

DESCRIPTION

TITLE: METHOD FOR PREPARING A THIN LAYER OF FERROELECTRIC MATERIAL

FIELD OF THE INVENTION

The present invention relates to a method for preparing a thin layer of ferroelectric material. More specifically, the invention relates to a preparation method that allows the single-domain character of the ferroelectric material to be maintained in the thin layer of the final product. This preparation method is used, for example, in the fields of microelectronics, micromechanics, photonics, etc.

TECHNOLOGICAL BACKGROUND OF THE INVENTION

By way of an introduction and reminder, a ferroelectric material is a material with electrical polarization in the natural state, with this polarization being able to be reversed by applying an external electric field. The ferroelectric domain refers to each continuous region of the material in which the polarization is uniform (all the dipole moments are aligned parallel to each other in a given direction). A ferroelectric material therefore can be characterized as "single-domain" in the event that this material is made up of a single region in which the polarization is uniform or can be characterized as "multi-domain" in the event that the ferroelectric material comprises a plurality of regions having polarities that may be different.

The present invention more specifically relates to the preparation of a thin ferroelectric layer obtained by applying Smart Cut™ technology, according to which a thin layer is removed from a solid substrate of ferroelectric material by fracturing

in the vicinity of a fragile zone (or fragilization plane) formed in the solid substrate by implanting "light" species, such as helium or hydrogen. A specific example of the implementation of this method can be found in document EP 3646374 B1.

According to this method, and after the step of removing the layer, it is often necessary to apply treatments thereto in order to improve its surface finish, its crystalline quality or to modify its thickness. However, the applicant has observed that these preparation steps, when they were applied to a thin ferroelectric layer attached to a silicon support, can lead to the formation of a plurality of ferroelectric domains within the thin layer, thus attributing a multi-domain character thereto.

Such a characteristic makes the layer unsuitable for use, as it affects the performance capabilities of devices that are to be formed on/in the thin layer, such as surface acoustic wave (SAW) devices, for example.

Document WO 2020/200986 discloses that the formation of ferroelectric domains in a surface portion of the layer is caused by the existence of hydrogen concentration gradients in the thin layer when applying a heat treatment. This hydrogen can notably correspond to the light species implanted in the solid substrate in order to form the fragilization zone therein from which the thin layer can be removed. The thickness of the surface portion can be of the order of 150 nm to 200 nm or more. In order to permanently restore the single-domain character of the thin layer, this document recommends thinning the thin ferroelectric layer after applying such a heat treatment.

This thinning notably can be carried out by chemical-mechanical polishing of the layer, but removing a relatively large thickness of materials by polishing tends to deteriorate the thickness

uniformity of this layer. By way of an example, removing a thickness of the order of 400 nm leads to the formation of a layer with a thickness uniformity (i.e., the difference between the largest thickness and the lowest thickness when this thickness measurement is carried out, for example, by reflectometry or ellipsometry, at multiple measurement points over the entire extent of the layer) that is of the order of 100 nm. This variability in thickness is not acceptable, as it does not allow devices with all the required characteristics to be collectively manufactured from such a layer.

There are alternatives to thinning using chemical-mechanical polishing. Notably, thinning the thin ferroelectric film by ion etching can be contemplated, for example, by Reactive Ion Etching (RIE). RIE is a type of dry etching that uses a plasma of chemically reactive ions to remove the surface material from a wafer. The plasma is generated under low pressure by an electromagnetic field. The high-energy ions of the plasma attack the surface of the layer and react therewith in order to pulverise it, and thus gradually thin said layer. Such an approach is notably described in the document filed under number FR 2111960.

However, and irrespective of whether it is carried out by chemical-mechanical polishing or by ion etching, removing a relatively large thickness of material in order to remove the multi-domain surface portion of the removed layer is a disadvantage of the methods of the prior art, as this step tends to make the manufacturing method longer and more complex to implement.

This elimination step also requires the removal of a layer with a relatively large thickness from the solid substrate of ferroelectric material, which requires the light species to be

implanted with a significant amount of energy. Beyond a certain threshold thickness, the energy required to define the removed layer is beyond the reach of existing implantation equipment. A method for preparing a thin single-domain layer of ferroelectric material of the prior art therefore has limitations that would be beneficial to overcome.

SUBJECT MATTER OF THE INVENTION

An aim of the invention is to propose a method for preparing a thin layer of ferroelectric material that at least partly addresses some of the aforementioned limitations. More specifically, an aim of the invention is to propose a method for preparing a thin layer of ferroelectric material that is simpler to implement than the methods of the prior art. Another aim of the invention is to propose a method for thinning a thin layer obtained by detaching a donor substrate at a fragilization plane, in which method the thickness of materials removed in order to remove the multi-domain surface portion of the removed layer is lower than the thickness removed in the methods of the prior art.

BRIEF DESCRIPTION OF THE INVENTION

With a view to achieving one of these aims, the subject matter of the invention proposes a method for preparing a thin single-domain layer of ferroelectric material, the method comprising:

- a step of implanting "light" species in a first face of a ferroelectric donor substrate in order to form a fragilization plane and to define a first layer between the fragilization plane and the first face of the donor substrate;

- a step of assembling the first face of the donor substrate on a support in order to form an intermediate assembly;
- a step of fracturing the intermediate assembly, comprising a first heat treatment, with this step leading to the fracturing of the donor substrate at the fragilization plane and to the formation of a free face of the first layer;
- a sequence for finishing the first layer comprising an annealing step including a second heat treatment and, after the annealing step, a step of thinning the first layer in order to form the thin single-domain layer.

According to the invention, the preparation method comprises, between the fracturing step and the finishing sequence, applying a treatment to the free face in order to produce a hydrogen concentration that is greater than 2.0×10^{21} at/cm³ in a surface thickness of the first layer.

According to other advantageous and non-limiting features of the invention, taken individually or according to any technically feasible combination:

- the surface thickness, in which the hydrogen concentration is greater than 2.0×10^{21} at/cm³, is greater than or equal to 100 nm;
- the surface thickness is greater than or equal to 200 nm;
- the treatment of the free face introduces a hydrogen dose into the first layer that is greater than or equal to 2.0×10^{16} at/cm²;
- the treatment of the free face comprises immersing the first layer in a first solution having a temperature higher than ambient temperature;

- the first solution comprises SC1 and/or SC2;
- the temperature of the first solution is greater than 50 °C, preferably greater than 65 °C;
- the treatment of the free face comprises a sequence of cleaning steps using a second solution, with the cleaning steps of the sequence being separated from one another by a waiting time of 24 hours or more;
- the second solution comprises deionized water, the cleaning steps simultaneously comprising brushing and dispensing deionized water onto the free face of the first layer;
- the second solution is at ambient temperature;
- the sequence of cleaning steps comprises at least three cleaning steps, preferably at least five cleaning steps;
- the treatment of the free face comprises depositing a covering layer onto the free face with a hydrogen concentration that is greater than 1.0×10^{20} at/cm³;
- the covering layer comprises silicon dioxide, silicon nitride or silicon oxynitride;
- the covering layer is 20 nm or more thick;
- the thin single-domain layer is made up of a single-crystal piezoelectric material, such as lithium tantalate or lithium niobate;
- the thin single-domain layer is made up of lithium niobate;

- the assembly step comprises forming a dielectric interlayer on the first face of the donor substrate and/or on the support.

BRIEF DESCRIPTION OF THE DRAWINGS

Further features and advantages of the invention will become apparent from the following detailed description of the invention, which is provided with reference to the appended figures, in which:

[Fig. 1]

Figure 1 shows a step of preparing a thin layer according to a first embodiment;

[Fig. 2]

Figure 2 shows a step of preparing a thin layer according to a second embodiment;

[Fig. 3]

Figure 3 is a graph of an analysis of a removed layer;

[Fig. 4]

Figure 4 shows a preparation method according to the invention.

DETAILED DESCRIPTION OF THE INVENTION

The invention relates to a method for preparing a thin single-domain layer 3 of a ferroelectric material transferred from a single-crystal donor substrate 1 onto a support substrate 7 using a transfer technique including implanting light species in a

donor substrate 1. Several embodiments exist for this step of providing a thin layer.

According to a first embodiment, shown in figures 1A to 1F, the donor substrate 1 is made up of a solid, single-crystal and single-domain block of ferroelectric material, for example, LiTaO₃, LiNbO₃, LiAlO₃, BaTiO₃, PbZrTiO₃, KNbO₃, BaZrO₃, CaTiO₃, PbTiO₃ or KTaO₃. The donor substrate 1 can assume the form of a standardized size circular wafer, for example, with a diameter of 150 mm or 200 mm. However, the invention is by no means limited to these sizes or to this shape. The donor substrate may have been removed from an ingot of ferroelectric materials in such a way as to form the donor substrate 1 with a predetermined crystal orientation. The orientation is selected as a function of the intended application. Thus, an orientation ranging between 30° and 60°RY, or between 40° and 50°RY, is usually selected when the intention is to use the properties of a thin layer of LiTaO₃ in order to form an SAW filter. However, the invention is by no means limited to a particular crystal orientation.

Irrespective of the crystal orientation of the donor substrate 1, the method comprises introducing at least one "light" species into the donor substrate 1, notably species selected from inert gases or hydrogen. This introduction can correspond to an implantation, i.e., ion bombardment of a flat face 4 of the donor substrate 1 by light species such as hydrogen and/or helium ions.

In a manner known per se, and as shown in figure 1B, the implanted ions are intended to form a fragilization plane 2 delimiting a first layer 3 of ferroelectric material to be transferred that is located on the side of the flat face 4, and another part 5 forming the rest of the substrate.

The type and dose of the implanted species and the implantation energy are selected as a function of the thickness of the layer to be transferred and of the physico-chemical properties of the donor substrate. In the case of a donor substrate 1 made of LiTaO_3 , implanting a hydrogen dose ranging between 10^{16} and 5×10^{17} at/cm² with an energy level ranging between 30 and 300 keV thus can be selected in order to delimit a first layer 3 of the order of 200 to 2,000 nm.

In a subsequent step, shown in figure 1C, the flat face 4 of the donor substrate 1 is assembled with a face 6 of a support substrate 7. The support substrate 7 can assume the same size and shape as the donor substrate 1. For availability and cost reasons, the support substrate 7 is a monocrystalline or polycrystalline silicon wafer. However, more generally, the support substrate 7 can be made up of any material, for example, silicon, sapphire or glass, and can assume any shape.

In a particular embodiment, the support substrate comprises a base substrate 7b, for example, made of single-crystal silicon, on which a charge trapping layer 7a is arranged. The base substrate 7b can have high resistivity, greater than 1,000 ohms.cm or, more conventionally, less than 1,000 ohms.cm. The charge trapping layer 7a, as is well known per se, can be formed by a polycrystalline silicon layer, and typically can have a thickness ranging between 500 nm and 10 microns.

Prior to the assembly step, the faces of the substrates to be assembled may be prepared by a step of cleaning, brushing, drying, polishing or plasma activation, for example.

The assembly step can involve bringing the donor substrate 1 into close contact with the support substrate 7 by molecular adhesion and/or electrostatic bonding. Optionally, in order to

facilitate the assembly of the two substrates 1, 7, notably when they are assembled by direct bonding, at least one amorphous interlayer can be formed prior to assembly, either on the flat face 4 of the donor substrate 1, or on the flat face 6 of the support substrate 7 to be assembled, or on both. This interlayer is made up of silicon oxide, silicon nitride or silicon oxynitride, for example. Its thickness can range between a few nanometres and a few microns.

An interlayer with a low concentration of hydrogen or forming a barrier to hydrogen diffusion is preferred in order to follow the teaching of document W0 2020/200986 and thus avoid the formation of a multi-domain zone at the interface between the first layer 3 and the amorphous interlayer, on the side of the second face of the first layer 3. The interlayer can be produced using various known techniques of the prior art, such as thermal oxidation or nitridation treatments, chemical deposition (PECVD, LPCVD, etc.), etc.

On completion of this assembly step, an assembly comprising the two associated substrates is available, with the flat face 6 of the support substrate 7 adhering to the flat face 4 of the donor substrate 1.

The assembly is then treated in order to detach the first layer 3 of ferroelectric material from the donor substrate 1, for example, by cleavage at the fragilization plane 2.

This detachment step thus can involve applying a heat treatment to the assembly in a temperature range of the order of 80 °C to 300 °C in order to allow the first layer 3 to be transferred to the support substrate 7. As an alternative to or in addition to the heat treatment, this step can include applying a blade or a jet of gaseous or liquid fluid to the fragilization plane 2.

Following this detachment step, the structure 9 shown in figure 1D is obtained. This structure 9 comprises the first layer 3 of single-crystal ferroelectric material comprising a first free face 8 and a second face 4 arranged on the support substrate 7.

Figures 2A to 2F show a second embodiment leading to the provision of this same structure 9. This second method is particularly suitable for producing a heterogeneous structure 9, in which the first layer 3 has a thermal expansion coefficient (in the main plane defining this layer) that is very different to that of the support 7, for example, having a difference of more than 10 % (at ambient temperature).

This second embodiment mainly differs from the first embodiment in terms of the nature of the donor substrate 1. For the sake of conciseness, therefore, only the elements of this second embodiment that differ from the first embodiment are described herein, all the other features of the first embodiment therefore can be provided.

With reference to figure 2A, the donor substrate 1 in this case is made up of a thick layer of ferroelectric material 1a with the same properties as those described for the solid block of ferroelectric materials in relation to the first embodiment, and a handling substrate 1b.

The handling substrate 1b is advantageously made up of a material (or a plurality of materials) conferring a thermal expansion coefficient thereto that is close to that of the support substrate 7. The term "close" means that the difference between the thermal expansion coefficient of the handling substrate 1b and that of the support is less, as an absolute value, than the

difference between the thermal expansion of the solid block of ferroelectric material and that of the support substrate 7.

Preferably, the handling substrate 1b and the support substrate have an identical thermal expansion coefficient. During the assembly of the donor substrate 1 and of the support 7, an assembly is formed that is capable of withstanding a heat treatment at a relatively high temperature. For the sake of ease of implementation, this can be obtained by selecting the handling substrate 1b so that it is made up of the same material as that of the support substrate 7.

In order to form the donor substrate 1 of this embodiment, a solid block of ferroelectric material is assembled with the handling substrate 1a beforehand, for example, by means of a molecular adhesion bonding technique, as described above, or using an adhesive layer. Next, the layer of ferroelectric material 1a is formed by thinning, for example, by grinding and/or chemical-mechanical polishing and/or etching. Before assembly, the formation of an adhesion layer (for example, by silicon oxide and/or silicon nitride deposition of an adhesive layer, for example, a polymer) on one and/or the other of the faces that have been brought into contact may have been contemplated. Assembly can comprise applying a low-temperature heat treatment (ranging, for example, between 50 and 300 °C, typically 100 °C) allowing the bonding energy to be sufficiently enhanced to allow the following thinning step.

The handling substrate 1b is selected so as to have a thickness that is substantially equivalent to that of the support substrate 7. The thinning step is conducted so that the thickness of the thick layer 1a is low enough for the stresses generated during the heat treatments applied throughout the remainder of the method to be of reduced intensity. At the same time, this

thickness is high enough to be able to remove the first layer 3, or a plurality of such layers, therefrom. This thickness can range, for example, between 5 and 400 microns.

The following steps of the method of this second embodiment are equivalent to those of the steps described in the first embodiment. Light species are implanted within the thick layer 1a in order to generate a fragilization plane 2 that demarcates the separation of the thin layer 3 from the rest 5 of the donor substrate 1, as shown in figure 2B. This step is followed by a step of assembling the donor substrate 1 on the support substrate 7, as shown in figure 2C. Next, the first layer 3 is detached from the rest of the substrate 5 in order to obtain the structure 9 shown in figure 2D.

This embodiment is advantageous in that the assembly formed by the donor substrate 1 and the support 7 can be exposed to a temperature that is much higher than that applied within the context of the first embodiment, without risking the uncontrolled fracturing of one of the substrates or the delamination of the donor substrate 1 from the thin layer 3. The balanced structure, in terms of the thermal expansion coefficient of this assembly, thus facilitates the step of detaching the first layer 3 by exposing the assembly to a relatively high temperature, for example, ranging between 100 °C and 500 °C.

Irrespective of the selected embodiment, and as specified in the introduction of this application, steps of finishing the first layer 3 are subsequently necessary in order to improve its crystal and surface quality, and to provide a thin layer 3' with a thickness that matches, or is close to, a target thickness. These finishing steps, which are schematically shown in figure 1E and figure 2E, are notably intended to eliminate a hard and

rough surface layer resulting from the cleavage and detachment of the thin layer 3 from the rest of the donor substrate.

As disclosed in document WO 2020/200986, a heat treatment step is initially applied to the first removed layer 3. This heat treatment allows any crystalline defects present in this layer 3 to be rectified, and even allows the roughness of its free face 8 to be reduced. Furthermore, it helps to consolidate its adhesion to the support 7. The heat treatment brings the structure to a temperature ranging between 300 °C and the Curie temperature of the ferroelectric material for a duration ranging between 30 minutes and 10 hours. This heat treatment is preferably carried out by exposing the free face of the first layer 3 to an oxidizing or neutral gaseous atmosphere, i.e., without covering this face of the thin layer with a protective layer.

For the avoidance of doubt, it should be noted that the finishing heat treatment step is quite distinct from the fracturing heat treatment applied to the assembled structure. It is notably carried out in equipment different from that used to apply the fracturing heat treatment.

A method according to the invention also includes, after the finishing heat treatment, a step of thinning the first thin layer. This step is notably aimed at eliminating the multi-domain surface portion of the first layer 3, with this portion having been created during the previous heat treatment step. It also aims to provide a thin single-domain layer 3', the thickness of which corresponds to a target thickness, as previously stated. This thinning can generally correspond to polishing of the first free face 8 of the thin layer 3, for example, by means of mechanical or chemical-mechanical thinning techniques. It can

also involve thinning carried out by ion etching, for example, by reactive ion etching.

In all cases, this thinning results in the elimination of at least the thickness of the multi-domain portion of the first layer 3, with this thickness typically being of the order of at least 150 nm, which leads to the disadvantages described in the introduction to this application. Figures 1F and 2F show the structure 9 obtained on completion of these treatments, with a thin single-domain layer 3' being arranged on the support 7.

In order to characterize the thickness of the multi-domain surface portion, a sample of this layer is usually inspected by means of transmission electron microscopy. This inspection technique allows the multi-domain surface portion and the underlying single-domain portion to be visualised and distinguished in the cross-section of the sample. However, the extent of this view remains limited to inspection fields of a few hundred microns, which does not fully represent the quality of the first layer 3 over its entire extent. It is also a long and complex technique to deploy.

In order to overcome this analysis problem, the applicant has developed a faster characterization technique that enables the thickness of the multi-domain portion to be assessed more broadly.

This technique comprises a first step involving removing a given thickness of the first layer 3 (for example, 125nm) by chemical-mechanical polishing. Following this first step, the topography of the revealed face is measured: a high topography indicates the multi-domain quality of the material whose face is exposed and, conversely, a low topography indicates the single-domain quality of the material whose face is revealed. The topography

measurement can be carried out by atomic force measurement (AFM) on measurement fields of 5 micrometres by 5 micrometres. Within the context of this description, "high topography" is understood to mean a surface with a peak-to-valley roughness of 10 nm or more, and, conversely, "low topography" is understood to mean a surface with a peak-to-valley roughness of less than 10 nm.

The speed of the chemical or physico-chemical etching of the thin layer 3 occurring during the polishing step is rendered variable by the nature of the polarity of the removed piezoelectric material: the Z- side of this layer is etched much faster than the Z+ side. Consequently, polishing a single-domain layer will have a much weaker surface topography than polishing a multi-domain layer.

Furthermore, according to the proposed characterization technique, by assessing the topography of the exposed face of the thin layer after it has been thinned by a given thickness, it is possible to very simply determine whether the multi-domain layer extended to a depth greater than the given thickness (revealed by a high topography) or whether it extended to a thickness less than the given thickness (revealed by a low topography). This somewhat rough characterization of the multi-domain thickness nevertheless allows an overall assessment to be provided of this thickness by repeating the topography measurement on a plurality of locations sampling the entire extent of this layer.

Using this global characterization technique, which is quick to implement, and by selecting a specific thickness of 125 nm, the applicant has realized that the method for preparing a thin layer 3 of a ferroelectric material as described above (according to two different embodiments) leads to the formation, after the step of heat treating the transferred thin layer 3 and before

the thinning step, of a multi-domain surface layer that is at least 125 nm.

In the search for a solution for reducing the thickness of this multi-domain surface portion, the applicant quite surprisingly discovered that introducing a sufficient hydrogen dose into a surface thickness of the first layer 3, before applying the heat treatment step to this layer 3, tended to reduce the thickness of the multi-domain surface portion that is revealed during this heat treatment.

This discovery results from experiments that were conducted and that involved applying, immediately after the step of fracturing a first layer of lithium tantalate, interlayer treatment to the free face 8 of the first thin layer. After this interlayer treatment step, the heat treatment was applied that led to the development of the multi-domain surface portion, which was characterized using the technique described above, by reducing the thickness of the first 125 nm layer by polishing and then measuring its topography.

These experiments included the following list of interlayer treatments of the first layer 3:

- Cleaning 1: cleaning the free face 8 of the first thin layer by brushing and dispensing deionized water at ambient temperature. Characterization of the layer after heat treatment revealed a thickness of more than 125 nm for the multi-domain surface portion.
- Cleaning 2: cleaning the free face of the thin layer 3 by successive immersion in baths of deionized water, SC1 and SC2, all at ambient temperature. Characterization of the layer after heat treatment revealed a thickness of more than 125 nm for the multi-domain surface portion.

- Cleaning 3: cleaning the free face by successive immersion in baths of deionized water, SC1 and SC2, with the SC1 solution being heated to 70 °C this time. Characterization of the layer after heat treatment revealed a thickness of less than 125 nm for the multi-domain surface portion.

In an attempt to understand the reason for this phenomenon, the applicant carried out SIMS (Secondary Ion Mass Spectrometry) measurements of the first layer 3 in order to determine the hydrogen concentration profile according to the depth of the first layer. Figure 3 thus shows the hydrogen concentration profile of the first layer 3 directly after fracturing (without applying the cleaning - NET0), after applying the cleaning 1 (NET1) and after applying the cleaning 3 (NET3). A hydrogen-rich surface zone can be seen that is of the order of 200 nm thick. Applying the cleaning 3 (which limits the thickness of the multi-domain surface portion) produces a hydrogen concentration in the surface zone of the order of $3.0 \text{ E}21 \text{ at/cm}^3$, whereas it does not exceed a concentration of $1.0 \text{ E}21 \text{ at/cm}^3$ in the other cases.

When these hydrogen concentration data are processed, it can be seen that the cleaning 1 introduces a hydrogen dose of $1.4 \text{ E}16 \text{ at/cm}^2$ into the first layer 3, yet without allowing the thickness of the surface multi-domain portion to drop below 125 nm. For its part, the cleaning 3 introduces a hydrogen dose of $4.3 \text{ E}16 \text{ at/cm}^2$ and reduces the thickness of the multi-domain portion below 125 nm.

These preliminary results tending to link the thickness of the surface multi-domain portion to the concentration of hydrogen in the first layer 3 were confirmed by other experimental measurements.

According to one of these measurements, applying a treatment to the free face of the thin layer 3 involved applying a sequence of 5 cleaning steps using a deionized water solution, with the cleaning steps being separated from each other by a waiting time of 24 hours or more. The solution was at ambient temperature. This treatment resulted in a multi-domain thickness of less than 125 nm, from the 3rd cleaning step in the sequence.

The effect of these cleaning steps is to eliminate a thin surface layer of Li_2CO_3 on the thin layer 3 obtained directly after the fracturing step. Its formation seems to be favoured by the particular conditions in which the fracturing step occurs. The presence of light species, namely hydrogen and/or helium, and the moderate temperature at which the fracturing occurs seem to render the lithium in the thin layer 3 particularly mobile and the surface of this layer 3 particularly reactive. This surface thickness of Li_2CO_3 is of the order of a nanometre or more thick. It is stable over time, i.e., it does not change in consistency or thickness when the thin layer 3 is kept exposed to the atmosphere. However, this surface layer of Li_2CO_3 is relatively fragile, and the applicant has observed that it could be eliminated by simple wet cleaning. The applicant has also observed that the thin layer 3, cleaned and devoid of its surface thickness of Li_2CO_3 , remained particularly reactive. By keeping the free face of the thin layer 3 exposed to the atmosphere for an extended period of time, amorphous dendrites, rich in lithium and hydrogen (and other species present in the atmosphere such as chlorine or fluorine) nucleate and develop again. Carrying out successive cleaning steps that are separated from each other by a sufficient duration tends to strip a surface thickness of the thin layer 3 of its lithium (which migrates and accumulates on the surface) and, at the same time, tends to introduce hydrogen elements as a replacement. When a sufficient hydrogen

dose has been introduced into the thin layer 3, the heat treatment applied to this layer leads to the formation of a multi-domain thickness of less than 125 nm, which confirms the hypothesis that increasing the hydrogen concentration in a surface thickness of the thin layer 3 tends to reduce the thickness of the surface multi-domain portion induced by the heat treatment of the finishing sequence of this layer.

As a final experimental measure, a 30 nm thickness of hydrogen-rich silicon oxide was formed on the thin layer 3 by PECVD deposition. The finishing sequence was then annealed, resulting in a significant hydrogen dose being injected into the thin layer 3 by diffusion. Characterization of this thin layer 3, after removing the silicon oxide layer, revealed that the multi-domain layer was less than 125 nm thick.

The applicant concluded from these experiments that treatment of the free face of the first layer 3 that is aimed at forming a high hydrogen concentration in a surface thickness of this layer, for example, greater than 2.0×10^{21} at/cm³ in a surface thickness of 100 nm or 200 nm, allowed the thickness of the surface multi-domain portion to be significantly reduced.

Advantageously, treatment of the free face results in a hydrogen dose being introduced into the first layer 3 that is greater than or equal to 2.0×10^{16} at/cm².

Under these conditions, the thickness of the surface multi-domain portion revealed by the heat treatment of the finishing sequence is less than 125 nm.

Therefore, the invention benefits from these results in order to propose a method for preparing a thin single-domain layer 3'. This method is schematically shown in figure 4. It uses all the

steps disclosed in relation to the description of figures 1A to 1E and 2A to 2E. In particular, this method comprises:

- a step of implanting "light" species in a first face 4 of a ferroelectric donor substrate 1 in order to form a fragilization plane 2 and to define a first layer 3 between the fragilization plane 2 and the first face 4 of the donor substrate 1;
- a step of assembling the first face 4 of the donor substrate 1 on a support 7 in order to form an intermediate assembly;
- a step of fracturing the intermediate assembly, comprising a first heat treatment, with this step leading to the fracturing of the donor substrate 1 at the fragilization plane 2 and to the formation of a free face 8 of the first layer 3;
- a sequence for finishing the first layer 3 comprising an annealing step including a second heat treatment and, after the annealing step, a step of thinning the first layer 3 in order to form the thin single-domain layer 3'.

According to the invention, the preparation method comprises, between the fracturing step and the finishing sequence, applying a treatment to the free face 8 of the first layer in order to produce a hydrogen concentration that is greater than $2.0 \text{ E}21 \text{ at/cm}^3$ in a surface thickness of the first layer 3.

This hydrogen-rich surface thickness of the first layer can be at least 100 nm thick from the free face 8 of this first layer. Advantageously, it is at least 200 nm thick.

The hydrogen concentration can be obtained by selecting the treatment of the free face so that this treatment introduces a hydrogen dose into the first layer 3 that is greater than or equal to $2.0 \text{ E}16 \text{ at/cm}^2$.

According to a first approach, treating the free face 8 comprises immersing the first layer 3 in a first solution that is at a temperature higher than ambient temperature, for example, higher than 50 °C, preferably higher than or equal to 65 °C. This first solution can be or can include SC1. The treatment of the first layer can notably correspond to RCA type cleaning, involving, as is well known per se, a sequence formed by successive immersion of the structure comprising the first layer 3 in baths of deionized water, SC1 and SC2. When the treatment of the free face of the invention is implemented by such RCA type cleaning, the temperature of the bath of SC1 in which the structure is immersed is higher than the ambient temperature.

According to another approach, the treatment of the free surface 8 comprises a sequence of cleaning steps using a second solution, with the cleaning steps being separated from one another by a waiting time of 24 hours or more. The cleaning steps can correspond to brushing the free face 8 and, at the same time, to dispensing deionized water onto the free face 8 of the first layer 3, with the deionized water then forming the second solution used during the cleaning steps. This second solution can be at ambient temperature or can be brought to a temperature above ambient temperature.

Advantageously, the sequence of cleaning steps comprises at least three cleaning steps, preferably at least five cleaning steps.

According to yet another approach, treating the free face 8 comprises depositing a hydrogen-rich covering layer onto the free face 8, for example, with a hydrogen concentration of more than 1.0×10^{20} at/cm³. The covering layer can notably comprise silicon dioxide, silicon nitride or silicon oxynitride formed, for example, by a chemical vapour deposition technique at sub-

atmospheric pressure. Irrespective of the nature of the covering layer, it can be 20 nm or more thick in order to contain a sufficient amount of hydrogen.

By introducing hydrogen into the first layer 3 before applying the finishing sequence to this layer, it is possible to reduce the thickness of the multi-domain surface portion revealed by this sequence. In particular, it is possible to limit this thickness to less than 125 nm.

Of course, the invention is not limited to the described embodiments, and alternative embodiments can be used without departing from the scope of the invention as defined by the claims.

In particular, treatment of the free face 8 can be applied between the fracturing step and the finishing sequence, combining the three illustrated approaches and according to any possible combination.

The treatment of the free face of the removed layer 3 can also correspond to any other treatment of this face leading to the introduction of a sufficient hydrogen dose into this layer.

CLAIMS

1. Method for preparing a thin single-domain layer (3') of ferroelectric material, the method comprising:
 - a step of implanting "light" species in a first face (4) of a ferroelectric donor substrate (1) in order to form a fragilization plane (2) and to define a first layer (3) between the fragilization plane (2) and the first face (4) of the donor substrate (1);
 - a step of assembling the first face (4) of the donor substrate (1) on a support (7) in order to form an intermediate assembly;
 - a step of fracturing the intermediate assembly, comprising a first heat treatment, with this step leading to the fracturing of the donor substrate (1) at the fragilization plane (2) and to the formation of a free face (8) of the first layer (3);
 - a sequence for finishing the first layer (3) comprising an annealing step including a second heat treatment and, after the annealing step, a step of thinning the first layer (3) in order to form the thin single-domain layer (3');

the preparation method being characterized in that it comprises, between the fracturing step and the finishing sequence, applying a treatment to the free face (8) in order to produce a hydrogen concentration that is greater than 2.0×10^{21} at/cm³ in a surface thickness of the first layer (3).

2. Preparation method according to the preceding claim, wherein the surface thickness, in which the hydrogen concentration is greater than 2.0×10^{21} at/cm³, is greater than or equal to 100 nm.

3. Preparation method according to the preceding claim, wherein the surface thickness is greater than or equal to 200 nm.
4. Preparation method according to any of the preceding claims, wherein the treatment of the free face (8) introduces a hydrogen dose into the first layer (3) that is greater than or equal to 2.0×10^{16} at/cm².
5. Preparation method according to any of the preceding claims, wherein the treatment of the free face (8) comprises immersing the first layer (3) in a first solution having a temperature higher than ambient temperature.
6. Preparation method according to the preceding claim, wherein the first solution comprises SC1 and/or SC2.
7. Preparation method according to any of the two preceding claims, wherein the temperature of the first solution is greater than 50 °C, preferably greater than 65 °C.
8. Preparation method according to any of the preceding claims, wherein the treatment of the free face (8) comprises a sequence of cleaning steps using a second solution, with the cleaning steps of the sequence being separated from one another by a waiting time that is 24 hours or more.
9. Preparation method according to the preceding claim, wherein the second solution comprises deionized water, and at least three cleaning steps comprising simultaneously brushing and dispensing deionized water onto the free face (8) of the first layer (3).

10. Preparation method according to any of the two preceding claims, wherein the second solution is at ambient temperature.
11. Preparation method according to any of the three preceding claims, wherein the sequence of cleaning steps comprises at least five cleaning steps.
12. Preparation method according to any of the preceding claims, wherein the treatment of the free face (8) comprises depositing a covering layer onto the free face (8) with a hydrogen concentration that is greater than $1.0 \times 10^{20} \text{ at/cm}^3$.
13. Preparation method according to the preceding claim, wherein the covering layer comprises silicon dioxide, silicon nitride or silicon oxynitride.
14. Preparation method according to the preceding claim, wherein the covering layer is 20 nm or more thick.
15. Preparation method according to any of the preceding claims, wherein the thin single-domain layer (3) is made up of a single-crystal piezoelectric material, such as lithium tantalate or lithium niobate.
16. Preparation method according to the preceding claim, wherein the thin single-domain layer (3) is made up of lithium niobate.
17. Preparation method according to any of the preceding claims, wherein the assembly step comprises forming a dielectric interlayer (3) on the first face of the donor substrate (1) and/or on the support (7).