DETECTION SCHEME FOR PARTICLE SIZE AND CONCENTRATION MEASUREMENT

Field of the Invention

The present invention is related to the field of measuring particle size and concentration. More specifically it relates to the use of optical methods for measuring particle size and concentration.

Background of the Invention

Many techniques exist for particle size and concentration analysis (PSA), they can be reviewed for reference in the book by Terry Alan "Introduction to Particle Size Analysis" T. Allen, Particle size analysis John Wiley & Sons; ISBN: 0471262218; June, 1983. The most commonly used techniques are optical, based on the interaction of the measured particles with laser radiation. Especially when approaching the particle size range around 1 micron and below, most of these techniques suffer from inaccuracies due to the effect of the real and imaginary part of the particle's refractive index. It is known, for example, that in some techniques, such as techniques based on Fraunhoffer diffraction analysis, light absorbing particles would be over sized due to energy loss resulting from the absorption, while in high concentration, particles would be under sized due to secondary scattering etc.

An optical technique that is less sensitive to these problems is known as Time of Transition or TOT. In this technique the interaction of a scanning, focused laser beam and the particles is analyzed in the time domain rather than in the amplitude domain, resulting in lower sensitivity to variation in the refractive index. A detailed description of the technique appears in a paper "Improvements in Accuracy and Speed Using the Time-of-Transition

Method and Dynamic Image Analysis For Particle Sizing by Bruce Weiner, Walter Tscharnuter, and Nir Karasikov",[Particle Size Distribution III; Assessment and Characterization; Editor(s): Theodore Provder1; Volume 693, Publication Date (Print): June 10, 1998; Copyright © 1998 American Chemical Society]. To a great extent, in this technique a de-convolution algorithm of the known laser beam profile from the interaction signal derives the size. The concentration is derived from the number of interactions per unit time within the known volume of the focused laser beam.

The interaction of the particles in the TOT technique is with a focused scanning laser beam. In order to measure smaller particles, a smaller focused spot should be used. However according to diffraction laws for a Gaussian laser beam, if the beam's waist is D, the divergence of the beam is proportional to λ/D where λ is the laser's wavelength. The trade-off between the ability to resolve small particles, to the focus volume and the accuracy in measuring concentration is obvious. Thus if the TOT technique is targeted to resolve and measure particles in the micron and sub-micron range it would be limited in its ability to measure low concentrations as the instantaneous focus volume is small and the interaction rate of particles is low. On the other hand, taking a larger spot will improve the concentration measurement rate but will degrade the quality and resolution of the size analysis.

An improvement could be achieved by using a shorter wavelength. This could have a limited effect of as high as a factor of 2 only since going to too short a wavelength will result in absorption of the laser light by the optics and, in the case of particles in liquid, also absorption by the liquid.

A previous invention by the inventors (US 7,746,469) introduced a new technique and means to decouple between the two contradicting

requirements: the ability to resolve small particles and the ability to measure low concentration using measurements based on single particle interactions using a structured laser beam.

It is therefore a purpose of the present invention to provide new detection schemes offering higher sensitivity due to lower particle diameter dependency of the interaction signal.

It is another purpose of the present invention to provide new detection schemes offering the ability to measure higher particle concentration due to inherent optical noise filtration.

It is another purpose of the present invention to provide new detection schemes offering the ability to characterize particles by their interaction signal both in forward and in back scatter.

Further purposes and advantages of this invention will appear as the description proceeds.

Summary of the Invention

The present invention provides a system and method of particle size and concentration measurement that comprises the steps of: providing a focused, synthesized, structured laser beam, causing the beam to interact with the particles, measuring the interaction signal and the number of interactions per unit time of the beam with the particles, and using algorithms to map the interaction signals to the particle size and the number of interactions per unit time to the concentration.

The particles can be fluid borne, airborne, or on a surface and have a size ranging from sub-micron to thousands of microns. In a preferred embodiment of the invention, the focused, synthesized, structured laser beam is a dark beam.

The structured beam can be generated by employing a mask over a Gaussian laser beam, by directly modifying the laser cavity, by combining the beams from several lasers, or by other manipulations of the laser beam such as in an interferometric or polarization modification scheme. The measurements can be made using the duration of interaction with a scanning beam, including dark field. The invention further provides a system for particle size and concentration measurement.

An alternative approach, which has the advantage of not using any moving parts to scan the beam, is to cause the particles to cross focal region of a focused laser beam.

Other aspects of the invention relate to an improved detection scheme capable of better particle characterization according to the forward and back scatter, detect particle fluorescence and measure the particle velocity.

The present invention introduces new detection schemes offering: higher sensitivity due to lower particle diameter dependency of the interaction signal (much lower than r^4 to r^6 as with conventional scattering of sub wavelength particles); the ability to measure higher particle concentration due to inherent optical noise filtration; the ability to characterize particles by their interaction signal in forward and back scatter, for example to discriminate between bubbles and particles flowing in a liquid; the ability to measure fluorescence from particles; and the ability to measure the particle's velocity. The latter enables a scanner free system where the flow of particles is either at a known velocity or the velocity of each particle is intrinsically measured.

The invention is a particle monitoring system comprising a laser that generates a Gaussian beam; means for converting the Gaussian laser beam

into a structured dark beam; a focusing lens that focuses the dark beam onto particles moving through the illuminating dark beam; and two detectors. One of the two detectors is positioned over each intensity lobe of the dark beam.

The particle monitoring system of the invention is arranged such that the particles move through the illuminating dark beam in a direction at an angle of 90 degrees relative to the direction of the dark beam.

The signals from the two detectors are recorded in at least one of the following ways:

a) as separate signals;

b) as a differential signal of the two detector signals; and

c) as the sum of the two detector signals.

Embodiments of the particle monitoring system of the invention comprise a beam splitter and a second set of detectors oriented in a perpendicular direction to the dark line of the dark beam.

Embodiments of the particle monitoring system of the invention comprise a beam splitter and a third detector arranged to allow simultaneous measurement of back scattered radiation from the particles.

All the above and other characteristics and advantages of the invention will be further understood through the following illustrative and non-limitative description of embodiments thereof, with reference to the appended drawings. In the drawings the same numerals are sometimes used to indicate the same elements in different drawings.

Brief Description of the Drawings

— Fig. 1 schematically shows an embodiment of a particle monitoring system;

— Fig.2 shows the positioning of the detectors of the system of Fig. 1 with respect to the illuminating dark beam pattern;

— Fig. 3(a) and Fig. 3(b) show typical signals measured by the two detectors in the system of Fig. 1;

— Fig. 4 is a scattering simulation showing half of the signals detected by the two detectors of the system of Fig. 1 for air bubbles and latex particles in water;

— Fig. 5 shows simulated signals for the difference between the signals from the two detectors of Fig. 1 as particles of various size move from the center outward;

— Fig. 6 schematically shows an embodiment of the detector system of Fig. 1 that has been modified to allow also the measurement of backscattering of radiation from the particles;

— Fig. 7 shows an example of how the invention can be used for classification by clustering;

— Fig. 8 shows an example of multi-dimensional clustering using an un-supervised learning method;

— Fig. 9 schematically shows the profile of the dark beam;

— Fig. 10A shows the differential signal of the two detector signals for three illuminating beam structures in the presence of noise and the advantage of the dark beam in suppressing common noise;

— Fig. 10B shows the summation signal of the two detector signals for same three illuminating beam structures as in Fig. 10A;

— Fig. 11 is a screen shot showing two shoulders in the interaction signal, which correspond to half the spot size; and

— Fig. 12 shows the positioning of the detectors with respect to the illuminating dark beam pattern in an embodiment of the system of the invention that comprises a second set of two forward detectors and a

beam splitter oriented in a perpendicular direction to the dark line of the dark beam.

Detailed Description of Embodiments of the Invention

Fig. 1 schematically shows an embodiment of a particle monitoring system. The system shown in Fig. 1 comprises a laser (1), which generates a Gaussian beam; collimating lens (2); phase mask (3), which converts the Gaussian laser beam into a structured dark beam; a focusing lens (4), which focuses the dark beam inside a cuvette (5) through which water containing particles (6) flows in the direction of the arrow; and two detectors (7) and (8). It is noted that in the case of airborne particles, the air stream bearing the particles need not be confined within a cuvette. The positioning of the detectors with respect to the illuminating dark beam pattern is shown in Fig.2. In this embodiment one detector is positioned over each intensity lobe of the original dark beam. As particles cross the beam the output intensity pattern is modified and the detectors sense the change. The detector spacing can be optimized for sensitivity by aligning it to the maximum intensity gradient of the dark beam. For various analytic purposes the detector signals can be recorded either as:

a) separate signals;

b) as a differential signal of the two detector signals; and

c) as the sum of the two detector signals.

In another embodiment, a second set of two forward detectors is used via a beam splitter in a perpendicular direction to the dark line of the dark beam. These two detectors are large relative to the beam size and integrate the total beam intensity. Fig. 12 shows the positioning of the second set of detectors with respect to the illuminating dark beam pattern in this embodiment, By checking the symmetry of the signals from these two detectors, one can derive if a particle has crossed the focal zone along its diameter (signals are equal), or along a chord (signals are different) as well

as important information on the particle size like the width of the interaction or the depth of modulation in the signal. The timing of these two signals provides information also on the alignment of the particle flow direction and to what extent it is laminar and perpendicular to the optical axis.

Typical signals measured by the two detectors in the system of Fig. 1 are shown in Fig. 3(a) and Fig. 3(b). In these figures the signals measured by detector 1 (7) are identified by numeral (10) and those by detector 2 (8) are identified by numeral (12). For proper signal interpretation it is necessary to confirm that the particle crossed the beam along the focal plane. According to the present invention the signals of the two detectors appear simultaneously when the particle crosses the beam at focus, as shown in Fig. 3a. If the particle does not cross along the focal plane one detector signal is delayed with respect to the other one as is shown in Fig. 3b. The shift direction determines also whether the particle crosses the beam before or after the focus. It should be noted that the shape of the signal represents intrinsic particle characteristics.

Since the detector signals qualitatively represent interferometric response they react to the phase modulation by the moving particles. Thus, a particle with refractive index larger than the surrounding medium, e.g. latex in water, will induce, as an example, first a negative signal in detector 1 and a positive signal in detector 2 while a particle with refractive index smaller than the medium (bubble in water) will generate the opposite signal. It should be noted that the main feature is the opposing signals. By changing the detectors order positive/negative could be reversed. Fig. 4 shows half of the simulated signals in the forms of graphs showing detector output vs. the distance moved by the particles for air bubbles and latex particles in water. As a consequence, it is possible to distinguish bubbles from particles. In the figure, curve (14) shows the signal from sensor 1 for air in water; curve (16)

shows the signal from sensor 2 for air in water; curve (18) shows the signal from sensor 1 for latex in water; and curve (20) shows the signal from sensor 2 for latex in water.

Another important aspect of the present invention is a detection scheme with a lower signal intensity dependence on the particle radius r. According to the classical scattering theory, the energy scattered behaves according to r^4 or even r^6 while in the present invention the signal is a result of a phase shift and the r dependence is between r^2 to r^3. Fig. 5 is an example of a simulation showing the dependence of the difference signal, i.e. the difference between the signals of the two detectors for three particles of different size (r=50nm - dotted line; r=100nm - solid line; r=200nm - dashed line) moving out from the center of the dark beam pattern shown in Fig. 2. The advantage over the prior art provided by the invention is of a lower required detector dynamic range and a simpler detection scheme. The challenge of having a detector with a dynamic range of 1:10,000 to 1:1,000,000, as needed according to Rayleigh to detect particles in the range 0.1 to 1 micron, is clear to anyone familiar with the art.

For particles typically larger than the spot size, the intensity on the two detectors will reach a plateau and the measuring parameter will be the detectors summation width, which is proportional to the particle size.

Fig. 6 schematically shows an embodiment of the detector system of Fig. 1 that has been modified to allow measurement of backscattering of radiation from the particles. The setup is as explained herein above for Fig. 1 with the addition of a beam splitter (24), collecting lens (26), pinhole (28), and the back scatter detector (30). The back scatter radiation from a particle (6) in the focus of the focusing lens (4) is collected by the focusing lens (4), collimated, reflected by beam splitter (24), and directed via the collecting lens (26), which focuses the radiation through pinhole (28) onto the back

scatter detector (30). In addition another lens (32) has been added, as may be needed by the type of laser output, between the laser (1) and collimating lens (2) such that lens (32) and (2) act together as a beam expander (34).

In the present invention the back scatter detector has four roles:

- The obvious use of back scatter detector (30) is in a confocal detection scheme to verify that the interaction with the particle was indeed in focus.
- To provide additional size information, where, for particles smaller than the dark spot, the dark beam modulation is inversely proportional to the particle size. On the other hand, for particles larger than the dark spot and moving with constant velocity, the interaction duration is proportional to the particle size.
- The back scattering interaction adds another dimension for differentiation among particle groups based on the fine details of the interaction fingerprint, which could include reflection properties.
- The back scatter detector can detect fluorescence generated by the illuminating beam. In this application the beam splitter (24) is replaced by a dichroic mirror that would reflect the fluorescent light to the BS detector. The ability to measure the fluorescent light in parallel to the detection with the forward detectors, adds a powerful classification tool in cases in which the particle population was stained with a fluorescent stain. This is extremely applicable to Algae to help characterize the algae type or to the detection of pathogenic organisms.

A combination of a beam splitter and a dichroic mirror would allow to detect by two back scatter detectors the back scattered light and the fluorescent light.

The two forward signals and the optional back scatter signals (with and without Fluorescence) are single particle interactions with a high resolution laser focused beam. These interactions function as a high resolution one dimensional scanning laser microscope and provide a lot of information on the particle infrastructure. This information could be used to characterize specific particles. Particles of the same size but different internal structure will have the same interaction width but the internal interaction pulse behavior will differ and be like a "fingerprint" of the particle. An example of how the invention can be used for classification by clustering is shown in Fig. 7 for algae.

Fig. 7 shows the data in a two dimensional feature space. A set of interactions were acquired for 3 different types of Algae: Chlorela, Tetrahedron, and Pediastrum (indicated in the figure respectively by x, +, and *). Validation filters on the interaction signals, including symmetry of the two channels; interaction rise time, and others were applied. The Validation filters assure that the interaction is in the focal zone of the dark beam. Tests were conducted with various sub sets of filters but Fig. 7 presents the data when all validation filters were applied. Features are extracted from the interactions that qualified the validation filters and show the clustering of the different types of algae.

Although the feature space is multi-dimensional, Fig. 7 presents a 2D scatter diagram wherein the X axis is the interaction pulse width in microseconds and the Y axis the maximal signal per interaction as detected by the detectors. Already in this 2D presentation there is a clear grouping of the different algae types. To help manifest this grouping ellipses were outlined in the figure to indicate the boundaries between the groups. In this 2D presentation there is still some overlap, which could be reduced in a multi-dimensional feature space. Artificial Intelligence clustering

techniques are then used, in the multi dimensional space, to identify the boundaries.

The application of this mechanism is such that once the clusters of known algae are established, it is possible to monitor, for example, water contaminated with algae and detect in a mixture whether algae complying with the clusters appear. This would give real time information on the algae population and feedback to any process attempting to reduce the algae population.

While the capability of the invention is demonstrated herein on algae, it can be used with all its detection options described above, to train the system on other events, such as pathogenic organisms and upon appearance of an event complying with the cluster of the said organism an alarm signal will be triggered. The analogy is drawn to a fingerprint, where objects of the same group will have commonality in the feature space and could be identified via this commonality in the same way a person is identified by his fingerprint.

The classification by clustering approach can be extended to a multi-dimensional space using artificial intelligence tools to teach the system the nature of specific events and then to monitor for the presence of such events. One embodiment of the invention is to cluster the detectors signals by un-supervised learning (Visit http://www.autonlab.org/tutorials/ for Andrew's repository of Data Mining tutorials.). Fig. 8 shows an example of multi-dimensional clustering using an un-supervised learning method.

The measuring systems of Fig. 1 and Fig. 6 can be used to measure the intrinsic velocity of the particles moving through the cuvette. This is possible because the interaction signal duration is scaled inversely to the particles velocity. While in some configurations a constant and known

velocity could be achieved a more general approach of the present invention is to extract the velocity information from the intrinsic information in the interaction signal. This is done by accounting for the profile (shown schematically in Fig. 9) of the dark beam. The distance between the intensity peaks of the lobes of the beam profile is equal to WO*2^0.5, where W0 is the Gaussian waist. This value is known and hence can be used to measure the crossing particle velocity, while the modulation depth is used to extract the size information. This is applicable for small particles where the "lens effect" of the particle is negligible. For larger particles there will be 2 shoulders in the interaction signal which correspond to half the spot size, as shown in Fig. 11.

Many particle monitoring applications are characterized by a large population of very small particles, doped with slightly larger particles. Examples could be colloids; CMP slurry; crystallization processes and more. The ratio of the tail concentration of the slightly larger particles could be 10^6:1 or so smaller compared to the main concentration. The state of the art instrumentation today is practically blind to these small concentrations. While small and challenging to measure, this small tail could cause damages and scratches in the case of CMP or other processes. The present invention offers the ability to measure concentrations which are 10^6:1 smaller in this tail. The spot size is selected such that the majority of the population is filtered out and becomes a background noise while the larger particles are shown as clear interactions.

The present invention, based on the dark beam illumination in conjunction with recording the detector signals as a differential signal of the two detectors is extremely robust to the background noise and can facilitate detection in a high level of background noise. This robustness is illustrated by the simulations presented in Fig. 10A and Fig. 10B. Fig. 10A shows the differential signal on an arbitrary scale while a 200 nm particle crosses the

beam from the center for three illuminating beam structures: Gaussian (dashed lines), Gauss-Laguerre (solid lines), and Dark beam (dotted lines). Fig. 10B is similar to Fig. 10A but for the summation of the two detector signals. The simulations were conducted under semi-dynamic noise containing 10% of the total illuminating power and the other optical parameters in the simulation were as follows:

- NA = 0.125
- λ = 400nm

In Fig. 10A a signal higher by a factor of two is achieved for the dark beam compared to the signals for the other two beam structures. The difference in sign of the curves is not material and depends on the order of the detectors in the subtraction relative to the particle direction.

Bearing in mind the larger spot and depth of field of the dark beam compared to the Gaussian beam, for achieving a valid measurement of particles smaller than spot, the noise immunity is even more apparent. The significant advantage in the noise reduction of the differential configuration (Fig. 10A) as compared to the summation configuration (Fig. 10B) is obvious.

In actual measurements a measurement set-up based on the present invention was able to detect the tail of larger particles, the concentration of which was 10^6 times smaller in concentration than the main population of the smaller particles.

Although embodiments of the invention have been described by way of illustration, it will be understood that the invention may be carried out with many variations, modifications, and adaptations, without exceeding the scope of the claims.

Claims

1. A particle monitoring system comprising a laser that generates a Gaussian beam; means for converting said Gaussian laser beam into a structured dark beam; a focusing lens that focuses said dark beam onto particles moving through the illuminating dark beam; and two detectors wherein one of said two detectors is positioned over each intensity lobe of said dark beam.

2. The particle monitoring system of claim 1, wherein the particles move through the illuminating dark beam in a direction at an angle of 90 degrees relative to the direction of said dark beam.

3. The particle monitoring system of claim 1, wherein the signals from the two detectors are recorded in at least one of the following ways:
   a) as separate signals;
   b) as a differential signal of the two detector signals; and
   c) as the sum of the two detector signals.

4. The particle monitoring system of claim 1, comprising a beam splitter and a second set of detectors oriented in a perpendicular direction to the dark line of the dark beam.

5. The particle monitoring system of claim 1, comprising a beam splitter and a third detector arranged to allow simultaneous measurement of back scattered radiation from the particles.