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3,361,965 ELECTRONIC PARTICLE STUDY APPARATUS WITH IMPROVED APERTURE TUBE Wallace H. Coulter and Joseph R. Coulter, Jr., Miami Springs, Fla., assignors to Coulter Electronics, Inc., Hialeah, Fla., a corporation of Illinois Filed Mar. 26, 1964, Ser. No. 354,980 15 Claims. (Cl. 324—71)

ABSTRACT OF THE DISCLOSURE

An improved aperture tube for use with an electrical particle study device known commercially as the Coulter electronic counting device wherein said improved aperture tube is formed as an elongate glass vessel having a bottom end carrying a minute aperture which defines a passage into the aperture tube, said aperture tube being conventional except for the provision of a metallic coating bonded to the exterior surface of the aperture tube in the 20 vicinity of the aperture but spaced from the aperture to define an uncoated area immediately adjacent to said aperture, the coating having an extension thereof arranged upwardly along the surface of the tube and terminating spaced from the liquid suspension of particles when the aperture tube is immersed in said suspension, means being provided to establish an electrical connection to said extension of the coating, the tube conventionally having means for connecting the upper end thereof to a manometric system for the purpose of applying a suction interior of the tube for drawing the suspension through the aperture when the lower end of the tube is immersed in a sample suspension, the coating functioning as the exterior electrode in the electronic particle study apparatus; the coating optionally defining a second uncoated area diametrically opposite the aperture to permit illumination of the aperture and visual monitoring thereof.

This invention relates generally to an aperture tube for Coulter electronic particle apparatus but more particularly is concerned with the construction of such a tube combined with a sensing electrode for establishing a current through or voltage across said aperture and enabling the detection of passage of particles through said aperture.

The electronic particle study apparatus with which the invention is concerned has come to be known as the Coulter electronic particle device, the commercial embodiments being familiar to those skilled in the particle field as the apparatus sold under the registered trademark "Coulter Counter" by Coulter Electronics, Inc. of Hialeah, Florida. A great variety of particles may be counted and sized with this apparatus including blood cells, other biological particles, fluids, fibers, etc. The said electronic particle study device uses a principle of operation which has become universally known as the Coulter principle and was set forth in Coulter U.S. Patent No. 2,656,508. The fluid metering system and scanning apparatus which has become familiarly associated with the commercial Coulter apparatus, is described in U.S. Patents No. 2,869,078 and 2,985,830. The Coulter principle of operation asserts that where a particle of microscopic material is suspended in fluid whose electrical impedance is different from the electrical impedance of the particulate matter, the presence or absence of the particle in a small volume of the fluid may be detected due to the effect of the particle upon the total impedance of the confined volume. In the commercial electronic particle apparatus, the confined volume described is the volume of fluid in a minute aperture whose dimension and contour are known precisely, in the manner taught in U.S. Patent 2,985,830. The Coulter

principle recognizes that the electrical change is a direct function of the volume of the particle passing through the aperture, and the duration of the change is a function of the time that the particle has been in the aperture.

Accordingly, an important part of the Coulter electronic particle counting apparatus is an elongate tube which is connected into a syphon system and hence is usually filled with a first body of liquid, say a saline solution. An electrode is carried suspended in the interior of the tube, and the tube is disposed in a second vessel, intended to hold the sample particle suspension. Ordinarily the second vessel is a beaker, and another electrode is suspended therein. The two electrodes are connected electrically to a detecting device. The lower end of the tube carries the very small aperture located in its side wall and spaced above the bottom of the tube. The lower end of the tube, of course, is immersed in a sample suspension of particles carried by the said second vessel. As the suspension from the second vessel is passed through the aperture into the tube, the electrical change caused in the liquid confined in the aperture is converted into an electrical signal which can be monitored by the detecting device. The amount of suspension drawn through the aperture is very carefully metered, as for example in accordance with the teachings of Patent 2,869,078.

The tube or vessel carrying the minute aperture has become known in the trade as a Coulter aperture tube and also is often referred to as the scanning element of an electronic particle study apparatus. Conventionally and commercially the aperture tube is usually of a construction generally shown in Patent 2,985,830. The aperture is normally formed by drilling a hole in a thin wafer of glass or preferably sapphire and cementing or fusing the wafer to the side of the aperture tube over an orifice

previously formed in the tube.

The primary concern of this invention is with the construction of such an aperture tube.

In the past, the electrodes referred to above are made of non-corrosive metal and are suspended in a fluid of both vessels, the beaker and the aperture tube, and external leads pass from these respective electrodes to the input of the detector device, ordinarily, the counter circuitry. Generally these electrodes have been platinum foil members soldered or welded to electrical leads and dipped into the fluid bodies of the respective vessels. While there was little difficulty with that electrode disposed on the interior of the aperture tube, the one disposed in the first vessel was subject to breakage, bending and interference. The interference occurred in connection with handling the vessel since it had to be filled, emptied and reinstalled from time to time, and it additionally occurred when an electric mixer was used to agitate the suspension in the first vessel. Since it is important to keep the aperture under continuous surveillance by means of a microscope, a clear passage through the tube at the level of the aperture and through the vessel had be be maintained, and often the hanging electrode obstructed the view.

The primary object of the invention is to obviate the disadvantages mentioned above by providing an aperture tube having a metallic coating on its surface as a permanent part of the tube, serving as the electrode for the first vessel and thereby providing a terminal for attachment of an electrical lead thereto.

Other objects will appear as the description of the inven-65 tion proceeds in which the details of the preferred embodiment are set forth. The drawing illustrates the same and in the said drawing:

FIG. 1 is a sectional view with portions shown in elevation of an aperture tube constructed in accordance with the invention inserted into a beaker and having some elements of an electronic particle study apparatus utiliz3

ing the Coulter principle of particle study associated there-

FIG. 2 is a sectional view on an enlarged scale taken generally along the line 2-2 of FIG. 1 and in the direction indicated.

FIG. 3 is a sectional view on an enlarged scale taken generally along the line 3-3 of FIG. 1 and in the direction indicated.

FIG. 4 is a fragmentary elevational view of the bottom end of the aperture tube viewed from the right side of 10 FIG. 1 looking toward the aperture.

Generally the invention is characterized by the provision of an aperture tube the contours of which are those of the conventional aperture tube used with an electronic particle apparatus utilizing the Coulter principle of particle study but in which the bottom end of the tube is selectively coated with a metallic layer to give rise to the advantages of the invention. The metallic layer is in the form of a coating or covering of platinum or other non-corrosive highly conductive metal applied by means 20 of commercially available platinum metallic paint and baked to hardness. The bottom end of the tube has uncoated areas for purposes to be explained, as do the sides of the tube, although the latter are not essential to the invention.

Referring now to the details, in FIG. 1 there is illustrated a glass aperture tube 10 shown disposed in a beaker 12, the beaker carrying a suspension 14 having therein particles it is desired to count and size. The aperture tube has an upper portion 16 provided with a ground 30 tapered socket 18 and integral hooks 20. An integral narrow diameter portion 22 extends downwardly from the portion 16 and terminates in the end 24 which is flattened on a lateral side as shown at 26 to provide a surface to which the wafer described below may be affixed.

The lower end 24 is aligned with a microscope 28. A light source 30 directs a beam 32 through a lens system shown symbolically at 33 to the microscope 28 which may either be disposed directly alongside of the beaker 12 or may be in the form of an optical system for projecting an image of the aperture for viewing by the user

A glass fitting 34 is shown engaged in the socket 18 so that its integral hooks 36 may be engaged by springs or rubberbands 38 tightly to hold the fitting 34 upon the upper end of the aperture tube 10. One outlet from the fitting is designated 38 and a stock cock 40 is shown in this outlet to control the vacuum required in accordance with the teachings of Patent 2,869,078. Another outlet at 42 is connected to a manometric device to provide for 50 the metering of suspension passing from the beaker 12 into the aperture tube 10. An electric agitator 44 is shown with its propeller 46 immersed in the body of fluid 14.

A platinum foil electrode 48 is disposed on the interior of the aperture tube 10 and is connected by a lead 49 sealed in and led through a wall of the fitting 34 to the counting circuitry. The other connection to the counting circuitry is required to establish electrical connection with the body of fluid 14 and in this respect the invention differs from known structures.

The narrow diameter portion 22 of the aperture tube 10 is provided with a coating of selective configuration adhered to the glass surface, this coating being of platinum or similar metal and therefore providing the electrical contact with the fluid 14. A ring 50 of the coating is shown at the upper end of the portion 22 at its juncture with the portion 16, and a platinum wire 52 is welded or soldered to the ring 50 at this point to enable connection of the electrical lead 54 thereto.

On opposite sides of the narrow portion 22, there are axially extending bands 56 of the applied metal coating that extend downwardly to the end 24. These narrow bands 56 enable the contents of the aperture tube 10 to be seen but it is not essential that they be provided since the entire portion 22 above the end may be covered. The 75 defined first uncoated area and a second uncoated area

bottom end 24 is also fully coated by means of the coating shown at 58 except for aligned circular areas at 60 and 62. The alignment is horizontal. As seen in FIG. 2 and in FIG. 4, the area 62 is concentric with the wafer 64 and its scanning aperture 68, the latter being aligned with the orifice 70 formed in the tube end 24. Reference may be had to Patent 3,122,431 for the method of mounting the wafer 64. The absence of coating enables the beam 32 freely to pass through the wall of the end 24 at the area 60 and illuminate the aperture 63 so that the microscope or projection system 28 may enable viewing

The diameter of the area 60 should be of dimension to provide sufficient space for the beam 32 freely to illuminate the aperture 68. One example had a diameter of approximately 1/4 inch. The diameter of the area 62 is of more critical dimension where greatest sensitivity is desired. It is shown relatively large in the drawing but if made smaller the sensitivity of the operation increases. The only limitation to decrease in diameter is that it should not be that small as to cause high current densities in the vicinity of the aperture 68. A rough rule is that the radius of the area 62 should be no less than ten times the diameter of the aperture 68. In the case of small aper-25 tures of the order of 15 to 50 micrcons there is no problem since the radius of the area 62 is usually much greater than ten times the aperture diameter. In the case of apertures of the order of several hundred microns, one must use caution in choosing the diameter of the area 62.

With respect to the particular type of coating, a platinum paint is available from the E. I. du Pont de Nemours of Wilmington, Delware, which is painted on and baked in accordance with instructions of the manufacturer.

Minor variations are capable of being made without 35 departing from the spirit or scope of the invention as embodied in the appended claims.

What it is desired to secure by Letters Patent of the United States is:

- 1. In an aperture tube for use in an electronic particle 40 study apparatus, wherein said tube is formed as an elongate glass vessel having an exterior surface and adapted to be filled with fluid, wherein the lower end of the vessel carries a minute scanning aperture which is immersible in a suspension of the particles and the vessel has means at its upper end for connection to an exterior device for applying internal suction to direct the suspension through the aperture and an inner electrode suspended within the vessel and an outer electrode is adapted to be suspended in the particle suspension exterior of the vessel for establishing a current path therebetween capable of an effective monitorable electrical change upon the passage of each suspended particle through the aperture: the herein invention which comprises, the electrode normally suspended in the particle suspension exterior of the vessel comprising an opaque, conductive coating bonded to the exterior surface of the vessel, said coating having a first portion substantially encircling the aperture but being spaced therefrom to define a first uncoated area isolating the surface of the vessel immediately surrounding the 60 aperture and an extension portion of the coating extending from the first portion continuously along the length of the vessel to a location remote from the first portion.
 - 2. A tube as claimed in claim 1 in which the coating is platinum intimately bonded to said lower end of the vessel.
- 3. A tube as claimed in claim 1 in which said extension portion of said coating extends upwardly a substantial distance along said vessel and includes a portion which 70 under normal use would be spaced above the surface of said suspension and means on the extension portion for connecting an electrical lead thereto.
 - 4. The aperture as claimed in claim 1 in which the coating covers the lower end of the tube except for the

diametrically opposite said first uncoated area and coaxial therewith and the aperture.

5. In an electronic particle apparatus which includes a first vessel adapted to have a suspension of particles disposed therein, a second vessel adapted to be filled with liquid and having a minute aperture in the bottom end thereof, the second vessel having its lower end with the aperture immersed in the first vessel, means connected to the upper end of the second vessel to establish a head between the vessels so that the suspension will tend to flow through the aperture carrying the particles therewith, a first electrode in the suspension, a second electrode in the liquid of the second vessel for establishing an electrical current through the aperture and a signal detecting device having its input connected to said electrodes for detecting signals generated by particles passing through the aperture, the invention herein which comprises: said first electrode being a metallic coating bonded to the exterior surface of the bottom portion of said second vessel adjacent to but spaced from said aperture to define an uncoated portion surrounding said aperture and said metallic coating extending upward a substantial distance in the form of vertical bands for a portion of said distance to terminate substantially above the suspension.

6. The structure as claimed in claim 5 in which there is a second uncoated area of said second vessel coaxial with the aperture diametrically opposite same and aligned therewith

7. In an electronic particle study apparatus; a vessel adapted to have a suspension of particles therein, an aperture tube with an aperture adjacent the bottom end thereof disposed in the vessel and the bottom end including the aperture adapted to be immersed in the suspension, the tube adapted to carry a body of fluid therein and the upper end thereof having means for connecting the fluid in a closed manometric system to apply suction to the fluid so as to draw suspension through the aperture, a metallic electrode in the tube and having an electrical lead extending from said tube to the outside thereof to be connected to one terminal of a detector, a second electrode on the exterior of the tube and having a second electrical lead extending therefrom to a second terminal of said detector, said second electrode being a metallic coating bonded to the exterior surface of said tube at the said bottom end adjacent to but spaced from said aperture to defined an uncoated area encircling the immediate area of the aperture, a portion of said coating being free of said liquid suspension and the second electrical lead connected to said portion.

8. An apparatus as claimed in claim 7 in which the radius of the uncoated area is greater than ten times the diameter of the aperture.

9. An aperture tube for use as a part of a particle analyzing apparatus in which a suspension of particles is passed through an aperture from a first vessel to a second vessel, the aperture being provided in a wall of the second vessel, in which each vessel has a fluid body therein electrically insulated one from the other except through said aperture, in which the second vessel is at least partially immersed in the first body of fluid of the first vessel with the aperture below the level of the first body of fluid, in which the first body of fluid is a sample sus-

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pension, in which an electric current is established through said aperture and in which means are provided to detect signals generated by the passage of particles through said aperture, said aperture tube comprising said second vessel of insulating material having said aperture in said wall thereof to provide a passageway for liquid and electric current through said aperture to opposite surfaces of said wall, an electrode for said electric current comprising a thin adherent conductive coating on one of said surfaces and having a part thereof immediately adjacent said aperture but not engaging same, said coating having a portion thereof free from contact with said first body of fluid to serve as an electrical terminal for connection of an electrical circuit or the like thereto.

10. A tube as claimed in claim 9 in which said part immediately adjacent the aperture substantially surrounds the same.

11. A tube as claimed in claim 9 in which said one surface is the exterior surface of said vessel.

12. A structure as claimed in claim 9 in which said portion comprises a continuous extension of said coating along one surface of the wall terminating remote from the aperture.

13. In a particle analyzing apparatus which includes vessel of insulating material having a first body of liquid in the bottom of said vessel, an aperture in the vessel, a second body of liquid engaging the exterior bottom end of said vessel, said aperture providing a fluid communication between said bodies of liquid, particles being suspended in one body and means for driving liquid with particles from said one body to the other, an electrode in each body and means for establishing an electric current to flow between electrodes through said aperture, the invention herein which comprises one of said electrodes being a thin conductive coating bonded to a surface of said vessel and having a portion thereof extending adjacent to said aperture and having at least said portion immersed in one of said bodies of liquid and at least another portion of said conductive coating is not immersed.

14. A structure as claimed in claim 13 in which at least another portion of said conductive coating is not immersed.

15. A structure as claimed in claim 13 in which said surface of said vessel is the exterior surface.

References Cited

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UNITED STATES PATENT OFFICE CERTIFICATE OF CORRECTION

Patent No. 3,361,965

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It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

In the drawing, Fig. 1, the reference character 49 should not be underline and further, a lead line from reference character 49 to the adjacent wire lead should be shown; column 4 line 73, after "aperture" insert -- tube --.

Signed and sealed this 27th day of May 1969.

(SEAL)
Attest:

Edward M. Fletcher, Jr.

Attesting Officer

Commissioner of Patents