United States Patent [19]
Karuhn et al.

[11] **3,739,258**
[45] **June 12, 1973**

[54] **METHOD AND APPARATUS FOR DETECTING AND SIZING MICROSCOPIC PARTICLES**

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[73] Assignee: **IIT Research Institute**, Chicago, Ill.

[22] Filed: **Aug. 20, 1971**

[21] Appl. No.: **173,372**

[52] **U.S. Cl.** .... **324/71 CP**
[51] **Int. Cl.** **G01n 27/00**
[58] **Field of Search** .... 324/71 R, 71 CP; 137/576; 138/44, 103

[56] **References Cited**

UNITED STATES PATENTS

3,628,140	12/1971	Hogg et al.	324/71 CP
3,638,677	2/1972	Baccarini	324/71 CP

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Attorney—William E. Anderson, Edwin M. Luedeka James J. Hamill et al.

[57] **ABSTRACT**

Improved accuracy of size measurement and size distribution of particles in a fluid have been obtained in an electrical zone sensing apparatus by a flow control director means which directs the fluid in a more streamlined, less turbulent flow through an aperture at which the particles are electrically sensed. Also, the particles were directed and guided to flow substantially parallel to longitudinal axis for the aperture. The preferred flow control detector means comprises a contoured orifice having a smooth tapered inlet wall blended at an intersection with a central cylindrical wall defining a tubular shaped aperture.

5 Claims, 11 Drawing Figures

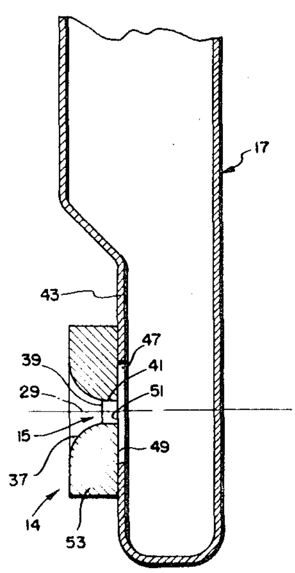


FIG.1

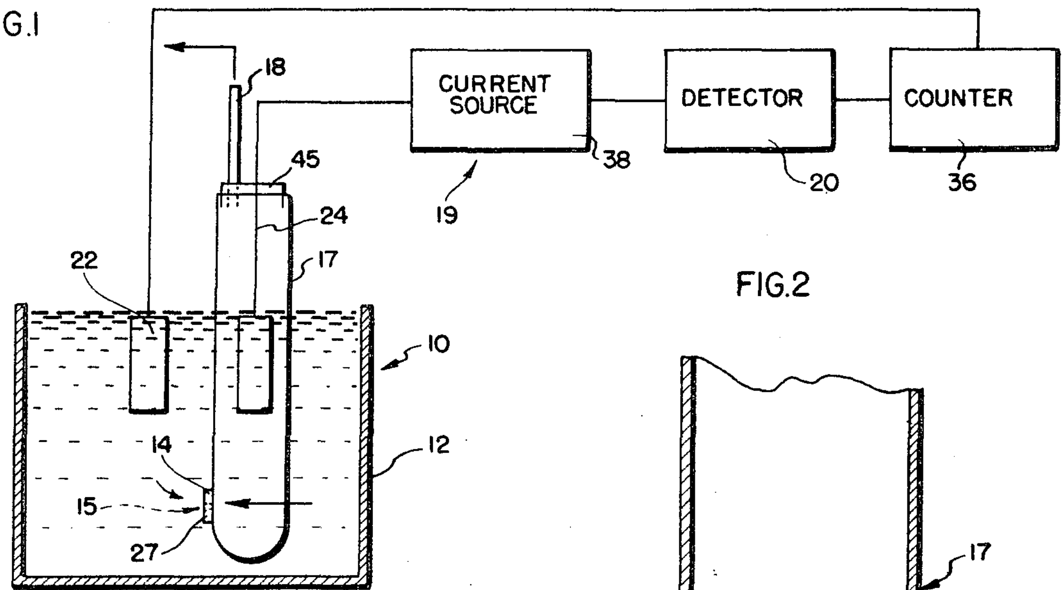


FIG.2

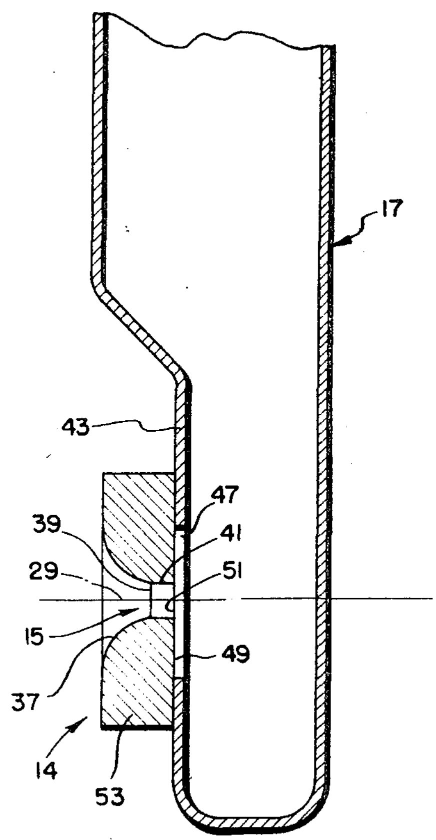
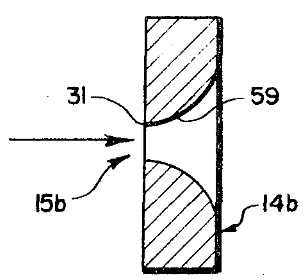


FIG.3



PRIOR ART

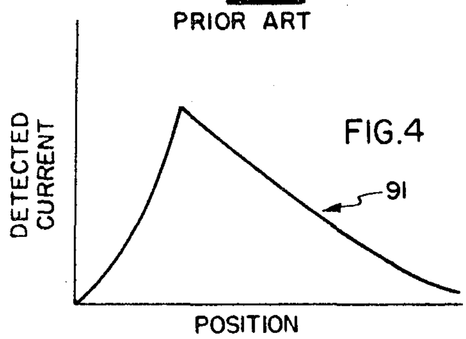


FIG.4

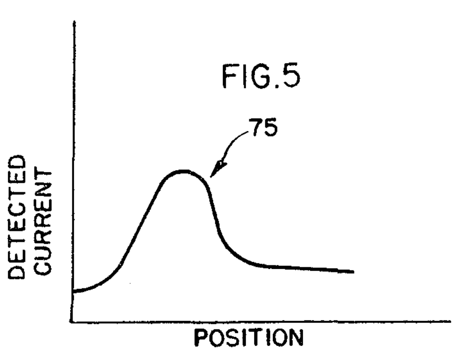


FIG.5

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FIG.6
PRIOR ART

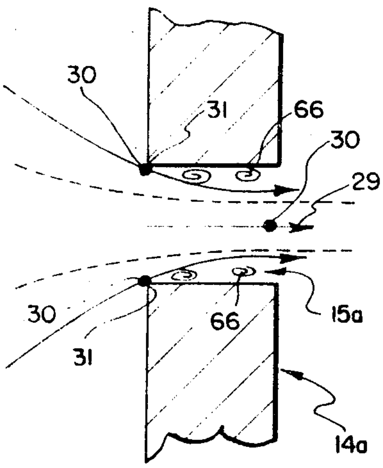


FIG.9

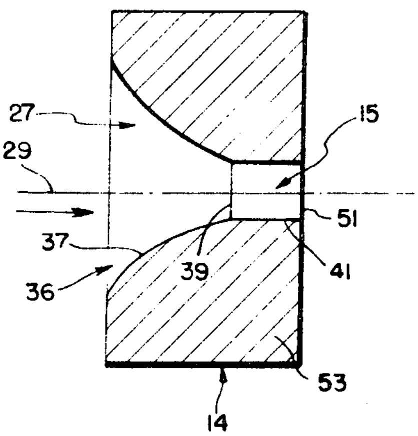


FIG.7

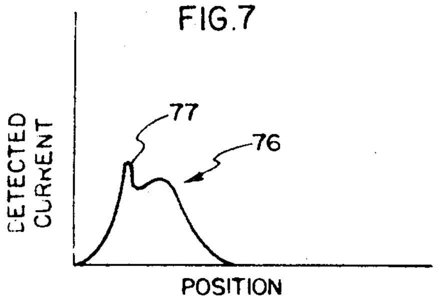


FIG.10

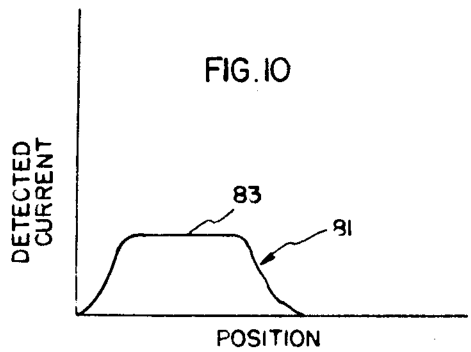


FIG.8

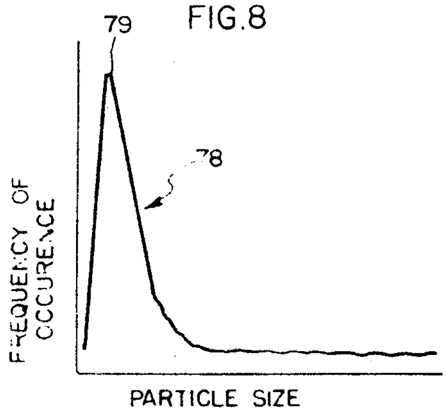
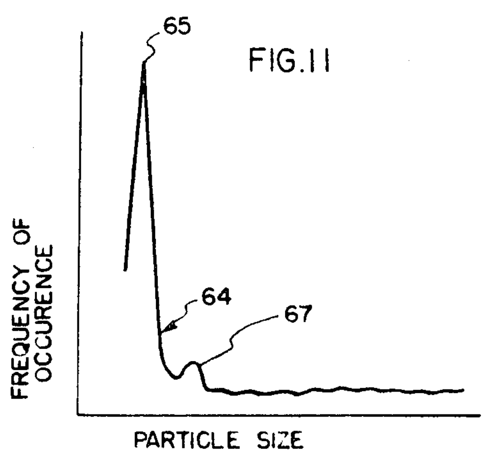


FIG.11



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METHOD AND APPARATUS FOR DETECTING AND SIZING MICROSCOPIC PARTICLES

The present invention relates to detecting and sizing microscopic particle in a liquid medium with an electrical zone sensing apparatus and more particularly to an improved method of and mechanism for signal production in a particle sensing zone of such an apparatus.

The electrical zone sensing method of particle detection and measurement is in use in laboratories throughout the world. The method is relatively fast and sensitive in detecting particles and determining their size distribution. Such a method is particularly useful in counting, measuring, grading and comparing biological and mineral particles, such as abrasives, foodstuffs, dye, blood cells, ceramics, pigments, polymer lattices, cement, pulp and paper fibers, clay, soil, powdered metals, etc.

One apparatus for electrical zone sensing particles suspended in a liquid is disclosed in Coulter U.S. Pat. No. 2,656,508. In that apparatus, a tube having an aperture is positioned within a larger vessel. Particles in liquid suspension are placed in the vessel and are induced to flow through the aperture by establishing a fluid pressure differential between the vessel and the tube. The vessel and the tube are both fabricated of an insulator, e.g. glass, and the liquid is an electrolyte. At the aperture, the presence of a particle in the liquid flow changes the electrical resistance detected from the resistance detected in the absence of a particle. If a constant electric current is placed across the aperture, the electric voltage will vary inversely with the resistance change each time a particle passes through the aperture. A detecting circuit determines the size of the passing particles from the change in resistivity caused by each particle replacing its own volume of electrolyte at the aperture and by the particular resistivity of the kind of particles being sized. This information is amplified and processed by suitable electronic circuitry.

The particle resistivity is usually several orders of magnitude greater than the resistivity of the electrolyte so that the particles may be readily sensed. However, metal powder and other apparently good conductors when in the form of microscopic particles exhibit properties more nearly like that of non-conductors than would be thought. It is hypothesized that this is due to the two oxide surface films and ionic inertia of the Helmholtz electrical double layer and associated solvent molecules at the surface of such particles. Also, electrical charges on the particles appear to have no effect on response of the electrical zone sensing apparatus. Particle density does not appear to have great effect on the response, it generally being assumed that distortion of electrical field in the aperture by the presense of a particle is related to the convex hull or "envelope volume" of the particle rather than to internal porosity in the particle. However, a great gross porosity through the particles results in a degree of electrical translucency and in proportionally less pulse height response. Thus, it is seen that the particles shape and structure may have some but generally have little effect on response and may be compensated for.

When making a size distribution with a commercially available electrical sensing zone counter of a standard batch of small microscopic spherical latex particles in an electrolyte, it was found that the size frequency distribution curve for these particles differed from that obtained with the use of an electron microscope. Also, the pulse shapes observed were significantly different from the theoretical pulse shapes expected to be observed when sensing the latex particles. More particularly, the frequency distribution curve obtained with use of the electrical sensing zone counter had a major peak which was much wider than that which should be present in the frequency curve and failed to show a secondary peak known to be present in the size distribution curve.

While it was first thought that the expected frequency curve could be obtained with improved electronic components to provide better resolution of the pulse shape and heights, such improvements did not satisfactorily produce the frequency curve. Then, it was discovered that with proper flow control of the electrolyte and particles, improved resolution and more accurate curves could be produced.

Accordingly, a general object of the present invention is to provide improved accuracy for an electrical zone counter for microscopic particles.

A principal object of the present invention is to provide an improved method and apparatus for detecting and sizing microscopic particles.

Another object of the invention is to provide an improved scanner element for use in an apparatus and method for detecting and sizing microscopic particles in a liquid suspension to provide greater accuracy and an extension of the particle size range which can be measured.

These and other objects of the invention will become apparent with reference to the following detailed description and accompanying drawings, in which:

FIG. **1** is a schematic representation of an apparatus for detecting and sizing microscopic particles;

FIG. **2** is an enlarged, fragmentary sectional elevational view of a portion of the apparatus shown in FIG. **1**;

FIG. **3** is a diagrammatic view of the scanner element of a prior art device;

FIG. **4** illustrates electrical pulse representations of the effects of particles detected with the scanner element of FIG. **3**;

FIG. **5** illustrates a normal pulse shape obtained with the scanner element of FIG. **6**;

FIG. **6** is a diagrammatic view of another scanner element of a prior art device;

FIG. **7** illustrates an abnormal pulse representation detected when using the scanner element of FIG. **6**;

FIG. **8** discloses a particle size and frequency of distribution curve obtained with the scanner element of FIG. **6**;

FIG. **9** is a diagrammatic view of a scanner element constructed in accordance with the invention; and

FIGS. **10** and **11** illustrate electrical pulse representations of the effects of particles detected with the scanner element of FIG. **9**.

As shown in the drawings for purposes of illustration, the invention is embodied in an apparatus **10** for detecting and measuring the size and the frequency of distribution of microscopic particles suspended in a fluid medium such as a liquid electrolyte. Very generally, the illustrated apparatus **10** comprises a vessel **12** such as a beaker containing the electrolyte and the suspended particles which are to be analyzed. The electrolyte containing the particles in the vessel is drawn through a small aperture **15** in a scanner element **14** into a receiver tube 17. The liquid flow through the scanner element 14 is caused by a fluid head, i.e. a pressure differential, such as by applying reduced pressure from a vacuum line 18 to the upper surface of the liquid in the tube. To measure the change in conductivity caused by a particle passing through the aperture 15 in the scanner element, there is provided an electrical circuit means 19 including a detector circuit 20 which is connected to an electrode 22 projecting into the electrolyte in the vessel and an electrode 24 projecting into the electrolyte in the tube.

It has been known that the shape and the density as well as the conductivity of the particle being tested effect the shape of the pulse electrically detected and could be compensated for. Using a standard, commercially available electrical zone sensing counter commonly used to count and size red blood cells, an electrolyte having a standard batch of spherical latex particles of microscopic size previously measured by an electron microscope was examined with the counter. It was found that the distribution of latex particles obtained with the counter varied considerably from that known to be present and that the pulse shape being provided by the counter was different from that of a theoretical pulse shape for the latex particles as they passed through the aperture. It was suspected that improvement of the electronic equipment and in particular improvement of the fidelity of the electrical amplifier would discriminate the successive voltage pulses and the respective pulse heights thereof and provide a distribution curve more closely approximating the curve which should theoretically be obtained. However, even with improved discrimination and sensitivity of the electronics employed, a skewed particle size distribution still was obtained with this counter. Thus, there was a need to improve the accuracy of such counters.

In accordance with the present invention, improved accuracy of size measurement and size distribution have been obtained by providing flow control, director means 27 upstream of the aperture 15 at which the electrical sensing is accomplished to cause a more streamlined, less turbulent flow of electrolyte through the aperture and to guide the particles toward a longitudinal axis 29, FIGS. 2 and 9 for the aperture 15, to flow along the axis through the scanner element. More specifically, it was discovered that particles, illustrated by dots 30 in FIG. 6, flowing at angle to the aperture axis 29 and past a sharp edge 31 (FIG. 6) of the aperture of prior art scanner element 14*a* were experiencing a turbulent flow thereabout. This turbulence was detected by high speed photography. The result of the turbulence was that particles flowing across the sharp edge 31 were detected as a pulse which was the combined result of detecting both the particle and the turbulence. In other instances, counters having the scanner element 14*a* appear to actually sense turbulent zones 33 and display them as tiny voltages which give the appearance of tiny particles in the electrolyte.

The illustrated flow control means 27 is in the form of a contoured orifice 36 defined by a smooth, tapered flow director inlet wall 37 blended at an intersection 39 with a cylindrical wall 41 which defines a tubular orifice 15 at which the electrical sensing is accomplished. The flow director wall 37 directs the electrolyte and particles therein along a curved uniform path toward the longitudinal axis 29 through the aperture 15. The illustrated contoured orifice 36 thus includes a smooth transition area from the large open volume of the vessel into the small tubular aperture 15; and this has been found to reduce the amount of turbulence particularly by providing a more laminar flow across the aperture 15. As explained in the copending application entitled Flow Directing Means For A Microscopic Particle Sensing Apparatus and filed of even date, other forms of flow control means may be used to provide a streamline flow and to eliminate deviations caused by detection of turbulence by the electrical sensing means.

Recapitulating, then, the method of the present invention includes the following steps: suspending particles in the electrically conductive liquid, inducing the liquid to flow and to carry the suspended particles therewith, directing the liquid and particles by the flow control means 27 to have a substantially streamlined and non-turbulent flow into the aperture 15, directing the particles to flow along a path at the aperture 15 substantially parallel to the axis 29 thereof, and electrically sensing and measuring the size characteristics of the particles passing through the aperture 15 by means of the electrical sensing means.

The scanner element 14 may be produced in a relatively economic manner by simply drilling the tubular wall to define the aperture 15 and machining the curved flow director wall 37 with known machines and tools. Thus, existing electrical sensing zone counters may be modified by replacing prior art scanner elements 14*a* and 14*b*, such as shown in FIGS. 3 or 6, with the contoured orifice scanner element 14. New original equipment counters may also be constructed with the contoured orifice scanner element 14 to provide the improved results of the invention.

Referring now in greater detail to the illustrated apparatus, the vessel 12 comprises a beaker or other receptacle of insulating material, such as glass, which is adapted to contain an electrically conductive liquid in which the particles to be detected and measured are scattered and suspended. In the examples given herein, the electrically conductive liquid or electrolyte is a sodium chloride solution in which are latex particles about 2.68 microns in size, although other suitable electrolytes can be used. The particles are scattered in the electrolyte so that groups of particles do not influence electrical sensing by the detector 20. The liquid and the particles are of different conductivity usually in magnitude of several different orders so that current or voltage change can be measured by the detector 20 as a function of change in conductivity. The detection tube 17 for receiving the fluid is also of insulating material, such as glass, and the tube rests within the vessel. In this instance, a lower side wall of the tube 17 is flattened to provide a flat wall 43, as best seen in FIG. 2, at its lower end and a stopper 45 (FIG. 1) closes the upper end of the tube. In the flattened wall 43, an opening 47 is formed and spaced from the bottom of the tube.

The scanner element 14 is mounted such as by a suitable adhesive or fusion technique to the flat wall 43 with the aperture 15 in register with a central portion of the larger diameter opening 47 in the tube. A rear side 49 of the scanner element covers the opening 47 except at an outlet 51 of the aperture 15. Thus, a path of fluid flow is defined from the vessel 12, through flow director means, aperture 15 and the tube wall opening 47 into the detection tube 17. As a difference in fluid head is established between the vessel and the tube, preferably by drawing through the pipe **18** a vacuum on the surface of the liquid in the tube, the liquid and particles are induced to flow between the vessel and tube.

In the illustrated embodiment, the scanner element **14** comprises a small block or wafer **53** of sapphire or another inert element. The wafer may be fused to the flattened wall **43** of the tube wall, the tube preferably being fabricated of a heat-resistant glass to receive the fused sapphire wafer without damage. In the wafer, the cylindrical wall **41** is formed by drilling while the tapered wall **37** is machined with known tools. The aperture **15** is typically between about 30 to 560 microns in diameter, and the aperture **15** has a length about equal to this diameter dimension. Generally, the thickness of the scanner element **14** is about 3 times the diameter of the cylindrical portion.

The flow directing wall **37** is formed without the sharp leading edge **31** at the entrance into the prior art type scanner elements shown in FIGS. **3** and **6** which edge **31** produces stream separation and causes turbulent eddies **66** within the orifice. The smooth converging wall **37** removes the sharp edges and reduces such fluid turbulence and results in a more uniform particle approach into the aperture and along a path substantially parallel to the axis through the aperture **15.** Preferably, the flow director wall **37** has a maximum diameter of about 5 times greater than the aperture diameter and it flares radially inwardly from its maximum diameter along a convex path smoothly to the intersection **39** which is at the inlet to the aperture and the aperture defining wall **41.** The illustrated contoured orifice is about twice as long as the aperture and has its axis aligned with the axis of the aperture **15.** It should be apparent that contoured orifices of other shapes and sizes might also be employed to provide the laminar, streamlined flow and to eliminate the edge effect of the edges **31** of the prior art scanner elements.

The detector **20** electrically senses, measures, and amplifies signals produced by changes in resistance in the aperture **15** due to particles passing therethrough. It is operatively connected to the electrode **22** disposed in the vessel **12** and to the electrode **24** located in the tube **17,** both electrodes being immersed in the electrolyte. The detector **20** is further operatively connected to a counter **36** which totals the number of signals. A current source operates the detector **20** and the counter **36.** A cathode ray oscilloscope (not shown) can be utilized to visually display the shape of the current or voltage pulses which have been detected. Circuitry for the detection and measurement of particle signals is well known and any suitable system can be employed.

As each particle passes through the aperture **15,** it displaces its own volume of the electrically conductive liquid within the aperture. As the liquid and particles are of different conductivity and resistance, and displacement of one by the other results in a momentary change in the detected resistance between the electrodes **22** and **24** on either side of the aperture. This resistance change produces a voltage pulse of short duration having a magnitude proportional to particle volume, if the current is kept constant. It should be noted that the method may also comprise maintaining the voltage constant while detecting a change in measured aperture current. Accordingly, voltage pulse height is proportional to amplifier gain, aperture current and the resistance change indicated upon passage of a particle through the aperture. Thus,

$\Delta E = GI\Delta R$,

where

ΔE = voltage pulse height

G = amplifier gain

I = aperture current

ΔR = change in resistance,

and

$\Delta R = (\rho o V/A2)\ (1/1-\rho o/\rho - a/A)^{-1}$

38 where

$\rho\ o$ = liquid resistivity

V = particle volume (the particle being a right cylinder having its axis aligned with and shorter than the aperture axis)

A = area of the aperture normal to its axis

ρ = particle resistivity

a = area of the particle taken normal to the aperture axis as oriented in the aperture.

It can be seen that, theoretically, response to passage of a particle through the aperture **15** is substantially linear with particle size, provided that the particle has less than about 40 percent of the diameter of the aperture. To ensure that the change in current or voltage induced by the passage of the particles through the aperture is dependent substantially on particle size, the fluid medium is chosen so that particle resistivity is effectively several orders of magnitude greater than liquid resistivity.

Referring now in greater detail to the effect of turbulence and to the pulse shapes with scanner elements **14***a* and **14***b* of the prior art and scanner element **14** of the present invention, disturbances frequently occur which are larger than the particles of the given size. These disturbances increase the standard deviation of the particle size distribution, when compared with that determined by an electron microscope. Generally, such disturbances are observed when particles do not pass through the cylindrical aperture **15** along its axis or parallel thereto and accordingly create areas of turbulence about the particles. If the particles enter the scanner element **14** at an angle to the aperture axis, interaction occurs between the sharp edge of the scanner element in which the aperture **15** is formed, the particle, and the liquid which produces turbulence about the particle. An eddy or vortex area **66** is created just inside the entrance to the cylindrical aperture and the swirling of liquid in this region produces the disturbances or noise or false signals. When the pulse produced by the turbulence is added to the pulse produced by the particle, a pulse much larger and of a different shape than the theoretical pulse shape results. The turbulence pulses might also be counted as additional small particles. On the other hand, particles entering the aperture **15***a* or **15***b* along the axis thereof produce a pulse height and shape more similar to the theoretical height and shape. Accordingly, elimination of this turbulence pulse permits more accurate reading and lowering the threshold of size detection.

More particularly, when using an apparatus similar to that disclosed in FIG. **1** but having the conventional scanner element **14***a* (FIG. **6**) the normal pulse shape detected for the latex particles was substantially in the form of pulse shape **75** illustrated in FIG. **5**; but other abnormal pulse shapes were frequently detected, the most typical of these abnormal pulse shapes **76** is illustrated in FIG. **7.** A contrast between the normal and abnormal pulse shapes of FIGS. **5** and **7** will show that an extra high peak **77** is present on the pulse shape of FIG. **7** and appears to be an addition superimposed on a more normal pulse shape such as the pulse shape **75** shown in FIG. **5.** It is theorized that the sharp peak **77** is a result of turbulence caused either by stream separations of the liquid at the sharp edge **31** of the aperture **15***a*, FIG. **6,** or by particle turbulence as a result of the particle **30** changing its flow direction to become more in the axial direction when the particle is carried past the sharp edge **31.** On the other hand, particles already flowing in a generally axial direction through the aperture **15***a* appear to produce the more normal pulse shape **75** shown in FIG. **5.**

As the abnormal pulse shapes **76** have a pulse height greater than that of the more normal pulse **75,** this effects a distribution frequency of distribution curve **78** graphed to show the results obtained with the conventional apparatus without the contoured orifice. The curve **78** (FIG. **8**) has only a single major peak **79** and lacks a distinctive secondary peak **67** such as shown in FIG. **11** for a similar curve obtained when using the scanner element **14** having the contoured orifice.

When using the contoured orifice scanner element **14** shown in FIG. **9,** the turbulence due to sharp edge effect is substantially reduced and a more consistent and a more ideal pulse shape such as the pulse shape **81,** as best seen in FIG. **10,** is obtained. This flat pulse shape **81** is better than either the pulse shapes **75** or **76** obtained with the prior scanner element **14***a*. As a result of elimination of the frequent abnormal pulse shapes **76**, a graphed frequency curve for the latex particles being analyzed had both a major peak **65** as well as a secondary peak **67.** In addition, the base of the frequency distribution curve **64** of FIG. **11** is substantially thinner than the base of the curve **78** shown in FIG. **8.** It has been found by testing the same or similar samples with other size measuring apparatus that the curve shown in FIG. **11** with the secondary peak **67** is a more accurate frequency distribution curve than the curve shown in FIG. **8.**

The better pulse shape **81** shown in FIG. **10** has a large flat top **83** and is of nearly ideal shape making it easier to measure the particle frequency and size thereby giving a higher resolution and more accurate results. In contrast to the more ideal pulse shape **81** shown in FIG. **10** obtained with the contoured orifice of the scanner element **14,** U.S. Pat. No. 2,656,508 discloses that a pointed sawtooth pulse shape **91** (FIG. **4**) is obtained when using a scanner element **14***b* having a sharp entrance edge **31** and a bore wall **59,** which flares outwardly from the entrance edge **31** in the direction of liquid and particle flow. Thus, the substantial reduction in turbulence provided by the contoured orifice produces not only a more ideal pulse shape but also a more accurate frequency distribution curve than obtained with the prior art devices.

Recapitulating, improved accuracy of the present invention with use of the scanner element **14** shown in FIG. **9**, results in the pulse shape **81** shown in FIG. **10** and the graphed frequency curve shown in FIG. **11.** By eliminating the sharp edges of the entrance to the aperture **15** and directing the flow along a converging inlet, more laminar flow is achieved through the tubular aperture **15** and a particle initially entering the scanner element along a line other than substantially parallel to the axis **29** of the aperture is guided smoothly toward the inlet to the aperture to flow in a path substantially parallel to the axis **29** without an abrupt change in direction of travel. This makes it easier to measure the particle frequency and size, gives a higher resolution and produces more accurate results.

As explained previously, the graphed curve of frequency of occurrence versus particle size in FIG. **11** illustrates that in addition to the major frequency peak **65,** the secondary peak **67** is obtained. The major peak **65** of the curve shown in FIG. **11** has a more narrow base than the base for the peak **79** of the frequency curve **78** shown in FIG. **8,** which indicates better size resolution. The distribution is also tighter in standard deviation than is the distribution achieved by the prior art devices.

Thus, the present invention provides an improved method and apparatus for detecting and sizing microscopic particles. A scanner element has been provided for use in such apparatus and method which reduces turbulent flow of the particles being detected and sized and which causes the particles to flow along paths substantially parallel to the axis of the aperture in the scanner element. This results in greater accuracy and an extension of the particle size range which can be measured.

While one embodiment of the invention has been shown and described, it should be apparent that various modifications could be made therein without departing from the scope of the invention.

Various of the features of the invention are set forth in the following claims.

What is claimed is:

1. A method of sensing and determining the size of small particles suspended in liquid by passing the same through an aperture in a wafer, comprising the steps of:

suspending the particles throughout the liquid which carries the particles through the aperture,

inducing liquid flow through an aperture to carry the suspended particles through said aperture,

directing the liquid upstream of said aperture into a substantially non-turbulent flow for passing into and through said aperture without substantial turbulence,

directing the liquid and particles to flow along paths substantially parallel to a central axis for said aperture and in said substantially non-turbulent liquid flow through said aperture,

and electrically sensing and measuring the size characteristics of the particles passing through said aperture.

2. A method in accordance with claim **1** in which directing the liquid into a non-turbulent flow comprises passing the liquid through a smooth, curved narrowing cross section transition zone prior to directing the liquid through said aperture.

3. In an apparatus for sensing and determining the size of small particles suspended in a liquid, the combination comprising: means for containing the liquid and suspended particles to be sensed and sized, receiving means for receiving the liquid and particles subsequent to the sensing and sizing thereof, a wafer including a wall defining an aperture interconnecting and in fluid communication with said containing means and said receiving means, means for inducing liquid flow through said aperture to carry the suspended particles through said aperture, a flow directing wall on said wafer upstream of said aperture having a converging wall smoothly blended with said wall defining said aperture for directing liquid and particles upstream of said aperture into a substantially non-turbulent flow through said aperture, said wafer having an outer upstream face wall and having a curved surface joining upstream face wall within said flowing directing wall without a sharp edge juncture to reduce turbulence in fluid flowing past said face wall to said flow directing wall, said flow directing wall directing the particles to flow along paths substantially parallel with the central axis of said aperture and in said substantially non-turbulent liquid flow through said aperture, and means for electrically sensing and measuring the size characteristics of the particles passing through said aperture.

4. An apparatus in accordance with claim **3** in which said curved wall is about 2 times the axial length of said aperture.

5. An apparatus in accordance with claim **3** in which said curved wall has a maximum diameter about 5 times the diameter of said aperture.

* * * * *

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,739,258 Dated June 12, 1973

Inventor(s) Richard F. Karuhn, Reg Davies, John Michael Clinch

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 1, line 5 "particle" should be -- particles --.

Claim 4, column 10, line 5, "curved" should be -- flow directing --.

Claim 5, column 10, line 8, "curved" should be -- flow directing --.

Signed and sealed this 5th day of March 1974.

(SEAL)
Attest:

EDWARD M.FLETCHER,JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents