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TITLE OF THE INVENTION

OBJECT SEPARATING APPARATUS AND METHOD, AND
METHOD OF MANUFACTURING SEMICONDUCTOR SUBSTRATE ✓

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BACKGROUND OF THE INVENTION

FIELD OF THE INVENTION

The present invention relates to an object separating
apparatus and method, and a method of manufacturing a
10 semiconductor substrate.

DESCRIPTION OF THE RELATED ART

A substrate (SOI substrate) having an SOI (Silicon On
Insulator) structure is known as a substrate having a
15 single-crystal Si layer on an insulating layer. A device
using this SOI substrate has many advantages that cannot be
achieved by ordinary Si substrates. Examples of the
advantages are as follows.

(1) The integration degree can be increased because
20 dielectric isolation is easy.

(2) The radiation resistance can be increased.

(3) The operating speed of the device can be increased
because the stray capacitance is small.

(4) No well step is necessary.

25 (5) Latch-up can be prevented.

(6) A completely depleted field-effect transistor can

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be formed by thin film formation.

Since an SOI structure has the above various advantages, researches have been made on its formation method for several decades.

5 As one SOI technology, an SOS (Silicon On Sapphire) technology by which Si is heteroepitaxially grown on a single-crystal sapphire substrate by CVD (Chemical Vapor Deposition) has been known for a long time. This SOS technology was once recognized as the most matured SOI
10 technology. However, the SOS technology has not been put into practical use to date because, e.g., a large amount of crystal defects are produced by lattice mismatch in the interface between the Si layer and the underlying sapphire substrate, aluminum that forms the sapphire substrate mixes
15 in the Si layer, the substrate is expensive, and it is difficult to obtain a large area.

Various SOI technology appeared following the SOS technology. For these SOI technologies, various methods have been examined aiming at reducing crystal defects or
20 manufacturing cost. There are a method of implanting oxygen ions into a substrate to form a buried oxide layer, a method of bonding two wafers via an oxide film and polishing or etching one of the wafers to leave a thin single-crystal Si layer on the oxide film, and a method of implanting hydrogen
25 ions to a predetermined depth from the surface of an Si substrate having an oxide film, bonding the Si substrate to





the other substrate, and peeling the latter substrate (the other substrate) by a heat treatment while leaving a thin single-crystal Si layer on the oxide film.

The present applicant has disclosed a new SOI technology
5 in Japanese Patent Laid-Open No. 5-21338. In this technology, a first substrate obtained by forming a non-porous single-crystal layer on a single-crystal semiconductor substrate having a porous layer is bonded to a second substrate via an insulating layer (SiO_2), and the
10 two substrates are separated from the porous layer to transfer the non-porous single-crystal layer to the second substrate. This technology is advantageous in that the SOI layer has high film thickness uniformity, the crystal defect density in the SOI layer can be decreased, the SOI layer has
15 high surface planarity, no expensive special fabrication apparatus is necessary, and SOI substrates having SOI films about a few hundred Å to 10 μm thick can be fabricated by the same fabrication apparatus.

In addition, the present applicant has disclosed
20 another technology in Japanese Patent Laid-Open No. 7-302889 in which, after the first and second substrates described above are bonded, the first substrate is separated from the second substrate without breaking, and the separated first substrate is reused by smoothening the surface and again
25 forming a porous layer. Since the first substrate can be economically used, this technology has the advantages that



the fabrication cost can be largely reduced and the fabrication process is also simple.

In the above technologies, however, when the two bonded substrates are separated it is necessary to prevent damages
5 to the substrates and protect the fabrication apparatus and the like from contamination caused by the generation of particles.

SUMMARY OF THE INVENTION

10 The present invention has been made in consideration of the above situation, and has as its object to apply a separating apparatus and method suitable to separate semiconductor substrates or other objects, and a method of manufacturing a semiconductor substrate.

15 According to the present invention, there is provided a separating apparatus characterized by comprising jet means having a fluid jet unit for ejecting a jet of fluid to an object to be separated to separate the object, and control means for changing a condition for ejecting the fluid from
20 the jet unit in accordance with progress of object separation processing.

The separating apparatus preferably further comprises a holding portion for holding the object to be separated.

In the separating apparatus, preferably, the object to
25 be separated has a plate shape, and the holding portion holds the plate-like object to inject the fluid against the

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plate-like object in a planar direction thereof.

In the separating apparatus, the control means preferably changes a pressure of the fluid to be ejected from the jet unit in accordance with progress of object separation
5 processing.

In the separating apparatus, the control means preferably controls the jet means to set the pressure of the fluid to be relatively high in separating a vicinity of a peripheral portion of a separation surface of the object to
10 be separated.

In the separating apparatus, the control means preferably controls the jet means to set the pressure of the fluid to be relatively high in separating a vicinity of a central portion of a separation surface of the object to be
15 separated.

In the separating apparatus, the control means preferably controls the jet means to set the pressure of the fluid to be relatively high in separating vicinities of peripheral and central portions of a separation surface of
20 the object to be separated and controls the jet means to set the pressure of the fluid to be relatively low in separating an intermediate portion between the vicinities of the peripheral and the central portions.

In the separating apparatus, the control means
25 preferably changes the condition for ejecting the fluid from the jet unit in accordance with a relative positional

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relationship between the jet unit and the object to be separated.

In the separating apparatus, the control means preferably changes the condition for ejecting the fluid from
5 the jet unit as a function of time.

The separating apparatus preferably further comprises scanning means for scanning the jet unit.

The separating apparatus preferably further comprises rotating means for rotating the object about an axis
10 perpendicular to a separation surface of the object to be separated.

The object to be separated preferably has a fragile layer as a separation layer. The fragile layer is preferably a porous layer or a layer having a microcavity.

15 According to the present invention, there is provided a separating method of separating an object by ejecting a jet of fluid from a jet unit to the object to be separated, characterized by comprising the control step of changing a condition for ejecting the fluid from the jet unit in
20 accordance with progress of object separation processing.

In the separating method, preferably, the object to be separated has a plate shape and is separated by injecting the fluid against the plate-like object in a planar direction thereof.

25 In the separating method, the control step preferably comprises changing a pressure of the fluid to be ejected from

the jet unit in accordance with progress of object separation processing.

In the separating method, the control step preferably comprises setting the pressure of the fluid to be ejected
5 from the jet unit to be relatively high in separating a vicinity of a peripheral portion of a separation surface of the object to be separated.

In the separating method, the control step preferably comprises setting the pressure of the fluid to be ejected
10 from the jet unit to be relatively high in separating a vicinity of a central portion of a separation surface of the object to be separated.

In the separating method, the control step preferably comprises setting the pressure of the fluid to be ejected
15 from the jet unit to be relatively high in separating vicinities of peripheral and central portions of a separation surface of the object to be separated and setting the pressure of the fluid to be ejected from the jet unit to be relatively low in separating an intermediate portion between the
20 vicinities of the peripheral and the central portions.

In the separating method, the control step preferably comprises changing the condition for ejecting the fluid from the jet unit in accordance with a relative positional relationship between the jet unit and the object to be
25 separated.

In the separating method, the control step preferably



comprises changing the condition for ejecting the fluid from the jet unit as a function of time.

The separating method preferably further comprises executing separation processing for the object while
5 scanning the jet unit along the separation surface of the object to be separated.

The separating method preferably further comprises executing separation processing for the object while rotating the object about an axis perpendicular to the
10 separation surface of the object to be separated.

In the separating method, the object to be separated preferably has a fragile layer as a separation layer. The fragile layer is preferably a porous layer or a layer having a microcavity.

15 According to an aspect of the present invention, there is provided method of manufacturing a semiconductor substrate, characterized by comprising the steps of forming a first substrate in which a porous layer and a non-porous layer are sequentially formed on one surface, bonding the
20 first substrate to a second substrate via the non-porous layer to form a bonded substrate stack, and separating the bonded substrate stack into two substrates while ejecting a jet of fluid to the porous layer of the bonded substrate stack, wherein the separation step comprises changing a
25 condition for ejecting the fluid in accordance with progress of the separation step.

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According to another aspect of the present invention, there is provided a method of manufacturing a semiconductor substrate, characterized by comprising the steps of implanting ions from a surface of a substrate consisting of a single-crystal semiconductor to a predetermined depth, thereby forming a first substrate in which a microcavity layer is formed, bonding a second substrate to a front surface side of the first substrate to form a bonded substrate stack, and separating the bonded substrate stack into two substrates while ejecting a jet of fluid to the microcavity layer of the bonded substrate stack, wherein the separation step comprises changing a condition for ejecting the fluid in accordance with progress of the separation step.

Further objects, features and advantages of the present invention will become apparent from the following detailed description of embodiments of the present invention with reference to the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

Figs. 1A to 1E are views for explaining a method of manufacturing an SOI substrate according to the preferred embodiment of the present invention;

Figs. 2A and 2B are views showing a force acting on a bonded substrate stack in the presence and absence of a V-shaped groove;

Fig. 3 is a view showing the schematic arrangement of

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a separating apparatus according to the first embodiment of the present invention;

Fig. 4 is a view showing the schematic arrangement of the separating apparatus according to the first embodiment
5 of the present invention;

Fig. 5 is a view showing the first arrangement of an adjustment mechanism;

Fig. 6 is a view showing the second arrangement of the adjustment mechanism;

10 Fig. 7 is a view showing an example of a jet nozzle driving robot;

Fig. 8 is a view showing another example of the jet nozzle driving robot;

15 Fig. 9 is a chart showing control in separation processing;

Fig. 10 is a chart showing control in separation processing;

Fig. 11 is a chart showing control in separation processing;

20 Fig. 12 is a view showing the force of jet acting on the bonded substrate stack;

Fig. 13 is a view showing the arrangement of a separating apparatus according to the second embodiment of the present invention;

25 Fig. 14 is a view showing the arrangement of a separating apparatus according to the third embodiment of the present

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invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

Preferred embodiments of the present invention will be
5 described below with reference to the accompanying drawings.

Figs. 1A to 1E are views for explaining a method of
manufacturing an SOI substrate according to the first
embodiment of the present invention.

In Fig. 1A, a single-crystal Si substrate 11 is
10 prepared, and a porous Si layer 12 is formed by anodizing
on the surface of the single-crystal Si substrate 11. In Fig.
1B, a single-crystal Si layer 13 as a non-porous layer is
epitaxially grown on the porous Si layer 12. After this, the
surface of the single-crystal Si layer 13 is oxidized to form
15 an SiO₂ layer 15. With this process, a first substrate 10
is formed.

In Fig. 1C, a single-crystal Si substrate 14 is prepared
as a second substrate 20. The first and the second substrates
10 and 20 are brought into contact with each other at room
20 temperature such that the SiO₂ layer 15 of the first substrate
10 faces the second substrate 20. The first and the second
substrates 10 and 20 are bonded by anode bonding, pressing,
heating, or a combination thereof. With this process, the
second substrate 20 and the SiO₂ layer 15 are firmly bonded.
25 The SiO₂ layer 15 may be formed on the single-crystal Si
substrate 11 side, as described above, or may be formed on



the second substrate 20 or on both the single-crystal Si substrate 11 and the second substrate 20 sides as far as the state shown in Fig. 1C is obtained by bringing the first and second substrates into contact.

5 In Fig. 1D, the two substrates bonded to each other are separated at the porous Si layer 12. The second substrate side has a multilayered structure of porous Si layer 12", single-crystal Si layer 13, SiO₂ layer 15, and single-crystal Si substrate 14. On the first substrate 10' side, a porous
10 Si layer 12' is formed on the single-crystal Si substrate 11.

After separation, the remaining porous Si layer 12' is removed from the substrate 10'. The surface of the substrate 10' is planarized, as needed, so the substrate 10' is used
15 again as a single-crystal Si substrate 11 for forming a first substrate 10.

After the bonded substrate stack is separated, in Fig. 1E, the porous layer 12" on the second substrate side (10' + 20) is selectively removed. With this process, a substrate
20 having a multilayered structure of single-crystal Si layer 13, insulating layer 15, and single-crystal Si substrate 14, i.e., an SOI structure is obtained.

In this embodiment, in the step shown in Fig. 1D, i.e., in the step of separating the two substrates bonded to each
25 other (to be referred to as a bonded substrate stack hereinafter), a separating apparatus for selectively



ejecting a high-pressure liquid or gas (fluid) to the porous Si layer as a separation region is used to separate the substrate stack into two substrates at the separation region.

[Basic Arrangement of Separating Apparatus]

5 This separating apparatus uses a water jet method. Generally, the water jet method ejects a high-speed, high-pressure stream of water upon an object to, e.g., cut or process a ceramic, a metal, a concrete, a resin, a rubber, or a wood member, remove a coating film from the surface,
10 or clean the surface ("Water Jet", Vol. 1, No. 1, page 4, (1984)).

 This separating apparatus ejects a high-speed, high-pressure stream of fluid to the porous layer (separation region) of the bonded substrate stack as a fragile structure
15 portion in the direction of substrate surface to selectively break the porous layer, thereby separating the substrate at the porous layer. The stream will be referred to as a "jet" hereinafter. The fluid forming a jet will be referred to as a "jet medium". As the jet medium, it is possible to use an
20 water, organic solvent such as alcohol, an acid such as fluoric acid or nitric acid, an alkali such as potassium hydroxide, a gas such as air, nitrogen gas, carbonic acid gas, rare gas, or an etching gas, or a plasma.

 In this separating apparatus, a jet is ejected to the
25 porous layer (separation region) exposed to the side surface of the bonded substrate stack, thereby removing the porous



layer from the peripheral portion to the central portion. With this process, the bonded substrate stack is separated into two substrates by removing only the separation region with low mechanical strength without damaging the main body.

5 Even when the side surface of the bonded substrate stack is covered with some thin layer, and the porous layer is not exposed, the thin layer can be removed by the jet, so the bonded substrate stack can be separated by the above-described method.

10 At the peripheral portion of the bonded substrate stack, the effect for splitting the bonded substrate stack into two substrates effectively acts when a V-shaped (concave) groove is formed in the bonded substrate stack along the side surface. Figs. 2A and 2B are views showing a force acting
15 on the bonded substrate stack in the presence and absence of a V-shaped groove. Fig. 2A shows a bonded substrate stack having a V-shaped groove 22. Fig. 2B shows a bonded substrate stack having no V-shaped groove.

As shown in Fig. 2A, in the bonded substrate stack having
20 the V-shaped groove 22, a force (to be referred to as a separation force hereinafter) is applied outward from the center of the bonded substrate stack, as indicated by an arrow 23. On the other hand, as shown in Fig. 2B, in the bonded substrate stack with a convex side surface, a force is applied
25 inward from the side surface of the bonded substrate stack, as indicated by an arrow 24. In the bonded substrate stack



having a convex side surface, the separation force does not act unless the side surface of the porous layer 12 as the separation region is removed by a jet 21.

Even when a thin film is formed on the side surface of the bonded substrate stack, the separation force acts on the bonded substrate stack as far as the bonded substrate stack has the V-shaped groove 22, as shown in Fig. 2A, so the thin layer can be easily broken.

To effectively use the jet, a width W_1 of the V-shaped groove 22 is preferably equal to or larger than a diameter d of the jet 21. For example, assume that each of the first substrate 10 and the second substrate 20 has a thickness of about 1 mm, and the bonded substrate stack has a thickness of 2 mm. Since the width W_1 of the V-shaped groove 22 is normally 1 mm, the diameter of the jet is preferably 1 mm or less. Since a general water jet apparatus uses a jet with a diameter of 0.1 to 0.5 mm, such a general water jet apparatus (e.g., a water jet nozzle) can be used.

The nozzle for ejecting a jet can have not only a circular shape but any other shapes. For example, when a slit-like nozzle is employed to eject a jet having a long rectangular section, the jet can be efficiently injected into the separation region (inserted between the two substrates).

The jet ejection conditions are determined in accordance with the type of separation region (e.g., a porous layer) or the shape of the side surface of the bonded substrate

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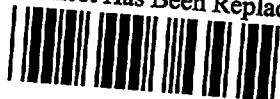


stack. Important parameters as the jet ejection conditions are the pressure applied to the jet medium, the jet scanning speed, the width or diameter of the nozzle (the nozzle diameter is almost the same as the jet diameter), the nozzle
5 shape, the distance between the nozzle and the separation region, and the flow rate of the jet medium.

The following techniques are used to separate the bonded substrate stack. 1) The jet is injected into the bonding interface parallel to the bonding interface while the nozzle
10 is scanned along the bonding interface. 2) The jet is injected into the bonding interface parallel to the bonding interface while the bonded substrate stack is scanned. 3) The jet is injected into the bonding interface parallel to the bonding interface and simultaneously scanned in a fan
15 shape at a pivot near the nozzle. 4) The jet is injected into the bonding interface parallel to the bonding interface while the bonded substrate stack is rotated about nearly the center of the bonded substrate stack (this technique is particularly effective when the bonded substrate stack has
20 a disk shape). 5) The jet is injected into the bonding interface parallel to the bonding interface while the jet nozzle directing the center of the bonded substrate stack... is scanned along the side surface of the bonded substrate stack. The jet need not always be ejected to be perfectly
25 parallel to the bonding interface.

To prevent damage to the substrate, the separation force

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applied to the bonded substrate stack in the axial direction is preferably several hundred gf per cm².

Fig. 3 is a view showing the schematic arrangement of a separating apparatus according to the preferred embodiment of the present invention. A separating apparatus 100 injects
5 a jet into the porous layer of the bonded substrate stack to separate the bonded substrate stack into two substrates.

The separating apparatus 100 has substrate holding portions 108 and 109 having vacuum chuck mechanisms 108a and
10 109a. A bonded substrate stack 101 is sandwiched by the substrate holding portions 108 and 109 and held. The bonded substrate stack 101 has a porous layer 101b as a fragile structure. The separating apparatus 100 separates the bonded substrate stack into two substrates 101a and 101c at
15 the porous layer 101b. In this separating apparatus 100, the bonded substrate stack is set such that the substrate 101a is placed on the first substrate 10' side in Fig. 1D and the substrate 101c is placed on the second substrate (10" + 20) side in Fig. 1D.

20 The substrate holding portions 108 and 109 are present on the same central axis. The substrate holding portion 108 is coupled to one end of a rotary shaft 106 which is rotatably axially supported by a support base 102 through bearings 104. The other end of the rotary shaft 106 is coupled to the rotary
25 shaft of a driving source (e.g., a motor) 110 fixed on the support portion 110. The rotational force generated by the



driving source 110 rotates the bonded substrate stack 101 vacuum-chucked by the substrate holding portion 108. In separating the bonded substrate stack 101, the driving source 110 rotates the rotary shaft 106 at a designated rotation speed in accordance with an instruction from a controller (not shown).

The substrate holding portion 109 is coupled to one end of a rotary shaft 107 which is rotatably and slidably axially supported by a support portion 103 through bearings 105. The other end of the rotary shaft 107 is coupled to a driving source (e.g., a motor) 111. The speed at which the driving source 110 rotates the rotary shaft 106 and that at which the driving source 111 rotates the rotary shaft 107 must apparently equal each other to prevent the bonded substrate stack 101 from twisting.

Both the driving sources 110 and 111 need not always be prepared, and either one of them may suffice. For example, when only the driving source 110 is used, before the bonded substrate stack 101 is separated, the rotary shaft 106, substrate holding portion 108, bonded substrate stack 101, substrate holding portion 109, and rotary shaft 107 integrally rotate. After the bonded substrate stack 101 is separated into two substrates, the members on the rotary shaft 107 side come to a halt.

Alternatively, the rotational force generated by one driving source may be branched into two forces so that the



rotary shafts 106 and 107 are rotated by the branched rotational forces, respectively.

A spring 113 for pressing the bonded substrate stack 101 is attached to the support portion 103 supporting the rotary shaft 107. Therefore, even when the bonded substrate stack 101 is not chucked by the vacuum chuck mechanisms 108a and 109a, the two substrates separated by the jet ejected from a jet nozzle 112 do not drop. In addition, when the bonded substrate stack 101 is separated while being pressed, the bonded substrate stack 101 can be stably held during separation.

A spring for pressing the bonded substrate stack 101 may also be arranged on the rotary shaft 106 side.

The separating apparatus 100 has an adjustment mechanism for adjusting the interval between the substrate holding portions 108 and 109.

Fig. 5 is a view showing the first arrangement of the adjustment mechanism. Fig. 5 shows an adjustment mechanism using an air cylinder 122. The air cylinder 122 is fixed on, e.g., the support portion 103. The driving source 111 is moved by a piston rod 121. To set the bonded substrate stack 101 in the separating apparatus 100, the air cylinder 122 is controlled to move the driving source 111 in a direction in which the interval between the substrate holding portions 108 and 109 increases (in the positive direction of X-axis). In this state, the bonded substrate stack 101 is set between



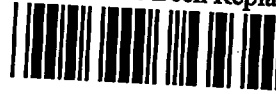
the substrate holding portions 108 and 109, and driving of the piston rod 121 by the air cylinder 122 is canceled. The substrate holding portion 109 presses the bonded substrate stack 101 by the action of the spring 113.

5 Fig. 6 is a view showing the second arrangement of the adjustment mechanism. Fig. 6 shows an adjustment mechanism using an eccentric cam 131 and a motor. The eccentric cam 131 is coupled to the motor (not shown). The interval between the substrate holding portions 108 and 109 is adjusted by
10 reciprocally moving a driving plate 132 coupled to the rear end of the motor 111. As described above, the spring 113 applies a force to the rotary shaft 107 in the negative direction of X-axis. When the bonded substrate stack 101 is to be held, a gap is formed between the eccentric cam 131
15 and the driving plate 132. Therefore, when the bonded substrate stack 101 is to be held, a press force acts on the bonded substrate stack 101.

 The above mechanism for adjusting the interval between the substrate holding portions 108 and 109 may also be mounted
20 on the substrate holding portion 108 side.

 A high-pressure pump 115 is coupled to the jet nozzle 112. When a high-pressure jet medium (e.g., water) is sent from the high-pressure pump 115 to the jet nozzle 112, a jet is ejected from the jet nozzle 112. The pressure applied to
25 the jet medium by the high-pressure pump 115 is controlled by a pressure control section 116.

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Substrate separation processing by the separating apparatus 100 will be described next.

To set the bonded substrate stack 101 in the separating apparatus 100, first, the bonded substrate stack 101 is
5 inserted between the substrate holding portions 108 and 109 by a conveyor robot and held while the center of the bonded substrate stack 101 matches that of the substrate holding portions 108 and 109. The bonded substrate stack 101 is vacuum-chucked by the substrate holding portion 108.

10 Next, the substrate holding portion 109 is pressed against the bonded substrate stack 101 by the force of the spring 113. More specifically, when the adjustment mechanism shown in Fig. 5 is employed to adjust the interval between the substrate holding portions 108 and 109, driving
15 of the piston rod 121 by the air cylinder 122 is canceled. When the adjustment mechanism shown in Fig. 6 is employed, the eccentric cam 131 is rotated such that the press force of the spring 113 acts on the bonded substrate stack 101.

When separation processing is to be executed, the bonded
20 substrate stack 101 need not always be vacuum-chucked by the vacuum chuck mechanisms 108a and 109a. This is because the bonded substrate stack 101 is held by the press force of the spring 113. However, when a weak press force is used, the bonded substrate stack 101 is preferably vacuum-chucked.

25 The rotary shafts 106 and 107 are rotated by the driving sources 110 and 111 in synchronism with each other. In this



state, the high-pressure jet medium (e.g., water) is sent from the high-pressure pump 115 to the jet nozzle 112 while the pressure is controlled by the pressure control section 116, thereby ejecting a high-speed, high-pressure jet from
5 the jet nozzle 112. The ejected jet is injected into the separation region of the bonded substrate stack 101. When the jet is injected, the porous layer 101b or the fragile structure of the bonded substrate stack 101 is broken, so the bonded substrate stack 101 is separated into two
10 substrates at the porous layer 101b.

Subsequently, while injecting the jet into the separation region (porous layer 101b) of the bonded substrate stack 101, the two separated substrates 101a and 101c are physically split from each other. More specifically, when
15 the adjustment mechanism shown in Fig. 5 is employed to adjust the interval between the substrate holding portions 108 and 109, the piston rod 121 is driven by the air cylinder 122 in the positive direction of X-axis (direction in which the spring 113 compresses) while the substrates are vacuum-
20 chucked by the substrate holding portions 108 and 109, respectively. When the adjustment mechanism shown in Fig. 6 is employed, the eccentric cam 131 is rotated to drive the rotary shaft 107 in the positive direction of X-axis (direction in which the spring 113 compresses) while the
25 substrates are vacuum-chucked by the substrate holding portions 108 and 109, respectively.



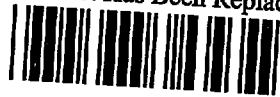
Then the substrates 101a and 101c are completely split, as shown in Fig. 4, jet ejection is stopped, and the substrates are detached from the substrate holding portions 108 and 109 by, e.g., the conveyor robot.

5 The jet nozzle 112 may be fixed in position but is preferably movable. This is because the position of the jet nozzle 112 is preferably adjusted in accordance with the type or size of bonded substrate stack 101.

Fig. 7 is a view showing the driving robot of the jet
10 nozzle 112. A driving robot 160 shown in Fig. 7 moves the jet nozzle 112 along a path 170. When the bonded substrate stack 101 is to be attached/detached to/from the substrate holding portions 108 and 109, the driving robot 160 moves the jet nozzle 112 to a retreat position 171. When the bonded
15 substrate stack 101 is to be separated and split, the driving robot 160 moves the jet nozzle 112 to an operation position 172. As described above, when the jet nozzle 112 is retreated in attaching/detaching the bonded substrate stack 101, the operation of attaching/detaching the bonded substrate stack
20 101 and, more particularly, the operation of attaching/detaching the bonded substrate stack 101 using the conveyor robot can be efficiently performed.

It is also effective to scan the jet nozzle 112 on the separation region of the bonded substrate stack 101 in the
25 planar direction of the bonded substrate stack 101 during separation and split of the bonded substrate stack 101. In

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this case, the bonded substrate stack 101 can be separated well without rotating it.

Fig. 8 is a view showing another example of the driving robot of the jet nozzle 112. A driving robot 161 shown in
5 Fig. 8 moves the jet nozzle 112 along a path 180. When the bonded substrate stack 101 is to be attached/detached to/from the substrate holding portions 108 and 109, the driving robot 161 moves the jet nozzle 112 to a retreat position 181. When the bonded substrate stack 101 is to be separated and split,
10 the driving robot 161 moves the jet nozzle 112 to an operation position 182. As described above, when the jet nozzle 112 is retreated in attaching/detaching the bonded substrate stack 101, the operation of attaching/detaching the bonded substrate stack 101 and, more particularly, the operation
15 of attaching/detaching the bonded substrate stack 101 using the conveyor robot can be efficiently performed.

It is also effective to scan the jet nozzle 112 on the separation region of the bonded substrate stack 101 in the planar direction of the bonded substrate stack 101 during
20 separation and split of the bonded substrate stack 101. In this case, the bonded substrate stack 101 can be separated well without rotating it.

The separating apparatus 100 separates the bonded substrate stack while appropriately changing the jet
25 pressure. The reason for this is as follows.

The jet pressure required to separate the bonded



substrate stack 101 changes depending on the portion of the separation region of the bonded substrate stack. For example, the separation force acting on the bonded substrate stack changes between the peripheral and the central portions
5 of the bonded substrate stack 101, so the jet pressure necessary for separation changes between the peripheral and the central portions. When the bonded substrate stack 101 is to be separated using a constant jet pressure, a high-pressure jet must be used throughout the separation
10 processing. In this case, the bonded substrate stack or separated substrates may break or be damaged, resulting in a low yield.

To solve this problem, the mechanical strength of the separation region can be lowered. However, when the
15 separation region is made excessively fragile, the separation region readily breaks in the step of bonding or cleaning the two substrates (first and second substrates), so it becomes difficult to manufacture a substrate with desired quality. In addition, particles may be generated
20 from the broken separation region to contaminate the manufacturing apparatus and the like.

To appropriately control the jet pressure during bonded substrate stack separation processing, the pressure is controlled on the basis of the positional relationship
25 between the jet nozzle 112 and the bonded substrate stack 101 or in accordance with time.



Figs. 9 to 11 are charts showing examples of jet pressure control during separation processing. The pressure control section 116 controls the pressure (jet pressure) generated by the high-pressure pump 115, on the basis of one of the
5 control procedures shown in Figs. 9 to 11.

Fig. 9 shows a control example in which separation processing is executed while fixing the jet nozzle 112 at the operation position 172 or 182 shown in Fig. 7 or 8 and rotating the bonded substrate stack 101. In the example
10 shown in Fig. 9, the jet pressure is adjusted in three steps. In a period T1, the peripheral portion of the bonded substrate stack 101 is mainly separated. During the period T1, the jet injected into the bonded substrate stack 101 is easily discharged, and the separation force hardly acts on the
15 bonded substrate stack 101, so the jet pressure is set to be high. At the peripheral portion of the bonded substrate stack 101, the jet medium is easily discharged, as described above. For this reason, even when the jet pressure is set to be relatively high, the bonded substrate stack 101 does
20 not break.

In a period T2, the intermediate portion between the peripheral portion and the central portion of the bonded substrate stack 101 (to be simply referred to as an intermediate portion hereinafter) is mainly separated.
25 Since the speed of jet lowers at the intermediate portion, the function of separating the bonded substrate stack 101



weakens due to the impact of the jet medium hitting the porous layer. However, at the intermediate portion, the discharge paths of the jet medium injected into the bonded substrate stack 101 decrease. The separation force due to the pressure of the jet medium injected into the bonded substrate stack 101 increases, and the bonded substrate stack 101 is separated mainly by this separation force. When the same pressure as that for the peripheral portion is applied to the jet medium, the bonded substrate stack 101 may break. For this reason, the pressure of the jet medium is set to be relatively low at the intermediate portion.

In a period T3, the central portion of the bonded substrate stack 101 is mainly separated. When the separated portion is close to the central portion, the separated portion of the bonded substrate stack 101 warps to increase the discharge paths of the jet medium. The pressure of the jet medium is reduced as compared to that in separating the intermediate portion, resulting in a smaller separation force. For this reason, at the central portion, the pressure of the jet medium is preferably set to be higher than that in separating the intermediate portion.

As described above, by appropriately changing the pressure of jet to be ejected from the jet nozzle 112 in accordance with the time elapse in separation processing, damage to the bonded substrate stack can be reduced while increasing the efficiency of separation processing.



Fig. 10 shows a control example in which separation processing is executed while scanning the jet nozzle 112 along the separation region of the bonded substrate stack 101 and simultaneously rotating the bonded substrate stack 101. Fig. 12 is a view showing the force of jet acting on the bonded substrate stack 101. When the jet is injected toward the peripheral portion of the bonded substrate stack 101, the jet injected against the bonded substrate stack 101 is easily discharged outward, as shown in Fig. 12, and the separation force of jet does not efficiently act on the bonded substrate stack 101. On the other hand, when the jet is injected toward the central portion of the bonded substrate stack 101, the jet injected against the bonded substrate stack 101 efficiently separates the bonded substrate stack 101. According to this example, when the jet nozzle 112 is to be scanned along the porous layer, the jet pressure is preferably changed in accordance with the positional relationship between the jet nozzle 112 and the bonded substrate stack 101.

In a section Y1, the jet pressure is stabilized. In a section Y2, the peripheral portion of the bonded substrate stack 101 is mainly separated. In this section Y2, since the jet is not efficiently injected against the bonded substrate stack 101, the jet pressure is preferably set to be relatively high, as described above.

In a section Y3, the intermediate portion of the bonded

substrate stack 101 is mainly separated. In this section Y3, since the jet efficiently separates the bonded substrate stack 101, as compared to the section Y2, the jet pressure is preferably set to be lower than that in the section Y2
5 to prevent damage to the bonded substrate stack 101.

In a section Y4, the central portion of the bonded substrate stack 101 is mainly separated. When the separated portion is close to the central portion by scanning the jet nozzle 112, the separated portion of the bonded substrate
10 stack 101 warps to increase the discharge paths of the jet medium. The pressure of the jet medium is reduced as compared to that in separating the intermediate portion, resulting in a smaller separation force. For this reason, at the central portion, the pressure of the jet medium is preferably
15 set to be higher than that in separating the intermediate portion. In this example, the bonded substrate stack 101 is separated while rotating it. Hence, separation of the bonded substrate stack 101 is complete when the jet nozzle 112 is scanned from the peripheral portion to the central portion
20 of the bonded substrate stack 101. In a section Y5, the pressure of the high-pressure pump is gradually reduced to stop the pump operation.

As described above, when the pressure of jet ejected from the jet nozzle 112 is appropriately changed in
25 accordance with the positional relationship between the jet nozzle 112 and the bonded substrate stack 101, damage to the



bonded substrate stack can be reduced while increasing the efficiency of separation processing.

Fig. 11 shows another control example in which separation processing is executed while scanning the jet nozzle 112 along the separation region of the bonded substrate stack and simultaneously rotating the bonded substrate stack 101. In this control example, the jet pressure is more smoothly changed in the section from the peripheral portion to the intermediate portion and in the section from the intermediate portion to the central portion, as compared to the control example shown in Fig. 10.

In the section Y1, the jet pressure is stabilized. In the section Y2, the peripheral portion of the bonded substrate stack 101 is mainly separated. In this section Y2, since the jet is not efficiently injected against the bonded substrate stack 101, the jet pressure is preferably set to be relatively high, as described above.

In the sections Y3 and Y4, the intermediate portion of the bonded substrate stack 101 is mainly separated. In the section Y3, the jet pressure is gradually reduced. In the section Y4, the jet pressure is gradually raised.

In the section Y5, the central portion of the bonded substrate stack 101 is mainly separated. In a section Y6, the pressure of the high-pressure pump is gradually reduced to stop the pump operation.

As described above, when the pressure of jet ejected



from the jet nozzle 112 is smoothly changed in accordance with the positional relationship between the jet nozzle 112 and the bonded substrate stack 101, the bonded substrate stack 101 can be separated by a jet having a more appropriate
5 pressure.

The methods of changing the pressure of jet ejected from the jet nozzle 112 are not limited to the above three examples. The method can be appropriately changed in accordance with the type or size of bonded substrate stack, the type or size
10 of separation layer, or the bonded substrate stack holding method.

The arrangement of a separating apparatus according to the second embodiment of the present invention will be described next. Fig. 13 is a view showing the arrangement
15 of the separating apparatus according to the second embodiment of the present invention.

A separating apparatus 400 has substrate holding portions 404 and 406 having vacuum chucks. A bonded substrate stack 101 is sandwiched by the substrate holding
20 portions 404 and 406 and held. The bonded substrate stack 101 has a porous layer 101b as a fragile structure. The separating apparatus 400 separates the bonded substrate stack into two substrates 101a and 101c at the porous layer 101b. In this separating apparatus 400, the bonded substrate
25 stack is set such that the substrate 101a is placed on the first substrate 10' side in Fig. 1D and the substrate 101c



is placed on the second substrate (10" + 20) side in Fig. 1D.

The substrate holding portion 404 is coupled to one end of a rotary shaft 403 which is rotatably axially supported by a support base 401 through a bearing 405. The other end of the rotary shaft 403 is coupled to the rotary shaft of a motor 402. The rotational force generated by the motor 402 rotates the bonded substrate stack 101 vacuum-chucked by the substrate holding portion 404. In separating the bonded substrate stack 101, the motor 402 rotates the rotary shaft 403 at a designated rotation speed in accordance with an instruction from a controller (not shown).

The substrate holding portion 406 is coupled to one end of a rotary shaft 408 which is rotatably and slidably axially supported by the support base 401 through a bearings 407. A compression spring 409 is attached to the other end of the rotary shaft 408. With this arrangement, the bonded substrate stack 101 receives a force from the compression spring 409 in a direction (X-axis direction) in which the substrate 101a and the porous layer 101b are spaced apart from each other. Consequently, when the bonded substrate stack 101 is separated into the substrate 101a side and the substrate 101c side by a jet from a jet nozzle 418, the substrate 101a side moves in the X-axis direction to be split from the substrate 101c side.

Before the bonded substrate stack 101 is separated, the

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rotational force of the rotary shaft 403 is transmitted to the substrate holding portion 406 through the bonded substrate stack 101. As a result, the rotary shaft 403, substrate holding portion 404, bonded substrate stack 101, 5 substrate holding portion 406, rotary shaft 408, and compression spring 409 integrally rotate. When the bonded substrate stack 101 is separated into two substrates, the rotary shaft 408 stops.

10 An air cylinder 411 is arranged on the rear end side (in the X-axis direction) of the rotary shaft 408. When the bonded substrate stack 101 is to be held by the substrate holding portions 404 and 406, the air cylinder 411 pushes a piston rod to move the rear end of the rotary shaft 408 in a direction (negative direction of X-axis) in which the 15 compression spring 409 compresses (the state shown in Fig. 13). After the bonded substrate stack 101 is held by suction by the vacuum chucks, the air cylinder 411 retracts the piston rod 410 (moves the piston rod 410 in the X-axis direction) to allow to separate the bonded substrate stack 101. That 20 is, the air cylinder 411 pushes the piston rod 410 in setting the bonded substrate stack 101 between the substrate holding portions 404 and 406 and retracts the piston rod 410 upon completing setting.

To set the bonded substrate stack 101 in the separating 25 apparatus 400, the bonded substrate stack 101 is mounted on a groove portion 412a of an alignment shaft 412 axially

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supported by the support base 401 through bearings 413 and 414, and then, the piston rod 410 is pushed to bring the substrate holding portion 406 into contact with the bonded substrate stack 101, as described above. In this state, the
5 vacuum chucks of the substrate holding portions 404 and 406 are actuated.

Two alignment shafts 412 are preferably arranged along the Y-axis. In this case, only by placing the bonded substrate stack 101 on the two alignment shafts 412, the
10 position of the bonded substrate stack 101 with respect to the three directions, i.e., the X-, Y-, and Z-directions can be defined. This facilitates manual setting of the bonded substrate stack 101. In addition, if a conveyor robot is to be employed, the arrangement of the conveyor robot can be
15 simplified.

To remove the separated substrates, the substrate 101a side moves in the X-axis direction upon completing separation processing to split the two substrates. After this, the two substrates are held by, e.g., the conveyor robot, and the
20 suction by the vacuum chucks of the substrate holding portions 404 and 406 is released.

A high-pressure pump 419 is coupled to the jet nozzle 418. When a high-pressure jet medium is sent from the high-pressure pump 419 to the jet nozzle 418, a jet is ejected
25 from the jet nozzle 418. The pressure applied to the jet medium by the high-pressure pump 419 is controlled by a

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pressure control section 420 in accordance with one of the control procedures shown in Figs. 9 to 11.

A shutter 415 is used to prevent the jet from hitting the bonded substrate stack 101 before the jet pressure
5 reaches a predetermined pressure.

In the separating apparatus 400 as well, the jet pressure is appropriately controlled while the bonded substrate stack is separated. For example, the jet pressure is controlled on the basis of the positional relationship
10 between the jet nozzle 418 and the bonded substrate stack 101 or in accordance with time. The control examples shown in Figs. 9 to 11 can also be applied to the separating apparatus 400.

The arrangement of a separating apparatus according to
15 the third embodiment of the present invention will be described next. Fig. 14 is a view showing the arrangement of the separating apparatus according to the third embodiment of the present invention. In a separating apparatus 200, substrate holding portions 205 hold a bonded substrate stack
20 101 fixed in position, and a jet nozzle 202 is scanned by a horizontal driving mechanism 204 along the separation layer of the bonded substrate stack 101. Vertical position adjustment of the jet nozzle 202 is performed by a vertical driving mechanism 203.

25 In each of the above separating apparatuses, when the strength of the separation region (porous layer) of the



bonded substrate stack is not uniform, the jet pressure is preferably adjusted in accordance with the strength.

Each of the above separating apparatuses can be applied to separate not only a semiconductor substrate such as a
5 bonded substrate stack but also various objects. The object preferably has a fragile structure as a separation layer.

Each of the above separating apparatuses processes one bonded substrate stack. However, when a plurality of bonded substrate stacks are aligned along the planar direction, and
10 the jet nozzle of the separating apparatus is scanned in the planar direction, the plurality of bonded substrate stacks can be separated at once.

Alternatively, when a plurality of bonded substrate stacks are aligned in the axial direction, and a mechanism
15 for scanning the jet nozzle of the separating apparatus in the axial direction is used, the plurality of bonded substrate stacks can be sequentially separated.

Application examples of the above separating apparatuses will be described below. The separating
20 apparatuses are especially suitable to separate a substrate having a fragile layer as a separation layer. As the separation layer, a porous layer, a microcavity layer formed by ion implantation, or a heteroepitaxial layer with distortion or defects concentrated at the crystal lattice
25 can be used. The separation layer may be formed from a plurality of layers having different structures, e.g., a



plurality of layers having different porosities.

(Example 1)

A first p- or n-type single-crystal Si substrate having a size of 6 inches and a resistivity of $0.01 \Omega \cdot \text{cm}$ was anodized
5 in an HF solution to form a porous Si layer (corresponding to the step shown in Fig. 1A). The anodizing conditions were as follows.

Current density: 7 (mA/cm^2)

Anodizing solution: $\text{HF} : \text{H}_2\text{O} : \text{C}_2\text{H}_5\text{OH} = 1 : 1 : 1$

10 Time: 11 (min)

Thickness of porous Si: 12 (μm)

The porous Si is used to form a high-quality epitaxial Si layer on the porous Si layer and also serves as a separation layer. The thickness of the porous Si layer is not limited
15 to the above thickness and is preferably several hundred to $0.1 \mu\text{m}$.

This substrate was oxidized in an oxygen atmosphere at 400°C for 1 hr. Upon oxidation, the inner wall of each pore of the porous Si layer was covered with a thermal oxide film.
20 The surface of the porous Si layer was treated with hydrofluoric acid to remove only the oxide film on the surface of the porous Si layer while leaving the oxide film on the inner wall of each pore. After this, single-crystal Si was epitaxially grown on the porous Si layer to $0.3 \mu\text{m}$ by CVD
25 (Chemical Vapor Deposition). The growing conditions were as follows.



Source gas: $\text{SiH}_2\text{Cl}_2/\text{H}_2$
Gas flow rate: 0.5/180 (l/min)
Gas pressure: 80 (Torr)
Temperature: 950 ($^{\circ}\text{C}$)
5 Growth rate: 0.3 ($\mu\text{m}/\text{min}$)

As an insulating layer, an oxide film (SiO_2 layer) having a thickness of 200 nm was formed on the surface of the epitaxial Si layer by thermal oxidation (corresponding to the step shown in Fig. 1B).

10 The first substrate and a second Si substrate which was independently prepared were stacked and brought into contact with each other such that the surface of the SiO_2 layer of the first substrate opposed the surface of the second substrate. The resultant structure was subjected to a heat
15 treatment at $1,100^{\circ}\text{C}$ for 1 hr to bond the two substrates (corresponding to the step shown in Fig. 1C).

Next, the resultant bonded substrate stack 101 was separated using the separating apparatus 200 shown in Fig. 14 (corresponding to the step shown in Fig. 1D). Details will
20 be described below.

First, as shown in Fig. 14, the bonded substrate stack 101 was sandwiched by the substrate holding portions 205 and vertically held. Pure water was ejected from the jet nozzle 202 having a diameter of 0.15 mm at a pressure of $2,200 \text{ kgf}/\text{cm}^2$.
25 The jet nozzle 202 was scanned, by the horizontal driving mechanism 204, immediately above the bonded substrate stack

101 along the concave portion of the bevelling at a predetermined speed of 10 mm/sec.

While the jet nozzle 202 moved from a portion immediately above the edge of the bonded substrate stack 101 toward the center (in the scanning direction) of the bonded substrate stack 101 by 15 mm, the jet pressure was maintained at 2,200 kgf/cm².

When the jet nozzle 202 passed the position, the jet pressure was continuously reduced. The rate of pressure reduction was -100 kgf/cm² per cm of the moving distance of the jet nozzle 202. When the jet nozzle 202 passed immediately above the center of the bonded substrate stack 101, the jet pressure was 1,600 kgf/cm².

After the jet nozzle 202 passed immediately above the center of the bonded substrate stack 101, the jet pressure was raised at a rate of 100 kgf/cm² per cm of the moving distance of the jet nozzle 202.

After the jet nozzle 202 passed a position 60 mm from a portion immediately above the center of the bonded substrate stack 101, the jet pressure was fixed at 2,200 kgf/cm².

An elastic member 206 (e.g., Viton, perfluoro rubber, or silicone rubber) is preferably placed at the contact portion between the substrate holding portion 205 and the bonded substrate stack 101. In this case, since the bonded substrate stack 101 separated into two substrates can be



easily spaced apart from each other, the jet can be easily injected against the bonded substrate stack 101 supported by the substrate holding portions 205.

With the above process, the bonded substrate stack 101
5 was separated into two substrates at the porous layer as a separation layer.

Various methods can be employed to hold the bonded substrate stack 101. For example, the bonded substrate stack 101 may be held from both sides or held by a substrate holding
10 portion with a vacuum chuck. In the latter case, the bonded substrate stack 101 may be pulled to both sides and held. With this arrangement, the two substrates which are physically separated are immediately split, so damage due to friction between the two substrates can be prevented.

15 As a result, in addition to the SiO_2 layer and epitaxial Si layer formed on the surface of the first substrate, part of the porous Si layer was transferred to the second substrate side. The porous Si layer was left on the surface of the first substrate.

20 When 100 bonded substrate stacks were separated by this method, about 90% of them were satisfactorily separated. On the other hand, when bonded substrate stacks were separated while maintaining a predetermined jet pressure, about 30% of bonded substrate stacks were damaged during processing.

25 The porous Si layer transferred to the second substrate was selectively etched under stirring in a mixed solution





of 49% hydrofluoric acid and 30% hydrogen peroxide
(corresponding to the step shown in Fig. 1E). The
single-crystal Si layer of the second substrate served as
an etch stop layer. The porous Si layer was selectively
5 etched and completely removed.

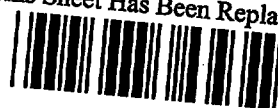
The etching rate of the non-porous Si single crystal
for the above etchant is very low. The selectivity ratio to
the etching rate of the porous layer is 10^5 or more. The
etching amount of the non-porous layer (about several ten
10 Å) is allowable for practical use.

With the above process, an SOI substrate having a 0.2- μm
thick single-crystal Si layer on the Si oxide film was formed.
The thickness of the single-crystal Si layer after the porous
Si layer was selectively etched was measured at 100 points
15 on the entire surface. The thickness was $201 \text{ nm} \pm 4 \text{ nm}$.

Section observation with a transmission electron
microscope revealed that no new crystal defects were formed
in the single-crystal Si layer, and satisfactory
crystallinity was maintained.

20 The resultant structure was subjected to a heat
treatment in hydrogen at $1,100^\circ\text{C}$ for 1 hr, and the surface
roughness was evaluated with an atomic force microscope. The
mean square roughness in a 50- μm square area was about 0.2
nm. This almost equals that of a commercially available Si
25 wafer.

The same result as described above was obtained even



when the oxide film (SiO_2) was formed not on the surface of the epitaxial layer but on the surface of the second substrate or on both surfaces.

5 The porous Si layer left on the first substrate side was selectively etched under stirring in a mixed solution of water, 40% hydrofluoric acid, and 30% hydrogen peroxide. When the resultant structure was subjected to a surface treatment such as hydrogen annealing or surface polishing, the substrate could be recycled as the first or second
10 substrate.

In the above example, one bonded substrate stack was processed. However, when a plurality of bonded substrate stacks are aligned along the planar direction, and the jet nozzle of the separating apparatus is scanned in the planar
15 direction, the plurality of bonded substrate stacks can be separated at once.

Alternatively, when a plurality of bonded substrate stacks are aligned in the axial direction, and a mechanism for scanning the jet nozzle of the separating apparatus in
20 the axial direction is used, the plurality of bonded substrate stacks can be sequentially separated.

In the above example, the jet nozzle was scanned. Instead, the bonded substrate stack may be scanned while the jet nozzle is fixed in position.

25 In the above example, the separating apparatus 200 shown in Fig. 14 was used. The separating apparatus 100 shown in

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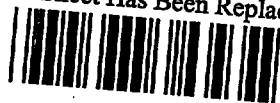


Fig. 3 or the separating apparatus 400 shown in Fig. 13 may be used.

(Example 2)

5 A first p- or n-type single-crystal Si substrate having a resistivity of $0.01 \Omega \cdot \text{cm}$ was subjected to two-step anodizing in an HF solution to form two porous layers (step shown in Fig. 1A). The anodizing conditions were as follows.

<Anodizing of First Step>

Current density: 7 (mA/cm^2)
10 Anodizing solution: $\text{HF} : \text{H}_2\text{O} : \text{C}_2\text{H}_5\text{OH} = 1 : 1 : 1$
Time: 10 (min)
Thickness of first porous Si: $4.5 \mu\text{m}$

<Anodizing of Second Step>

Current density: 20 (mA/cm^2)
15 Anodizing solution: $\text{HF} : \text{H}_2\text{O} : \text{C}_2\text{H}_5\text{OH} = 1 : 1 : 1$
Time: 2 (min)
Thickness of second porous Si: $2 \mu\text{m}$

A porous Si layer having a two-layered structure was formed. The surface porous Si layer anodized at a low current
20 first was used to form a high-quality epitaxial Si layer, and the lower porous Si layer (layer with high porosity) anodized at a high current was used as a separation layer, so the functions of the two layers were separated. The thickness of the porous Si layer formed at a low current is
25 not limited to the above thickness ($4.5 \mu\text{m}$) and is suitably several hundred to $0.1 \mu\text{m}$. The thickness of the porous Si

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layer formed at a high current is also not limited to the above thickness ($2\text{ }\mu\text{m}$) as far as a thickness at which the bonded substrate stack can be separated by jet is ensured.

After formation of the second porous Si layer, a third
5 layer or more layers having different porosities may be formed.

This substrate was oxidized in an oxygen atmosphere at 400°C for 1 hr. Upon oxidation, the inner wall of each pore of the porous Si layer was covered with a thermal oxide film.

10 The surface of the porous Si layer was treated with hydrofluoric acid to remove only the oxide film on the surface of the porous Si layer while leaving the oxide film on the inner wall of each pore. After this, single-crystal Si was epitaxially grown on the porous Si layer to $0.3\text{ }\mu\text{m}$ by CVD
15 (Chemical Vapor Deposition). The growing conditions were as follows.

	Source gas:	$\text{SiH}_2\text{Cl}_2/\text{H}_2$
	Gas flow rate:	$0.5/180\text{ (l/min)}$
	Gas pressure:	80 (Torr)
20	Temperature:	$900\text{ (}^\circ\text{C)}$
	Growth rate:	$0.3\text{ (}\mu\text{m/min)}$

As an insulating layer, an oxide film (SiO_2 layer) having a thickness of 200 nm was formed on the surface of the epitaxial Si layer by thermal oxidation (corresponding to
25 the step shown in Fig. 1B).

The first substrate and a second Si substrate which was

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independently prepared were stacked such that the surface of the SiO_2 layer opposed the surface of the second substrate. The resultant structure was subjected to a heat treatment at $1,100^\circ\text{C}$ for 1 hr to bond the two substrates (corresponding to the step shown in Fig. 1C).

Next, the resultant bonded substrate stack 101 was separated using the separating apparatus 400 shown in Fig. 13 (corresponding to the step shown in Fig. 1D). Details will be described below.

First, the bonded substrate stack 101 was held vertically between the substrate holding portions 404 and 406, and the piston rod 410 was pushed to bring the substrate holding portion 406 into contact with the bonded substrate stack 101. In this state, the vacuum chucks of the substrate holding portions 404 and 406 were actuated. The distance between the bonded substrate stack 101 and the jet nozzle 418 is preferably 10 to 30 mm. In this example, the distance was set to be 15 mm.

Subsequently, water as a jet medium was sent from the high-pressure pump 419 to the jet nozzle 418, and stabilization of the jet was waited. The jet pressure was set to be 500 kgf/cm^2 under the control of the pressure control section 420. When the jet stabilized, the shutter 415 was opened to inject the jet against the concave portion of the bevelling of the bonded substrate stack 101. At this time, the substrate holding portion 404 was rotated by the motor



402 to rotate the bonded substrate stack 101.

Separation processing was performed by controlling the high-pressure pump 419 in accordance with the procedure shown in Fig. 9. First, separation processing was started while
5 maintaining the jet pressure at 500 kgf/cm². For a period of 20 to 80 sec after the start of separation processing, separation processing was continued while maintaining the jet pressure at 200 kgf/cm². For a period of 80 to 100 sec after the start of separation processing, the unseparated
10 portion was separated at a higher jet pressure of 400 kgf/cm².

With the above processing, the bonded substrate stack 101 was separated into two substrates by an appropriate separation force without being damaged.

According to the separating apparatus 400, since the
15 two substrates separated from the bonded substrate stack 101 are immediately split from each other, the two substrates after separation are unlikely to be damaged due to friction.

Although the method of separating the bonded substrate stack without rotating it but instead scanning the jet
20 nozzle, as in the separating apparatus 200 shown in Fig. 14, is effective, the bonded substrate stack can be separated using a low-pressure jet when the bonded substrate stack 101 is separated while being rotated, as in the separating apparatus 400 shown in Fig. 13. For example, when the
25 separating apparatus 200 shown in Fig. 14 uses a jet nozzle 202 having a diameter of 0.15 mm, a jet having a pressure



in the neighborhood of 2,000 kgf/cm² is required. However, in the separating apparatus 400 shown in Fig. 13, the bonded substrate stack can be separated using a jet having a pressure of several hundred kgf/cm². This is because when the jet is
5 injected against the central portion of the bonded substrate stack 101, the separation force efficiently acts on the internal portion of the bonded substrate stack 101.

The following effects can be obtained by reducing the jet pressure.

- 10 1) Break or damage to the bonded substrate stack can be prevented.
- 2) Since the high-pressure pump has sufficient capability, a plurality of jet nozzles can be simultaneously used.
- 3) The high-pressure pump can be made compact and
15 lightweight.
- 4) Since the degree of freedom in selection of the material of the high-pressure pump or pipe system increases, a material suitable for the type of jet medium (e.g., pure water) can be selected.
- 20 5) Since the noise of high-pressure pump or jet ejection decreases, soundproofing measures can be easily taken.

In the separating apparatus 400 shown in Fig. 13, only the substrate holding portion 404 side is driven, so a twisting force may act on the bonded substrate stack 101.
25 If this force poses a problem, another motor is preferably coupled to the substrate holding portion 406 side to drive



the substrate holding portions 404 and 406 in synchronism with each other.

After the bonded substrate stack 101 was separated, the porous Si layer transferred to the second substrate was
5 selectively etched under stirring in a mixed solution of 49% hydrofluoric acid and 30% hydrogen peroxide (corresponding to the step shown in Fig. 1E). The single-crystal Si layer served as an etch stop layer. The porous Si layer was selectively etched and completely removed.

10 The etching rate of the non-porous Si single crystal for the above etchant is very low. The selectivity ratio to the etching rate of the porous layer is 10^5 or more. The etching amount of the non-porous layer (about several ten Å) is allowable for practical use.

15 With the above process, an SOI substrate having a 0.2- μ m thick single-crystal Si layer on the Si oxide film was formed. The thickness of the single-crystal Si layer after the porous Si layer was selectively etched was measured at 100 points on the entire surface. The thickness was $201 \text{ nm} \pm 4 \text{ nm}$.

20 Section observation with a transmission electron microscope revealed that no new crystal defects were formed in the single-crystal Si layer, and satisfactory crystallinity was maintained.

The resultant structure was subjected to a heat
25 treatment in hydrogen at 1,100°C for 1 hr, and the surface roughness was evaluated with an atomic force microscope. The



mean square roughness in a 50- μ m square area was about 0.2 nm. This almost equals that of a commercially available Si wafer.

5 The same result as described above was obtained even when the oxide film (SiO_2) was formed not on the surface of the epitaxial layer but on the surface of the second substrate or on both surfaces.

10 The porous Si layer left on the first substrate side was selectively etched under stirring in a mixed solution of water, 40% hydrofluoric acid, and 30% hydrogen peroxide. When the resultant structure was subjected to a surface treatment such as hydrogen annealing or surface polishing, the substrate could be recycled as the first or second substrate.

15 Even when the separating apparatus 100 shown in Fig. 3 was used as a separating apparatus, the same effects as described above were obtained.

Even when the separating apparatus 200 shown in Fig. 14 was employed, a satisfactory substrate was manufactured.
20 (Example 3)

An oxide film (SiO_2 layer) having a thickness of 400 nm was formed on the surface of a first single-crystal Si substrate by thermal oxidation as an insulating layer. Next, ions were implanted from the surface of the first substrate
25 such that the projection range stayed in the Si substrate. With this process, a layer serving as a separation layer was

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formed as a distortion layer due to a microcavity layer or a heavily doped ion species layer at a depth corresponding to the projection range.

The first substrate and a second Si substrate which was
5 independently prepared were brought into contact such that the surface of the SiO₂ layer opposed the surface of the second substrate. The resultant structure was subjected to a heat treatment at 600°C for 10 hrs to bond the two substrates. Before the two substrates were bonded, the substrates were
10 treated by N₂ plasma or the like to increase the bonding strength.

The resultant bonded substrate stack 101 was separated using the separating apparatus 400 shown in Fig. 13. Separation processing was executed under the same conditions
15 as those in Example 2 except that the jet pressure was raised by about 150 kgf/cm² throughout processing.

As a result, in addition to the SiO₂ layer and surface single-crystal layer which were formed on the surface of the first substrate to have a thickness of about 500 nm, part
20 of the separation layer was transferred to the second substrate side. The separation layer was left on the surface of the first substrate.

The separation surface of the separated second substrate was polished by about 300 nm by a CMP (Chemical
25 Mechanical Polishing) apparatus to remove the damage layer formed upon ion implantation and separation processing and

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was planarized.

With the above process, an SOI substrate having a 0.2- μm thick single-crystal Si layer on the Si oxide film was formed. The thickness of the single-crystal Si layer was measured
5 at 100 points on the entire surface. The thickness was 201 nm $\pm$ 4 nm.

The resultant structure was subjected to a heat treatment in hydrogen at 1,100°C for 1 hr, and the surface roughness was evaluated with an atomic force microscope. The
10 mean square roughness in a 50- μm square area was about 0.3 nm. This almost equals that of a commercially available Si wafer.

In this example, the surface region of the single-crystal Si substrate (first substrate) is transferred to the
15 second substrate via the separation layer formed by ion implantation. Alternatively, an epitaxial wafer may be used. A separation layer is formed at the lower portion of the epitaxial layer by ion implantation, and the epitaxial layer is transferred to the second substrate by separating
20 the substrate at the separation layer.

In the above example, after formation of the separation layer by ion implantation, the SiO₂ layer on the surface of the first substrate may be removed. After an epitaxial layer and an SiO₂ layer are formed, the first substrate is bonded
25 to the second substrate, and the substrate is separated at the separation layer, thereby transferring the epitaxial

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layer and SiO₂ layer to the second substrate.

Even when the separating apparatus 100 shown in Fig. 3 was used as a separating apparatus, the same effects as described above were obtained.

5 Even when the separating apparatus 200 shown in Fig. 14 was employed, a satisfactory substrate was manufactured. (Example 4)

An SOI substrate was manufactured by the same method as in Example 2 except that separation processing was
10 executed while scanning the jet nozzle on the separation region of the bonded substrate stack.

A substrate 101 formed by the same method as in Example 2 was separated using the separating apparatus shown in Fig. 3. Details will be described below.

15 The bonded substrate stack 101 was vertically held between the substrate holding portions 108 and 109 while being pressed by the substrate holding portion 109. The distance between the bonded substrate stack 101 and the jet nozzle 112 is preferably 10 to 30 mm. In this example, the
20 distance was set to be 15 mm.

In this example, the jet pressure was controlled on the basis of the control example shown in Fig. 10. First, the jet nozzle 112 was retreated to a position where the jet ejected from the jet nozzle 112 did not hit the bonded
25 substrate stack 101 (the jet nozzle 112 may be retreated in advance). A jet medium was sent from the high-pressure pump



115 to the jet nozzle 112 to eject a jet from the jet nozzle 112, and at the same time, scanning of the jet nozzle 112 was started (section Y1). In the section Y1, the jet pressure was raised to 500 kgf/cm².

5 In the section Y2, the peripheral portion of the bonded substrate stack 101 was mainly separated while maintaining the jet pressure at 500 kgf/cm². By separating the peripheral portion of the bonded substrate stack 101 using a high-pressure jet, the separation force can be efficiently applied
10 to the bonded substrate stack 101.

In this example, the section Y3 was set for 10 to 90 mm from the edge portion of the bonded substrate stack 101, i.e., the portion where separation was started (to be referred to as a separation start position). In the section
15 Y3, the jet pressure was set to be 200 kgf/cm². By setting the jet pressure to be relatively low in the section Y3, damage to the bonded substrate stack 101 can be prevented.

Also, in this example, the section Y4 was set for 90 to 100 mm from the separation start position of the bonded
20 substrate stack 101. In this section, the jet pressure was raised to 400 kgf/cm² to separate the unseparated portion. When the jet pressure is raised in separating the central portion of the bonded substrate stack 101, an appropriate separation force can be applied to the internal portion of
25 the bonded substrate stack 101. For this reason, the bonded substrate stack 101 can be efficiently separated while



preventing damage to it.

In the section Y5, the jet pressure was gradually reduced.

Even when the separating apparatus 400 shown in Fig. 13 was used as a separating apparatus, the same effects as described above were obtained.

Even when the separating apparatus 200 shown in Fig. 14 was employed, a satisfactory substrate was manufactured. (Example 5)

10 This example is the same as in Example 4 except that the jet pressure was controlled on the basis of the control example shown in Fig. 11.

The bonded substrate stack 101 formed by the same method as in Example 2 was vertically supported between the 15 substrate holding portions 108 and 109 while being pressed by the substrate holding portion 109. The distance between the bonded substrate stack 101 and the jet nozzle 112 is preferably 10 to 30 mm. In this example, the distance was set to be 15 mm.

20 In this example, the jet pressure was controlled on the basis of the control example shown in Fig. 11. First, the jet nozzle 112 was retreated to a position where the jet ejected from the jet nozzle 112 did not hit the bonded substrate stack 101 (the jet nozzle 112 may be retreated in 25 advance). A jet medium was sent from the high-pressure pump 115 to the jet nozzle 112 to eject a jet from the jet nozzle



112, and at the same time, scanning of the jet nozzle 112 was started (section Y1). In the section Y1, the jet pressure was raised to 500 kgf/cm².

5 In the section Y2, the peripheral portion of the bonded substrate stack 101 was mainly separated while maintaining the jet pressure at 500 kgf/cm². By separating the peripheral portion of the bonded substrate stack 101 using a high-pressure jet, the separation force can be efficiently applied to the bonded substrate stack 101.

10 In this example, the section Y3 was set for 10 to 60 mm from the separation start position of the bonded substrate stack 101. In the section Y3, separation processing was executed while reducing the jet pressure from 500 kgf/cm² to 200 kgf/cm². By separating the bonded substrate stack while
15 gradually reducing the jet pressure in the section Y3, damage to the stack can be prevented.

In this example, the section Y4 was set for 60 to 90 mm from the separation start position, and separation processing was executed while gradually raising the jet
20 pressure to 400 kgf/cm². The reason why the jet pressure is gradually raised in the section Y4 is as follows. As the separation region comes close to the central portion of the bonded substrate stack 101, the discharge paths of the jet medium injected into the bonded substrate stack 101 increase,
25 and accordingly, the separation force acting on the internal portion of the bonded substrate stack 101 weakens. To



efficiently perform separation processing, the separation force must be compensated.

In this example, the section Y5 was set for 90 to 100 mm from the separation start position of the bonded substrate stack 101. In this section, the unseparated portion was separated while raising the jet pressure to 400 kgf/cm². When the jet pressure is raised in separating the central portion of the bonded substrate stack 101, an appropriate separation force can be applied to the internal portion of the bonded substrate stack 101, so the bonded substrate stack 101 can be efficiently separated while preventing damage to it.

In the section Y6, the jet pressure was gradually reduced.

Even when the separating apparatus 400 shown in Fig. 13 was used as a separating apparatus, the same effects as described above were obtained.

Even when the separating apparatus 200 shown in Fig. 14 was employed, a satisfactory substrate was manufactured.

As described above, by appropriately changing the jet pressure in accordance with the progress of separation processing, damage to the object to be separated can be prevented while efficiently performing separation processing.

According to the separating apparatus or method, a high-quality semiconductor substrate can be manufactured at a high yield.

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According to the present invention, an object can be efficiently separated and split.

In addition, according to the present invention, a satisfactory semiconductor substrate can be manufactured.

5 The present invention is not limited to the above embodiments and various changes and modifications can be made within the spirit and scope of the present invention. Therefore, to apprise the public of the scope of the present invention, the following claims are made.

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WHAT IS CLAIMED IS:

1. A separating apparatus for separating an object comprising:

jet means having a fluid jet unit for ejecting a jet
5 of fluid to the object to be separated to separate the object;
and

control means for changing a condition for ejecting the
fluid from the jet unit in accordance with progress of object
separation processing.

10 2. The apparatus according to claim 1 further comprising
a holding portion for holding the object to be separated.

3. The apparatus according to claim 2 wherein the object
to be separated has a plate shape, and the holding portion
holds the plate-like object to inject the fluid against the
15 plate-like object in a planar direction thereof.

4. The apparatus according to any one of claims 1 to 3,
wherein said control means changes a pressure of the fluid
to be ejected from the jet unit in accordance with progress
of object separation processing.

20 5. The apparatus according to claim 4, wherein said control
means controls said jet means to set the pressure of the fluid
to be relatively high in separating a vicinity of a peripheral
portion of a separation surface of the object to be separated.

6. The apparatus according to claim 4, wherein said control
25 means controls said jet means to set the pressure of the fluid
to be relatively high in separating a vicinity of a central





- portion of a separation surface of the object to be separated.
7. The apparatus according to claim 4, wherein said control means controls said jet means to set the pressure of the fluid to be relatively high in separating vicinities of peripheral and central portions of a separation surface of the object to be separated and controls said jet means to set the pressure of the fluid to be relatively low in separating an intermediate portion between the vicinities of the peripheral and the central portions.
8. The apparatus according to any one of claims 1 to 7, wherein said control means changes the condition for ejecting the fluid from the jet unit in accordance with a relative positional relationship between the jet unit and the object to be separated.
9. The apparatus according to any one of claims 1 to 7, wherein said control means changes the condition for ejecting the fluid from the jet unit as a function of time.
10. The apparatus according to any one of claims 1 to 9 further comprising scanning means for scanning the jet unit.
11. The apparatus according to any one of claims 1 to 9, further comprising rotating means for rotating the object about an axis perpendicular to a separation surface of the object to be separated.
12. The apparatus according to any one of claims 1 to 11, wherein the object to be separated has a fragile layer as a separation layer.

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13. The apparatus according to claim 12, wherein the fragile layer is a porous layer.
14. The apparatus according to claim 12, wherein the fragile layer is a layer having a microcavity.
- 5 15. A separating method of separating an object by ejecting a jet of fluid from a jet unit to the object to be separated, comprising:
- the control step of changing a condition for ejecting the fluid from the jet unit in accordance with progress of
- 10 object separation processing.
16. The method according to claim 15, wherein the object to be separated has a plate shape and is separated by injecting the fluid against the plate-like object in a planar direction thereof.
- 15 17. The method according to claim 15 or 16, wherein said control step comprises changing a pressure of the fluid to be ejected from the jet unit in accordance with progress of object separation processing.
18. The method according to claim 17, wherein said control
- 20 step comprises setting the pressure of the fluid to be ejected from the jet unit to be relatively high in separating a vicinity of a peripheral portion of a separation surface of the object to be separated.
19. The method according to claim 17, wherein said control
- 25 step comprises setting the pressure of the fluid to be ejected from the jet unit to be relatively high in separating a

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vicinity of a central portion of a separation surface of the object to be separated.

20. The method according to claim 17, wherein said control step comprises setting the pressure of the fluid to be ejected
5 from the jet unit to be relatively high in separating vicinities of peripheral and central portions of a separation surface of the object to be separated and setting the pressure of the fluid to be ejected from the jet unit to be relatively low in separating an intermediate portion between the
10 vicinities of the peripheral and the central portions.

21. The method according to any one of claims 15 to 20, wherein said control step comprises changing the condition for ejecting the fluid from the jet unit in accordance with a relative positional relationship between the jet unit and
15 the object to be separated.

22. The method according to any one of claims 15 to 20, wherein said control step comprises changing the condition for ejecting the fluid from the jet unit as a function of time.

20 23. The method according to any one of claims 15 to 22, further comprising executing separation processing for the object while scanning the jet unit along the separation surface of the object to be separated.

24. The method according to any one of claims 15 to 23,
25 further comprising executing separation processing for the object while rotating the object about an axis perpendicular





to the separation surface of the object to be separated.

25. The method according to any one of claims 15 to 23, wherein the object to be separated has a fragile layer as a separation layer.

5 26. The method according to claim 25, wherein the fragile layer is a porous layer.

27. The method according to claim 25, wherein the fragile layer is a layer having a microcavity.

28. A separating method comprising separating an object
10 using said separating apparatus of any one of claims 1 to 14.

29. A method of manufacturing a semiconductor substrate, comprising the steps of:

preparing a substrate having a separation layer; and
15 separating the object at the separation layer using the method of any one of claims 15 to 28.

30. A method of manufacturing a semiconductor substrate, comprising the steps of:

forming a first substrate in which a porous layer and
20 a non-porous layer are sequentially formed on one surface;
bonding the first substrate to a second substrate via the non-porous layer to form a bonded substrate stack; and
separating the bonded substrate stack into two substrates while ejecting a jet of fluid to the porous layer
25 of the bonded substrate stack,

wherein the separation step comprises changing a





condition for ejecting the fluid in accordance with progress of the separation step.

31. A method of manufacturing a semiconductor substrate, comprising the steps of:

5 implanting ions from a surface of a substrate consisting of a single-crystal semiconductor to a predetermined depth, thereby forming a first substrate in which a microcavity layer is formed;

 bonding a second substrate to a front surface side of
10 the first substrate to form a bonded substrate stack; and

 separating the bonded substrate stack into two substrates while ejecting a jet of fluid to the microcavity layer of the bonded substrate stack,

 wherein the separation step comprises changing a
15 condition for ejecting the fluid in accordance with progress of the separation step.

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