensors make them potential candidates for consideration. In addition to meeting the specific measuring requirements, viable sensors must be non-intrusive, and must work reliably in high ambient temperature, high ambient light intensity, and high dust particle density environments. Usually non-intrusive sensors are based on electric, electromagnetic or optical principles, the latter being most developed in fields obviously related to atomization. Optical methods for particle sizing can be divided into imaging and non-imaging techniques. The imaging techniques include microphotography, holography and cinematography; non-imaging techniques can be further divided into ensemble or multi-particle techniques and single particle counting techniques.

The rest of this paper surveys the sensing principles that we believe are suitable for atomization processing of molten metals. These include three laser scattering methods (diffraction particle analyzers *LDPAs*, sizing and velocity measurement instruments *PCSVs*, and phase/doppler particle analyzer instruments *P/DPAs*, image analysis techniques, two somewhat speculative electrical principle based monitors using surface ionization and triboelectrification respectively, and the isokinetic sampling probe approach to on-line particle collection and characterization.

Laser Scattering Techniques

Laser light is typically highly monochromatic, spatially and temporally coherent, intense, and in the time domain either of potentially very stable intensity at moderate power (continuous wave lasers) or very short duration at very high power (pulsed lasers). These properties make it easy to collimate and transmit laser light with very little angular divergence over large distances, to focus it to a diffraction-limited spot size, to split and recombine laser beams to observe various interference, diffraction, and heterodyning effects, and to observe, with high signal-to-noise ratio, even weak optical phenomena in the presence of large backgrounds of continuum radiation. This properties can be exploited in numerous configurations to make particle concentration, size, and velocity measurements. Future instrument development is anticipated to make it also possible to measure the internal variables of individual particles, including shape and microstructure. For the present, we concentrate on three laser light scattering techniques for particle concentration, size, and velocity measurements: moderate angle forward scattering, realized as laser diffraction particle analysis or *LDPA*, small angle forward or backward scattering, realized as particle counting, sizing and velocity measurement or *PCSV*, and doppler shift measurements, realized as phase/doppler particle analysis, or *P/DPA*.

Laser Diffraction Particle Analysis

Laser diffraction particle analysis *LDPA* is an ensemble particle technique that has found widespread application for non-intrusive *in situ* measurement in the fields of spray, atomization and combustion [5, 12, 21, 32]. Its advantages include a large measurement range (approximately 1 μm to 2 mm), and independent measurement of location and velocity of particles.

The light source is a laser whose monochromaticity and coherence are exploited. The beam is expanded to about 1 cm diameter and passed through the particle field. In the Rayleigh scattering regime, *i.e.*, when the particle diameters are substantially smaller than the wavelength of the light used (0.6328 μm 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. For the larger particles of primary interest in metal atomization, the more complicated Mie scattering theory (of which Rayleigh theory is the small particle limit) must be used. In any case, the positions of the particles within the beam and their velocities are not important: the angular distribution of diffracted light intensity depends only on the particle size distribution. Thus when a lens intercepts the transmitted and scattered light, the undiffracted portion arrives at a diffraction spot at the center of the focal plane, and the diffracted or scattered light arrives elsewhere. Recalling that the focal plane is an angular focusing locus, i.e., all rays parallel to each other are focused to the same point in the focal plane, and all rays with the same inclination to the axis are focused to the same annulus, it follows that an array of concentric annular detectors will report the distribution of scattering angles, and thus, with an appropriate deconvolution model, the distribution of particle sizes. The Rayleigh scattering model obtained by using Fraunhoffer diffraction theory for a monodisperse spherical powder is

$$I(\theta) = I_0 \, \frac{\alpha^4 \lambda^2}{16 \pi^2} \, \left(\frac{2 J_1(\alpha\theta)}{\alpha\theta}\right)^2 \tag{1}$$

where $I(\theta)$ is the angular distribution of intensity, I_0 is the incident intensity, λ is the optical wavelength, α is the dimensionless particle size parameter $\pi D/\lambda$ where D is the particle diameter, and J_1 is the first order Bessel function of the first kind. The observed angular distribution can be fit to this form directly. The practical case of a distribution of particle sizes can be handled by Fourier analysis methods.

As is universal in instrumentation, the method has limitations. The first limitation is increasingly poor resolution at larger scattering angles corresponding to smaller particles: the inevitable signal vs resolution tradeoff is made by increasing the annular detector width with increasing scattering angle, thus avoiding large dynamic range demands on the signal handling electronics. The second limitation is on particle concentration: the method presupposes that each out-of-beam photon has been scattered only once, by a single particle. Failure to take multiple scattering into account results in a shift of the apparent particle size distribution toward smaller particles. Since the particle density can be estimated from the attenuation of the primary beam, some of the local computing power that is part and parcel of intelligent sensors could well be devoted to unraveling this complication, thus increasing the accuracy of both the size distribution measurement and the concentration measurement. However when the opacity (product of particle concentration, optical path length, and mean particle diameter) is less than 0.5 only small corrections are necessary; an opacity of at least $\cap$ 005 is generally needed to obtain adequate signal. Reasonable concentrations fall in the 10^6 to 10^9 particle-m^{-3} range for typical apparatus and particle parameters. 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 can also contribute to errors in size measurements. Use of an argon ion laser, with its green and blue spectral lines well out of the intense thermal radiation region, along with narrow band optical filters, should be a straightforward solution. Finally, several thermally related problems, e.g., beam steering by thermally induced index of refraction gradients, are common to all the optical the optical techniques we discuss.

Particle Counting, Sizing and Velocity Measurement

The single particle counting technique PCSV relies on a robust theory for scattering in the near-forward direction. Because there is no inherent spatial averaging, if this technique is to be used for real-time monitoring in industry, the spatial sampling point must be carefully chosen to be in some sense "typical." The illumination source is a focused laser beam whose (1/e)-diameter is 20-30 μm. The geometry and optical system are designed to provide a monotonically increasing signal amplitude with particle size, with minimum sensitivity to complex refractive index and shape of

particies. This is accomplished by recognizing and exploiting the fact that the small angle diffraction component of scattered light depends almost exclusively on the projected surface area of tile scatterer. The scattering angle limit imposed by the apparatus geometry is the order of $1°$.

The amplitude of scattered light is a function not only of particle size but also of particle trajectory through the measurement volume: particles passing through the laser beam scatter light with an intensity *vs* time profile that maps the laser beam profile. Since lasers are typically designed to have strictly TEM_{00} Gaussian profiles, it follows, as a special property of the Gaussian function, that along any trajectory, whether or not it passes through the laser beam axis, the intensity *vs* time profile is also Gaussian. It is thus not possible to discriminate a big particle grazing the measurement volume from a small particle passing through its center. To overcome the ambiguity, an inversion technique assuming an equal probability of passing through any element of the cross section of the sample volume, and equal mean velocity, is suggested by Holve [16, 17]. However finding the parameters needed to effect the inversion requires calibration with a monodisperse particle distribution of known diameter and concentration, passing through the measurement volume at known mean velocity. We might speculate that an alternate intelligent sensing approach might make use of a non-TEM_{00} laser; in this scenario the time dependence of the scattering from each particle could contain sufficient information to specify the relevant parameters of its trajectory, in which case the scattering intensity could provide particle-by-particle sizing.

In another alternative, if the optical properties and shapes of the particles are known and invariant, backward scattering geometries are possible [26]. Backward scattering might be preferred in industrial settings, since it requires access to only one side of the tower, all optical components are fixed to a single platform, and small changes in pointing direction are uncritical because both the incident and scattered light pass through the same paths and experience the same change of the average refractive index. However the disadvantages of backward mode include lower signal intensity and greater sensitivity to particle shape and optical properties. If these are not well understood, the forward scattering configuration is to be preferred despite its lesser convenience. This contrast is another opportunity for intelligent sensing methods: the more convenient method is the weaker in the absence of contextual knowledge, and it is contextual knowledge that the methods of artificial intelligence methods are designed to access and utilize.

Phase/Doppler Particle Analysis

The *P/DPA* methods are single particle counting techniques that give velocity distribution, particle concentration, and particle size in a small measurement volume, effectively a point on the size scale of atomization processes. Doppler measurements are primarily velocity sensitive; however since it is the velocities of individual particles that are detected and measured, the particle count rate also serves to measure particle concentration. Secondary features of the signal provide at least some measure of particle size. The apparatus consists of light source, light detector, and signal processor. The source is usually a continuous gas laser, *e.g.*, helium-neon or argon ion. A beam splitter separates the highly collimated light beam into two slightly displaced parallel beams of equal power. The two beams are focused to the same point by a good quality lens. The region of intersection is the measurement (or sample) volume. In this volume interference between the two beams results in spatial modulation of the light intensity that can be imagined as a grating of light and dark stripes on a microscopic distance scale. A particle passing through this region scatters light whose temporal intensity pattern corresponds to the grating's spatial intensity pattern, the conversion factor being the particle velocity. The receiver includes a collection lens (usually shared with the transmitter), an aperture, and one or more photomultiplier tubes and signal preamplifiers. The signal processor is mainly of an input conditioner and a timer. While signal amplitude provides a crude measure of particle size, the phase difference between the signals received at two detectors does a better job.

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Three detectors are required to ensure an unambiguous measurement when phase differences over 360^0 are possible. To calibrate doppler measurement systems gas velocity and turbulence must be inferred from seed particle measurements. A conventional LDV cannot discriminate signals from small and large particles that have different scattering properties.

In more detail, with an alternative (and in some ways more rigorous) description of the signal generation mechanism, the intersection of the two coherent beams is an ellipsoid whose size depends on the laser power, the illuminating beam focusing, the scattering properties of the particles, and the photodetector sensitivity. When a particle passes through the measurement volume, it is simultaneously illuminated by both beams, and it coherently scatters light from both beams. Because the beams point in different directions, the two doppler shifted scattering signals are different; with this interpretation the ultimate signal is due to the heterodyning of the two scattering signals, in which each beam serves as the reference beam for the other. 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 each photodetector, and the doppler shift signals are detected via their square-law behaviors, i.e., their intensity rather than amplitude sensitivities. The doppler signal frequency is a function of particle velocity, laser wavelength, and intersection angle:

$$f_d = 2u_x \sin \frac{\beta}{\lambda} \tag{2}$$

where f_d is the doppler signal frequency, u_x is the velocity component of the viewed particle in the direction in the plane of and perpendicular to the bisector of the two laser beams, β is the intersection angle of the beams, and λ is the optical wavelength. As long as the scattered light is dominated by reflection the phase difference between the signal received at multiple detectors depends only on the ratio of particle diameter to spatial fringe spacing in the measurement volume, and explicitly is thus independent of the index refraction of the particles. For metal particles meeting this requirement is practically guaranteed.

Since the doppler technique is based on frequency and time measurements rather than on intensity measurements it has high immunity to noise, signal attenuation, and environmental interference. It also offers good spatial resolution, and is capable of following rapid velocity fluctuations. The linear relationship between measured phase and powder size creates a uniform size sensitivity. The backward mode, relying on reflected light, is ideal for opaque metallic 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 the velocity measurement per se, but in practice other techniques and instruments are needed to set up instrument parameters properly, and to calibrate the size measurements.

One approach to particle size measurement with LDV apparatus is to relate the particle size to the visibility or contrast of the signal, defined by the envelope of the reflectance signal seen at the detector as the particle passes through the measurement volume. The visibility in forward or backward scattering is given by Adrian [1]

$$V = \frac{|J_1(\frac{\pi D}{d_f})|}{\frac{\pi D}{d_f}} = \frac{I_{max} - I_{min}}{I_{max} + I_{min}} \tag{3}$$

where d_f is the spatial fringe spacing, I_{max} is the signal envelope maximum, and I_{min} is the envelope minimum at the instant the maximum is attained. Some work has been done to measure particle size by using this relationship between visibility and particle size but the method has significant limitations. First, the size range that can be measured is limited. When the visibility exceeds 0.95 the differential

response is small, and the size resolution for small particles is correspondingly poor. At the other extreme, measurement ambiguities arise at the low end of the visibility scale. Second, since the visibility is obtained via intensity measurement it is sensitive to the individual beam intensities. When either beam is momentarily attenuated, *e.g.*, by a large particle passing through it even far away from the measurement volume, it contributes substantially to system noise.

Imaging Techniques

Atomization processes are currently controlled by operators who rely primarily on their visual observations of the process. The number of expert operators is regrettably small. Machine vision techniques including image acquisition, image processing, image understanding, and image based feedback control, probably mediated by expert systems, thus suggest themselves as a supplement to the experienced operator base. Both visible and infrared cameras are possible, and methods combining both would probably be preferred. The hottest regimes would be observed via their visible luminosity. Infrared radiation, whose emitted power increases as the fourth power of the temperature, is particularly attractive for observing thermal regimes not hot enough to be visibly luminous, but still hot enough to be luminous in the infrared. The coolest stages would be observed primarily in reflection, visible or infrared or both. Externally supplied illumination, possibly from intense pulsed lasers, might be useful for freezing particle motions, or even for following individual particles, although the latter would be an admittedly difficult task.

By sampling and analyzing the metal powders from the process, and then comparing stored images with the analyzed data, the inferred relationship between the characteristics of the cone (shape, color and angle) and the final properties of the powder could be found for specific operating conditions. These quantitative connections could be augmented by qualitative or heuristic connections obtained by observing and interviewing experienced operators and successful process designers. The combined data and rules would serve as the knowledge base portion of an expert system. This method has been shown in other contexts to be suitable for processes that are not understood exactly or are difficult to model from first principles. The knowledge base of the expert system would be enlarged and modified by ongoing accumulation of data and experience that would enhance understanding and improve control of the process. Since the most pressing need for understanding the nature and control of the atomization process is the study and organization of the relationships between the cone properties and parameters of the final products, this approach is ideal in that it immediately aids the production operation while at the same time making each production station a research laboratory generating new data.

This approach sounds so natural, straightforward, and, since it uses only existing, commercially reliable apparatus such as television cameras, lasers, and computers, so inexpensive, that it seems that it ought to be pursued even to the exclusion of other instrumentation modalities. The catch is that existing image understanding methods are extremely primitive compared to the visual scene understanding abilities of even the most inexperienced and naive human observers, to say nothing of experienced operators. The state-of-the-art may be illustrated by an elaborate camera, laser rangefinder, and high speed parallel processing computer driven vehicle under development at CMU: at a slow walking pace it will usually stay on the road, increasingly infrequently trying to climb a tree whose converging lines it mistakes for the path. This is impressive progress, and reason for real confidence in the future viability of the methodology we have outlined; but implementation will require a substantial research effort directed at image understanding in the atomization context.

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Electrical Methods

The disadvantages of sensors using optical principles include complex alignment of optical system, high cost and potential of contamination of lenses, all of which limit their application in industry. While these sensors could give parameters of metal powders with high accuracy, there is room for sensors that simply monitor the process and provide an indication of changes in specific parameters such as concentration and average diameters. Here we describe two non-optical approaches, surface ionization and electrostatic noise, which we collectively label "electric principles of monitoring particles" or EPMP.

When an atom or molecule whose ionization potential is comparable with the work function of a metal comes into contact with heated metal surface, the atom or molecule may leave the surface as a positive ion. In the SIMP (surface ionization for monitoring particles) instrument, ions produced by the impact of particles on a heated filament are collected by an ion collection electrode. The burst of ions accompanying impact of a particle containing surface ionizable impurities produce a sizable electrical pulse. Sodium and potassium, which are the sixth and seventh most abundant elements on earth, are easy to surface-ionize. Besides sodium and potassium 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, ohmically heated in some feedback loop that approximates constant temperature operation. An electrical potential difference between the filament and an adjacent ion collection electrode collects the ion burst. The powders produced by atomization usually, contain enough impurities with low ionization potentials that we would expect them to be sensitively detected.

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 (0.01 μm in diameter is detectable) and simplicity. This detector has selective behavior [10]: while sensitively detecting particles containing alkali and other surface ionizable constituents, it responds only 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.

Another technique is called the 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 spray, 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. However the fluctuations in the signal current, i.e., the noise, does depend on particle size via the shot noise effect: many small particles vs few large particles may carry the same current, but the noise on the latter signal is greater. A sample device is used to draw samples from the spray. By using a cyclone separator the sample stream is divided into two streams: overflow with fine powders and underflow with coarse powders. The relative mass flowrates of powders in the two stream are determined by the size distribution of the powder. The size measurement 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 spray with the gas stream, otherwise taken away for on-line or off-line analyzing if necessary. Similarly the height analyzer used in the SIMP method could be available to analyze the electric noise signals.

Isokinetic Sampling

It is difficult or impossible to measure some powder characteristics on line, e.g., shape, microstructure, and chemical composition. Furthermore there is a need to check the measurements

obtained using the on-line sensors described above. One way is directly to withdraw particles from the cone at a desired point, and to characterize the sample off line. The key point of this sampling approach is isokinetic sampling. This means the velocity in the sample probe should be equal to the velocity at the measuring point, which assures a representative sample. Were the sampling velocity greater than the isokinetic rate, the sampled fraction of fine particles would be anomalously high, whereas were the sampling velocity low the fraction of coarse particles would be anomalously high. Of course obtaining this condition implies at least a relative velocity measurement, and since this measurement is an integral part of the sampling operation the method is in fact partly on-line and only partly off-line.

A suitable sampling probe consists of two adjacent tubes parallel to each other and parallel to the direction of stream motion. One is just a short length of tubing through which the stream flows freely, the other leads, generally through a 90° bend, to the sampling apparatus, e.g., filters, cyclones, etc. Each tube contains, on axis not far from the entrance end, a small flow sensor; the sampling rate is adjusted to maintain identical flows through the open tube and into the sample collector, i.e., isokinetic flow. A typical flow balancing arrangement might use thermistors maintained somewhat above ambient temperature by ohmic heating. The resistance of a thermistor is sensitive to temperature; it can be maintained at constant temperature by adjusting the heating voltage so as to keep the resistance constant in a changing environment. This can easily be accomplished by a self-regulating electrical circuit. The required heating voltage is a function of the rate at which heat is removed from the thermistor, which in turn depends on environmental temperature and flow rate. Since the reference and sampling tubes have to be at essentially the same temperature, when the two thermistor resistances are balanced the flow rates must be balanced. Calibration of the flow measurements gives the stream velocity. Powder samples are removed periodically and analyzed by any appropriate set of techniques.

Prospects and Needs

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 process has not yet been full described by a mathematical model, a combination of the best available models and a heuristic understanding of atomization can be used to develop intelligent sensors and 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 with respect to advantages, limitations and capabilities. Every sensor is based on a different principle and each has its inherent limitations: no one is considered to be "the best." The comparison between methods based on different principles is very difficult: measurement volumes vary, the sampling periods are not the same, etc. Single particle counting, sizing and velocity measurement seems to be suitable for research to study the atomization process. With this approach, particle size distribution, velocity and concentration are given in absolute units simultaneously. The electrical methods may be worth developing as industrial monitoring techniques because of their relative simplicity and ruggedness, and the sanguine prospects for integrating them into intelligent materials processing systems. However, any instrument will require of calibration to standardize signals and to relate them output to relevant processing parameters and conditions.

Bibliography

1. Adrian, Ronald J., and Orloff, Kenneth L. "Laser Anemometer Signals: Visibility Characteristics and Application to Particle Sizing". *Applied Optics 16*, 3 (March 1977), 677ff.

2. Anderson, I. private communication, 1986. unpublished. Author is with Ames Laboratory.

3. Bussiere F.G. "On-line Measurement of the Microstructure and Mechanical Properties of Steel". *Materials Evaluation 44* (April 1986), 560ff.

4. Chigier, Norman. Comparative Measurements Using Different Particle Size Instruments. Liquid Particle Size Measurement Techniques, ASTM, 1984, pp. 169ff

5. Dodge, L.G. "Change of Calibration of Diffraction-Based Particle Sizer in Dense Sprays". *Optical Engineering 23*, 5 (September/October 1984), 626ff.

6. Dodge, L. G., and Cerwin, S. A. Extending the Applicability of Diffraction-Based Drop Sizing Instrument. Liquid Particle Size Measurement Techniques, ASTM, 1984, pp. 72ff.

7. Duszczyk, J., and Jongenburger, P. "Properties of Particles Produced by Different Rapid Solidification Techniques". *Powder Metallurgy 29*, 1 (1986), 20ff.

8. Felton, P.G. Hamidi, A.A., and Aigal, A.K. Measurement of Drop Size in Dense Sprays by Laser Diffraction. Proceedings of the Third International Conference on Liquid Atomization and Spray System, ICLASS-85, Institute of Energy, Imperial College, London, England, July, 1985.

9. Fischemeister H.F., Ozershia, A.D.,and Olssonf, L. "Solidification Structure of Gas Atomized High Speed Steel Powder". *Powder Metallurgy 25*, 1 (1982).

10. Fite W.L., Myers, R.L., and Siegel, M.W. Particle Monitoring Using Positive and Negative Surface Ionization. Proceedings of the Pittsburgh Conference, 1976.

11. Grant Nicholas J. "Rapid Solidification of Metallic Particulates". *Journal of Metals 35*, 1 (January 1983), 20ff.

12. Hirleman, Dan E. Particle Sizing by Optical, Nonimaging Techniques. Liquid Particle Size Measurement Techniques, ASTM, 1984, pp. 35ff.

13. Hirleman E.D., and Dodge, L.G. Performance Comparison of Malvern Instruments Laser Diffraction Drop Size Analyzers. Proceedings of the Third International Conference on Liquid Atomization and Spray System, ICLASS-85, Institute of Energy, Imperial College, London. England. July, 1985.

14. Holve, D.J. "Optical Particle Counting Sizing and Velocimeter Using Intensity Deconvolution". *Optical Engineering 23*, 5 (September/October 1984), 591-603.

15. Holve, Donald J. "In Situ Optical Particle Sizing Technique". *J. Energy 4*, 4 (1988). 80-0020R.

16. Holve, Donald J., and Davis, G. Wesley. "Sample Volume and Alignment Analysis for an Optical Counter Sizer and Other Applications". *Applied Optics 24*, 27 (April 1985), 998ff.

17. Holve, Donald J., Tichenor, Daniel, Wang, James C.F., and Herdesty, Donald R. "Design Criteria and Recent Developments of Optical Single Particle Counters for Fossil Fuel System". *Optical Engineering 20*, 4 (June/August 1981), 529ff.

18. Lawley, Alan. "Modern Powder Metallurgy Science and Technology". *Journal of Metals 38*, 8 (Aug. 1986), 15ff.

19. Lubanska, H. "Correction of Spray Ring Data for Gas Atomization of Liquid Metals". *Journal of Metals 22*, 2 (February 1970), 45-9.

20. Mehrabian, R., and Wadley, Hadyn N.G. "Needs for Process Control in Advanced Processing of Materials". *Journal of Metals 37*, 2 (February 1985), 51ff.

21. Meyer, Patricia. *An Analysis of the Atomization Process in Coal-Water Slurry Sprays.* Ph.D. Th., Carnegie Mellon University, 1986.

22. Myers, Richard Lee. *Electrical Detection of Airborne Particulates Using Surface Ionization Techniques.* Ph.D. Th., University of Pittsburgh, 1973.

23. Nichiporenko, O.S., and Naida, Yu. "Heat Exchange Between Metal Particle and Gas in the Atomization Process". *Sov. Powder Metall. Met. Ceram. 7,* 67 (1968), 509-12.

24. Oberdier, L.M. An Instrumentation System to Automate the the Analysis of Fuel-Spray Images Using Computer Vision. Liquid Particle Size Measurement Techniques, ASTM, 1984, pp. 123ff.

25. Rai, G., Lavernia, E., and Grant, N. J. "Powder Size Distribution in Ultrasonic Gas Atomization". *Journal of Metals 37,* 8 (August 1985), 22ff.

26. Ricks, R.A., Adkins, N.J.E., and Clyne, T.W. "Production of Ultrafine Aluminum Powder by High Pressure Gas Atomization". *Powder Metallurgy 29,* 1 (1986), 27ff.

27. Savage, S.J., and Froes, F.H. "Production of Rapidly Solidified Metals and Alloys". *Journal of Metals 36,* 4 (April 1984), 20ff.

28. Schegk, Shafer M., Umhauer, C. Location Measurement of Particle Concentration, Size, and Velocity in the Sheet of a Flat Spray Nozzle with Double Pulse Holograph. Proceedings of the Third International Conference on Liquid Atomization and Spray System, ICLASS-85, Institute of Energy, Imperial College, London, England, July, 1985.

29. Seaton, C., Henein, H., and Glatz, M. "The Atomization of Molten Metals Using the Coanda Effect". *Powder Metallurgy 1* (March 1987), 37-47.

30. See, J. Bruce, and Johnston, Gwynne. "Interactions between Nitrogen Jets and Tin Stream". *Powder Technology 21* (1978), 119-33.

31. See, J. Bruce, Runkle, Joseph C., and King, Thomas B. "The Disintegration of Liquid Lead Streams by Nitrogen Jets". *Metall. Trans. 4* (1973), 2669-73.

32. Smith, N.P., Norris, J.O.W., Hemsley, D.J., and Yeoman, M.L. Measurement of the Size and Velocity of Liquid Metal Drops During Atomization. Proceedings of the Third International Conference on Liquid Atomization and Spray System, ICLASS-85, Institute of Energy, Imperial College, London, England, July, 1985.

33. Svarovsky, L. and Hadi, R.S. A New Simple and Sensitive On-Line Particle Size Analyser for Fine Powders Suspended in Gases. In *Fine Particles Processing,* (publisher unknown), 1979, pp. 366-78.

34. Tan, G.H., and Sheppard, T. "Process-Structure Relationship of Extrusion Produced from Rapidly Solidified Al-Mg-Mn Powders". *Powder Metallurgy 29,* 2 (1986), 143ff.

35. Tewari, N., and Kumar, Mahenda. "Dendritic Microstructure in Argon Atomized Superalloy Powders". *Metallurgical Transactions A 17,* 11 (November 1986), 2099ff.

36. Wadley, Haydn N.G. "Sensors for Intelligent Processing of Materials". *Journal of Metals 38,* 10 (October 1986), 49ff.

37. Wang, James C.F. and Hencken, Kenneth R. "In Situ Particle Size Measurements Using a Two-Color Laser Scattering Technique". *Applied Optics 25,* 5 (March 1986), 653ff.

38. Yolken, H. Tomas, and Modfin, Leonard. "Automated Processing of Advanced Materials" *ASTM Standardization News 14* (October 1986), 32ff.

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