

US Patent & Trademark Office

Patent Public Search | Text View

United States Patent Application Publication

20250258080

Kind Code

A1

Publication Date

August 14, 2025

Inventor(s)

SAKURAMOTO; Keijiro et al.

PARTICLE ANALYSIS DEVICE

Abstract

In order to enable a device to be installed in a limited space on the periphery of a line and to keep a path for transportation of a sample from the line short, the present invention is configured so as to comprise a flow cell through which a sample including particles flows, a light source for radiating light to the particles in the flow cell, a photodetector for detecting secondary light from the particles, and a computation circuit for detecting an autocorrelation function from a light intensity signal outputted from the photodetector and analyzing the autocorrelation function or the particles included in the sample, an optical system unit including the flow cell and the light source, and a control unit including the photodetector and the computation circuit being separate from each other and connected via a light guiding member for guiding the secondary light to the photodetector.

Inventors: SAKURAMOTO; Keijiro (Kyoto-shi, Kyoto, JP), MORI; Tetsuya (Kyoto-shi, Kyoto, JP)

Applicant: HORIBA, Ltd. (Kyoto-shi, Kyoto, JP)

Family ID: 1000008618517

Appl. No.: 18/702425

Filed (or PCT Filed): January 17, 2023

PCT No.: PCT/JP2023/001141

Foreign Application Priority Data

JP 2022-007799

Jan. 21, 2022

Publication Classification

Int. Cl.: G01N15/0205 (20240101)

Background/Summary

TECHNICAL FIELD

[0001] This invention relates to a particle analysis device.

BACKGROUND TECHNOLOGY

[0002] Slurry particles are sometimes used for polishing semiconductors in a semiconductor manufacturing process, and if too large particles are contained in the slurry particles, they may damage the semiconductors. Therefore, there are some cases that particle analysis devices that use in-line or on-line dynamic scattering methods to measure particle size distribution are used (Patent document 1).

[0003] However, environment around the line in which particles flow varies depending on a manufacturing site, and there may be a case that an installation space of the device is limited. A conventional particle analysis device, in which a flow cell, a light source, a photodetector, and other components are integrally housed in a single case, is bulky in size, and if the device does not fit the installation space, it requires that a shape of the line be changed.

[0004] It is a matter of course if the particle analysis device is installed in a wide area away from the line and samples are transported from the line to that area, it is possible to conduct analysis without changing the shape of the line. However, in this case, the line to transport the sample becomes long so that other problems, such as increased sample loss, changes in sample properties, and decreased responsiveness, would occur.

PRIOR ART REFERENCES

[0005] Patent document 1: Japanese Unexamined Patent Application Publication No. 2002-22644

SUMMARY OF THE INVENTION

Problem to be Solved by the Invention

[0006] The present claimed invention is made to solve the above-mentioned problems, and its main subject is to make it possible for the particle analysis device to be installed in a limited space around the line and to keep a route for transporting the sample from the line short.

Means to Solve the Problems

[0007] More specifically a particle analysis device in accordance with this invention comprises a flow cell through which a sample containing particles flows, a light source that irradiates light to the particles in the flow cell, a photodetector that detects secondary light from the particles, and an arithmetic circuit that calculates an autocorrelation function from a light intensity signal output by the photodetector and analyzes the particles contained in the sample using the autocorrelation function, and is characterized by that an optical system unit including the flow cell and the light source, and a control unit including the photodetector and the arithmetic circuit are separately provided from each other and are connected through a light guide member that guides the secondary light to the photodetector.

[0008] In accordance with the particle analysis device having the above configuration, since the optical system unit and the control unit are separately provided from each other, and the photodetector is arranged on the control unit side, the optical system unit becomes much more downsized compared with a conventional device as a whole.

[0009] As a result of this, even if the space around the line where particles flow at a manufacturing site is limited, analysis becomes possible as long as the optical system unit can be installed in the installation space, and as a result of this, a route for transporting the sample from the line can be

kept short, which in turn minimizes sample loss, sample degeneration, and response degradation as much as possible.

[0010] In order to secure an accuracy of analyzing particles by the dynamic scattering method, it is not enough to acquire only the intensity of the scattered light, but also its phase information is necessary. In other words, it is necessary to guide photons emitted from the particle by light irradiation to the photodetector in the same order in which the photons emerge. Therefore, if the optical system unit and the control unit are connected by, for example, a rather thick optical fiber, the phase information will be lost due to the thick optical fiber, resulting in a problem that the measurement accuracy cannot be secured.

[0011] Therefore, in order to secure the measurement accuracy by the dynamic scattering method, it is preferable that the light guide member guides the secondary light emitted from the particles to the photodetector without losing phase information of the secondary light. In other words, it is best that the core diameter of the light guide member is single-mode for the light emitted from the light source.

[0012] However, since it is difficult for the single-mode optical fiber to obtain sufficient intensity of the scattered light because the core diameter is extremely small, it is preferable that the light guide member is an optical fiber having a core diameter of up to 20 times that a single-mode core diameter.

[0013] On the other hand, if the optical system unit and the control unit are connected by a thin optical fiber as described above, the positioning of the incident side end part of the optical fiber becomes severe in order to secure the intensity of the scattered light to be detected.

[0014] Therefore, it is preferable that the particle analysis device further comprises a connection portion that is placed between the light guide member and the optical system unit and that connects the light guide member and the optical system unit, and the connection portion is operated from outside the optical system unit.

[0015] In accordance with such a configuration, the position of the incident side end part of the optical fiber can be easily adjusted even after the particle analysis device is installed in a site where in-line or on-line analysis can be conducted.

[0016] To explain the above-mentioned positioning more concretely, in case that the light from the light source is irradiated horizontally to the flow cell, the scattered light should be strong at the same height position as that of the light. If the incident end side part of the optical fiber is displaced vertically from this height, the intensity of the detected scattered light will drop significantly.

[0017] Therefore, it is preferable that the connection portion is configured to allow adjusting a position of the incident side end part of the light guide member in a direction orthogonal to the irradiation direction of the light from the light source to the flow cell.

[0018] In accordance with this configuration, for example, if the light from the light source is emitted in a horizontal direction, it is possible to adjust a position of the incident side end part of the light guide member in a vertical direction orthogonal to the horizontal direction so that the intensity of the scattered light to be detected can be secured.

[0019] It is preferable that the optical system unit comprises a heat dissipating member placed between the light source and a casing constituting the optical system unit.

[0020] In accordance with this configuration, a cooling structure using, for example, a Peltier element is not necessary so that the optical system unit can be further downsized.

[0021] In addition, since a cooling fan is not required, it is also possible to prevent dust and dirt from entering the optical system unit from outside.

[0022] It is preferable that the optical system unit comprises a mirror that reflects transmitted light that is emitted from the light source and that is transmitted through the flow cell, and a low reflectance member to which the transmitted light reflected by the mirror is irradiated.

[0023] In accordance with this configuration, it is possible to absorb the transmitted light by the low reflectance member so that stray light can be prevented from entering into the light guide

member such as the optical fiber.

[0024] It is preferable that the optical system unit comprises one or more light reducing filters that reduce the light from the light source and a motor that drives the one or more light reducing filters, and the control unit comprises a control circuit that controls the motor.

[0025] In accordance with this configuration, it is possible to further downsize the optical system unit by arranging the control circuit for the motor on the control unit side.

Effects of the Invention

[0026] In accordance with this configuration, it is possible to downsize the optical system unit and to keep a short route for transporting the samples from the line by arranging the optical system unit in a limited space around the line through which particles flow, thereby reducing sample loss, sample denaturation, and response degradation as much as possible.

Description

BRIEF DESCRIPTION OF THE DRAWINGS

[0027] FIG. 1 A schematic diagram showing an overall configuration of a particle analysis device of one embodiment.

[0028] FIG. 2 A schematic diagram showing an optical system unit and a control unit of this embodiment.

[0029] FIG. 3 A schematic diagram showing an internal structure of the optical system unit of this embodiment.

[0030] FIG. 4 A schematic diagram showing the internal structure of the optical system unit of this embodiment.

[0031] FIG. 5 A schematic diagram showing an internal structure of the control unit of this embodiment.

BEST MODE FOR EMBODYING THE INVENTION

[0032] One embodiment of a-particle analysis device of the present claimed invention will be explained with reference to drawings.

[0033] The particle analysis device of this embodiment is used together with a manufacturing system, such as, for example, a semiconductor manufacturing system, used for a prescribed manufacturing process and performs in-line or on-line measurement on particle size distribution of particles used in the manufacturing system.

[0034] An example of the particles as an object to be measured can be represented by, for example, polishing slurries such as CMP slurries.

[0035] The particle analysis device measures particle size distribution using a dynamic light scattering (DLS) method and is capable of measuring the particle size distribution in a range that includes, for example, a nanoscale particle diameter area.

[0036] If explaining a configuration of the particle analysis device **100** schematically, as shown in FIG. 1, the particle analysis device **100** comprises a flow cell (C) through which a sample containing particles flows, a light source **10** that irradiates light to the particles in the flow cell (C), one or more photodetectors **20** that detect secondary light from the particles, and an arithmetic circuit **30** that calculates the particle size distribution based on fluctuations in the light intensity signal output from the photodetectors **20**.

[0037] As shown in FIG. 2, the particle analysis device **100** comprises an optical system unit **U1** and a control unit **U2**, which are separately provided from each other, and these units **U1** and **U2** are connected through a light guide member **L1** that guides the above-mentioned secondary light.

[0038] The optical system unit **U1** and the control unit **U2** in this embodiment are connected also through a power supply cable **L2** and an IO line **L3** as well as the light guide member **L1**.

[0039] First, the optical system unit **U1** will be explained.

[0040] As shown in FIG. 3 and FIG. 4, the optical system unit U1 is connected to a piping constituting the above-mentioned manufacturing system and the sample containing the particles is guided from the piping and comprises at least the above-mentioned flow cell (C) and the light source 10.

[0041] The flow cell (C) and the light source 10 are, as shown in FIG. 2, housed in a first casing 40 in which a sample introduction port P1 and a sample derivation port P2 are formed.

[0042] The flow cell (C) and the light source 10 in this embodiment are arranged, as shown in FIG. 3, so that a flow direction (X) of the sample containing the particles flowing through the flow cell (C) is orthogonal to an irradiation direction (Y) (namely, a direction along an optical axis of the light passing through the flow cell (C)) of the light from the light source 10 to the flow cell (C).

[0043] The light source 10 is, for example, a laser light source that emits a laser beam and the light source 10 is held by a holding member 50 made of a metal such as aluminum.

[0044] The holding member 50 locates between the light source 10 and the first casing 40 and is thermally connected thereto and functions as a heat dissipating member that transfers heat from the light source 10 to the first casing 40.

[0045] As shown in FIG. 3 and FIG. 4, the optical system unit U1 of this embodiment comprises a plurality of light reduction filters 60 that reduce light from the light source 10 and a motor (not shown in drawings) that drives a plurality of the light reduction filters 60 so as to be able to selectively set the light reduction filter 60 through which the light from the light source 10 passes among a plurality of the light reduction filters 60.

[0046] More concretely explained, a plurality of the light reduction filters 60 are held by a common retainer 61 placed between the light source 10 and the flow cell (C) and are mounted on the retainer 61 at a plurality of locations on, for example, a circumference of the retainer 61.

[0047] The motor is a stepping motor that, for example, rotates and drives the above-mentioned retainer 61 and is so configured that the light reduction filter 60 to be placed between the light source 10 and the flow cell (C) is selectively determined by controlling a rotational angle of the motor.

[0048] Furthermore, the optical system unit U1 in this embodiment comprises a mirror (M) that reflects the transmitted light emitted from the light source 10 and transmitting through the flow cell (C) and a low reflectance member (not shown in drawings) to which the transmitted light reflected by the mirror (M) is irradiated. For example, a black material can be used as the low reflectance member, and as an example of the low reflectance member, flock paper can be used. A part, whose reflectance is low, of a member constituting the optical system unit U1, may be used as the low reflectance member without using flock paper.

[0049] Next, a control unit U2 will be explained.

[0050] As shown in FIG. 2, the control unit U2 is placed at a position distant from the above-mentioned optical system unit U1 and, as shown in FIG. 5, comprises at least the above-mentioned photodetector 20 and the arithmetic circuit 30.

[0051] The photodetector 20 and the arithmetic circuit 30 are housed in a second casing 70 that is different from the above-mentioned first casing 40 and are connected through connection wiring.

[0052] The photodetector 20 detects the scattered light from the particles and outputs a light intensity signal indicating the scattered light intensity to the arithmetic circuit 30. Concretely, the photodetector 20 makes use of a photomultiplier tube that can detect microscopic light.

[0053] The arithmetic circuit 30 comprises an FPGA, a CPUs, a memory, an AD converter and a DA converter, and calculates an autocorrelation function from the light intensity signal output by the photodetector 20 and calculates the particle size distribution of the particles contained in the sample from the autocorrelation function by cooperatively working each of the devices and by executing the program stored in the memory.

[0054] The arithmetic circuit 30 is not necessarily limited to calculating the particle size distribution, however, the arithmetic circuit 30 may analyze various characteristics of the particles

such as calculating, for example, gel characteristics like an average lattice spacing of gel from the autocorrelation function.

[0055] The control unit **U2** of this embodiment further comprises a control circuit **80** that controls the above-mentioned motor, as shown in FIG. 5. The control circuit **80** receives an input selection signal indicating the light reduction filter **60** selected by a user through an input means and controls the rotational angle of the motor based on the input selection signal.

[0056] Next, a light guide member **L1** that connects the above-mentioned optical system unit **U1** and the above-mentioned control unit **U2**, and its peripheral structures will be explained.

[0057] The light guide member **L1** is so configured that an incident side end part **L1a** through which the light enters is connected to the optical system unit **U1** and an emission side end part **L1b** through which the light is emitted is connected to the control unit **U2**, as shown in FIG. 2, and the scattered light generated from the particles in the flow cell (C) is received by the incident side end part **L1a** and then the scattered light is transmitted to the emission side end part **L1b** and injected into the photodetector **20**. The light guide member **L1** is concretely an optical fiber.

[0058] The optical fiber as being the light guide member **L1** guides photons the as being the scattered light generated from the particles to the photodetector **20** without losing or almost losing its phase information. Concretely, the light guide member **L1** has a core diameter ranging from a core diameter to be single-mode for the laser beam emitted from the light source **10** to a core diameter of up to 20 times that diameter.

[0059] As shown in FIG. 4, the particle analysis device **100** of this embodiment further comprises a connection portion **90** that is placed between the light guide member **L1** and the optical system unit **U1** and that connects the light guide member **L1** and the optical system unit **U1**.

[0060] This connection portion **90** is mounted on the first casing **40** and supports the incident side end part **L1a** of the light guide member **L1**, and the incident side end part **L1a** of the light guide member **L1** is supported in a manner of being able to adjust the position of the incident side end part **L1a**.

[0061] If explained in more detail, the connection part **90** is configured to make it possible to adjust the position of the light guide member **L1** in a direction (A) orthogonal to an irradiation direction (Y) of the light from the light source **10** to the flow cell (C) by being operated from outside the optical system unit **U1**.

[0062] Concretely, the connection portion **90** has a fixing portion **91** that is fixed to the first casing **40**, and a support portion **92** that is mounted on the fixing portion **91** by means of screws or the like to enable relative movement to the fixing portion **91** and that also supports the incident side end portion **L1a** of the light guide member **L1**.

[0063] With this configuration, the position of the incident side end portion **L1a** of the light guide member **L1** is adjusted by positioning the holding portion relative to the fixing portion **91**. In this embodiment, the position of the incident side end portion **L1a** of the light guide member **L1** is configured to be able to be adjusted along a flow direction (X) of the sample in the flow cell (C).

[0064] In accordance with the particle analysis device **100** having the above-mentioned configuration, the optical system unit **U1** and the control unit **U2** are arranged separately from each other, and since the photodetector **20** is arranged in the control unit **U2**, the optical system unit **U1** is much more downsized compared with an entire conventional device.

[0065] In accordance with this configuration, even in case that the space around the line in which the particles flow at the manufacturing site is limited, it becomes possible to conduct analysis as long as the optical system unit **U1** can be installed in the installation space. As a result of this, it is possible to keep the path for transporting the sample from the line to the particle analysis device **100** short, thereby to suppress deterioration in sample loss, sample denaturation, and response degradation as much as possible.

[0066] In addition, since the control circuit **80** of the motor that drives the light reduction filter **60** is housed in the control unit **U2**, it is possible to further downside the optical system unit **U1**.

[0067] Furthermore, since the light guide member **L1** is the optical fiber having the core diameter ranging from the core diameter to be single-mode for the light emitted from the light source **10** to the core diameter up to 20 times that diameter, in other words, the light guide member **L1** is an extremely thin optical fiber, it is possible to guide the photons as being the scattered light emitted from the particles to the photodetector **20** without losing or almost without losing its phase information so that the measurement accuracy can be secured.

[0068] On the other hand, if a thin optical fiber is used as mentioned above, the incident side end part **L1a** of the optical fiber also becomes minute. Then, positioning the incident side end part **L1a** of the optical fiber becomes a very difficult and delicate work in order to secure the intensity of the scattered light detected by the photodetector **20**.

[0069] In contrast, since the connection portion **90** of this embodiment is configured so that the incident side end part **L1a** of the light guide member **L1** can be adjusted to position along the direction (A) orthogonal to the irradiation direction (Y) of the light source **10** to the flow cell (C), and concretely along the flow direction (X) of the sample in the flow cell (C), it becomes possible to secure the intensity of the scattered light to be detected.

[0070] In addition, since the holding member **50** that holds the light source **10** is placed between the light source **10** and the first casing **40** and functions as a heat-dissipating member that transfers heat of the light source **10** to the holding member **50**, a cooling structure using, for example, a Peltier element can be made unnecessary so that the optical system unit **U1** can be further downsized. This can eliminate the need for a cooling fan and prevent dust and dirt from entering the optical system unit **U1** from the outside.

[0071] Furthermore, since the transmitted light that is emitted from the light source **10** and transmitted through the flow cell (C) is reflected by the mirror (M) toward a low reflectance member, the transmitted light can be absorbed by the low reflectance member and stray light can be prevented from entering the optical fiber.

[0072] The present invention is not limited to the above-mentioned embodiment.

[0073] For example, the flow cell (C) and the light source **10** are housed in the first casing **40** in the above-mentioned embodiment, however, they are not necessarily be housed in a casing and may be mounted on, for example, a surface plate.

[0074] In addition, the photodetector **20** and the arithmetic circuit **30** are also housed in the second casing **70** in the above-mentioned embodiment, however, they are not necessarily be housed in a casing and may be mounted on, for example, a surface plate. Furthermore, the photodetector **20** and the arithmetic circuit **30** do not necessarily need to be integrally provided, however, may be, for example, housed in separate casings or mounted on separate surface plates.

[0075] Furthermore, the connection portion **90** may be configured so that the incident side end **L1a** of the light guide member **L1** can be adjusted to position not only in the flow direction (X) of the sample in the flow cell (C), but also, for example, in the light irradiation direction (Y), in the direction of advancing or retracting with respect to the first casing **40**, or at an angle different from the previously mentioned directions.

[0076] In addition, the dimension of the optical fiber described in the above-mentioned embodiment is one mode of the light guide member **L1**, and an optical fiber with a diameter larger than the diameter described in the above-mentioned embodiment may be used as the light guide member **L1**.

[0077] Furthermore, the optical system unit **U1** of the above-mentioned embodiment has a configuration in which the light reduction ratio can be changed in stages using a plurality of the light reduction filters **60**, however, instead of a plurality of the light reduction filters **60**, a single light reduction filter that continuously changes the light reduction ratio by being rotated may be used.

[0078] It is a matter of course that the present invention is not limited to the above-mentioned embodiments, and various modifications are possible without departing from a spirit of this

invention.

Possible Applications in Industry

[0079] In accordance with the present invention, it becomes possible to install the particle analysis device in a limited space around the line and to keep a route for transporting the samples from the line short.

EXPLANATION OF CODES

[0080] **100** . . . particle analysis device [0081] **C** . . . flow cell [0082] **U1** . . . optical system unit [0083] **U2** . . . control unit [0084] **L1** . . . light guide member [0085] **10** . . . light source [0086] **20** . . . photodetector [0087] **30** . . . arithmetic circuit [0088] **40** . . . first casing [0089] **50** . . . holding member [0090] **60** . . . light reduction filter [0091] **70** . . . second casing [0092] **80** . . . control circuit [0093] **90** . . . connection portion [0094] **M** . . . mirror

Claims

1. A particle analysis device comprising a flow cell through which a sample containing particles flows, a light source that irradiates light to the particles in the flow cell, a photodetector that detects secondary light from the particles, and an arithmetic circuit that calculates an autocorrelation function from a light intensity signal output by the photodetector and analyzes the particles contained in the sample using the autocorrelation function, wherein an optical system unit including the flow cell and the light source, and a control unit including the photodetector and the arithmetic circuit are separately provided from each other and are connected through a light guide member that guides the secondary light to the photodetector.
 2. The particle analysis device described in claim 1, wherein the light guide member guides the secondary light emitted from the particles to the photodetector without losing phase information of the secondary light.
 3. The particle analysis device described in claim 1, wherein the light guide member is an optical fiber having a core diameter ranging from a core diameter to be single-mode for the light emitted from the light source to a core diameter of up to times that diameter.
 4. The particle analysis device described in claim 1, further comprising a connection portion that is placed between the light guide member and the optical system unit and that connects the light guide member and the optical system unit, wherein the connection portion is operated from outside the optical system unit.
 5. The particle analysis device described in claim 4, wherein the connection portion is configured to allow adjusting a position of the incident side end part of the light guide member in a direction orthogonal to the irradiation direction of the light from the light source to the flow cell.
 6. The particle analysis device described in claim 1, wherein the optical system unit comprises a heat dissipating member placed between the light source and a casing constituting the optical system unit.
 7. The particle analysis device described in claim 1, wherein the optical system unit comprises a mirror that reflects transmitted light that is emitted from the light source and that is transmitted through the flow cell, and a low reflectance member to which the transmitted light reflected by the mirror is irradiated.
 8. The particle analysis device described in claim 1, wherein the optical system unit comprises one or more light reducing filters that reduce the light from the light source and a motor that drives the one or more light reducing filters, and the control unit comprises a control circuit that controls the motor.
-