A METHOD OF FABRICATING AN EPITAXIALLY GROWN LAYER

The present invention relates to a method of fabricating an epitaxially grown layer, i.e. a layer obtained by epitaxy, intended in particular for applications in the fields of optics, optoelectronics, or electronics.

Certain materials, for example silicon, having properties that are particularly interesting in the fields mentioned above, can be obtained in large quantities and with excellent quality using ingot pulling techniques that are well known to the skilled person.

The ingots obtained are then readily sliced into wafers which, after a large number of processing steps, become substrates that are used to produce integrated circuits, for example.

Other materials such as gallium arsenide, gallium nitride, indium phosphide, germanium, or silicon carbide, are also of interest. However, not all of those materials can be obtained by pulling ingots of high crystalline quality. Further, it is not always possible to fabricate substrates from such ingots because the costs are too high or the method is too difficult to implement.

Thus, for example, no method currently exists for fabricating gallium nitride (GaN) ingots on an industrial scale.

The document "Bulk and homoepitaxial GaN growth and characterization", Porowski-S, Journal of Crystal Growth, vol 189-190, June 1998, pp. 153-158, describes a method of growing a monocrystalline GaN ingot in the liquid phase at a pressure of 12 to 20 kbars (12 to 20 $\times 10^8$ Pascals (Pa)) and at a temperature in the range 1400°C to 1700°C. However, those conditions are difficult to implement under mass production

conditions. Further, they can only produce crystals with a maximum diameter of 18 millimeters (mm).

Other research teams have also worked on a method of growing an ingot in the liquid phase at reduced pressure (less than 2 bars (2×10^5 Pa)) and at a temperature of 1000°C. The diameter of the crystals obtained is larger, close to 50 mm, but the crystalline quality obtained is not as good as with the above-mentioned technique.

Finally, the document "Growth and characterization of GaN single crystals", Balka et al, Journal of Crystal Growth, vol 208, January 2000, p 100-106, discloses the growth of monocrystalline GaN by sublimation. The fabrication conditions employed are a pressure of less than 1 bar ((10^5 Pa) and a temperature of 1000°C to 1200°C. The crystalline quality obtained is very high, but the crystal size is 3 mm, which is clearly insufficient for the envisaged applications.

Thus, the market currently offers no monocrystalline gallium nitride in the bulk form, of high quality, of a sufficient diameter, and at a reasonable price.

The prior art discloses a number of attempts to fabricate substrates by epitaxy or heteroepitaxy in order to overcome the problem of obtaining certain specific materials.

Epitaxy can combine materials with different natures in the form of thin films and combine their properties to produce components, for example high electron mobility transistors (HEMTs), diodes, or lasers.

Heteroepitaxy consists of depositing the desired material onto a support of different crystallographic nature by epitaxy, and then eliminating the support, if possible and necessary for the remainder of the process.

The main drawback of that technique is that the material constituting the support and that deposited by epitaxy generally have different lattice parameters and thermal expansion coefficients.

The differences in lattice parameters between the support and the epitaxial layer create a large number of crystalline defects in the epitaxially grown material, such as dislocations or stacking defects, for example.

Added to that is the fact that epitaxial growth is generally carried out at high temperatures, above 600°C and possibly up to 1000°C to 1100°C, for example, when growing gallium nitride epitaxially by metal organic chemical vapor deposition (MOCVD). For that reason, as the structure which is formed cools to ambient temperature, the epitaxial layer obtained develops a number of residual stresses and strains connected with differences in thermal expansion between it and its support.

To overcome that drawback, the material selected as the support preferably has crystalline structure and thermal expansion coefficient very close to those of the materials which are to be grown epitaxially. As an example, gallium and indium arsenide or gallium and aluminum arsenide can be grown epitaxially on a gallium arsenide support with crystallographic quality that is sufficient to produce components.

However, other materials do not always have a compatible support that is in the form of a substrate. This is particularly the case with materials such as gallium nitride or cubic silicon carbide.

Until now, components having one of those two materials as the active layer have been grown by heteroepitaxy.

Thus, for gallium nitride, light-emitting diodes (LEDs) and lasers emitting in the blue, violet and ultraviolet as well as high frequency power components

have been produced using sapphire, hexagonal silicon carbide, or silicon as the support.

For silicon carbide, which is unavailable in the form of a substrate in its cubic crystalline structure, micro electromechanical components (MEMS) or power transistors have been produced by depositing silicon carbide onto a silicon substrate by epitaxy.

However, in order to further improve the quality of the components obtained, it would be desirable to fabricate bulk gallium nitride or cubic silicon carbide substrates of the same nature as the epitaxially grown layer deposited over it.

Attempts made in the past have all resulted in products with a certain number of disadvantages.

As an example, one intermediate route consists in using a technique termed "high growth rate epitaxy" to produce an epitaxially grown film that is as thick as the substrate supporting it. That support substrate is then eliminated and only the thick epitaxially grown film is retained, which in turn becomes a substrate for conventional epitaxy. Methods of that type exist for producing gallium nitride and silicon carbide, but the quality of the substrates obtained is generally mediocre because of the influence of the original support substrate of different crystallographic nature.

Thus, typically, large residual stresses are observed in the case of the epitaxial growth of cubic silicon carbide on silicon. Such stresses generally result in very substantial curvature of the epitaxially grown silicon carbide once the original silicon support substrate has been removed. That curvature renders it unusable for all subsequent forming steps.

Similarly, for the gallium nitride obtained, the influence of the support substrate is illustrated by the appearance of a very large number of dislocations and by cracking of the epitaxially grown film as its

temperature falls, in particular when that epitaxially grown film exceeds a certain critical thickness.

United States patent No. 6 146 457 describes a further method that makes use of the stresses that appear as the temperature falls following epitaxy as the driving force for detaching an original support substrate from a thick epitaxially grown layer. That result is obtained by inserting a layer termed a "weak" layer between the support substrate and the thick epitaxially grown layer such that when the system stresses increase, the weak layer ruptures and thus ensures controlled detachment of the support from the thick epitaxial layer. However, that detachment technique is difficult to control with a large specimen. Further, it involves inserting a particular layer as epitaxial growth commences or during epitaxial growth, which may be deleterious to the crystallographic quality of that epitaxially grown layer.

Further, the document "Physical properties of bulk GaN crystals grown by HVPE", Melnik et al, MRS Internet Journal of Nitride Semiconductor Research, vol 2, art 39, describes a method of growing gallium nitride (GaN) monocrystals by HVPE on a monocrystalline silicon carbide (SiC) substrate, and removing that substrate using a reactive ionic etching (RIE) technique. However, that SiC substrate takes a long time to remove since it is highly inert chemically.

Finally, the document "Large free-standing GaN substrates by hydride vapor phase epitaxy and laser induced lift-off", Kelly et al, Jpn J Appl Phys, vol 38, 1999, describes a method of growing GaN by hydride vapor phase epitaxy (HVPE) on a sapphire substrate, then removing that substrate by laser-induced lift-off. That technique is based on using a laser of a wavelength that is absorbed only by gallium nitride and not by sapphire. Scanning the resulting structure with

the laser ensures that the two materials become detached by local modification of the properties of the GaN after passage of the laser.

However, that lift-off technique is difficult to implement when treating large areas, since laser beam scanning takes a long time. It is also possible to remove the sapphire substrate by mechanical polishing, but that method is also lengthy and furthermore, it runs the risk of breaking the gallium nitride layer when lifting off the substrate, by releasing the existing stresses.

There is therefore a need to overcome or at least ameliorate the disadvantages of the prior art cited above.Disclosed herein is a method of fabricating an epitaxially grown layer of excellent crystallographic quality that can readily be detached from its epitaxy support.

More particularly, the method can be applicable to all materials which have previously been obtainable only by heteroepitaxy, in particular wide band gap materials.

Also disclosed herein is an epitaxy support substrate and a method for its fabrication.

In a first aspect, there is provided a method of fabricating an epitaxially grown layer in particular for applications in the fields of optics, optoelectronics, or electronics.

Said method is characterized in that it comprises the following steps consisting in:

a) implanting atomic species within a first substrate termed the "support substrate" to define therein a zone of weakness which demarcates a thin layer termed the "thin support layer" from the remainder of said substrate;

b) transferring onto the free surface of said thin support layer a thin nucleation layer of a nature which is suitable for subsequent epitaxial growth of said

epitaxially grown layer, by implementing a bonding interface between the two;

c) detaching the remainder of the support substrate along the corresponding zone of weakness, but while maintaining the thin support layer in contact with said remainder;

d) growing said epitaxially grown layer by epitaxy on said nucleation layer; and

e) moving away the remainder of the support substrate from the thin support layer.

The method can enable the nucleation layer to be detached from its support before the start of epitaxial growth of the desired material, and thus enables the influence of the support substrate during subsequent epitaxial growth to be minimized.

In addition, that method can overcome the problem of eliminating the support substrate; such a step can be difficult to implement, as mentioned above in the analysis of the prior art. As an example, eliminating hard materials such as sapphire or silicon carbide is lengthy and difficult using conventional lapping or chemical or ionic etching techniques.

The method may also present the following advantageous and non-limiting characteristics, taken in isolation or in combination:

According to a first embodiment of the invention, step b) of transferring the thin nucleation layer is carried out:

- by implanting atomic species within a second substrate termed the "nucleation substrate" to define therein a zone of weakness which demarcates said thin nucleation layer from the remainder of said substrate, then

- by bonding the two substrates one against the other, so that their respective thin support and nucleation layers are located facing each other, and

- by detaching the remainder of the nucleation substrate along the corresponding zone of weakness,

the parameters for the two implantations being selected so that when combined with the nature of the pair of materials constituting said support substrate and said nucleation substrate, the energy budget to be supplied for subsequently carrying out detachment of the remainder along the zone of weakness is greater than the cumulative energy budget supplied during all of the steps preceding said detachment.

Advantageously, the parameters for the two implantations are selected so that, when combined with the nature of the materials constituting said support substrate and said nucleation substrate, the thermal budget to be supplied for subsequently carrying out detachment of the remainder along the zone of weakness is greater than the cumulative thermal budget supplied during all of the steps preceding said detachment, and the detachment steps are carried out by heating.

Advantageously, the remainder of the nucleation substrate or the remainder of the support substrate is detached by application of additional energy of mechanical, optical, and/or chemical origin.

According to a second embodiment of the invention, step b) of transferring the thin nucleation layer is carried out:

- by bonding a second substrate, termed the "nucleation substrate" onto said thin support layer,

- by reducing the thickness of said substrate from its back face by lapping and/or chemical etching and/or ionic etching to obtain said thin nucleation layer.

Advantageously, bonding the two thin support and nucleation layers is carried out by means of at least one layer of intermediate bonding material, made for example of dielectric material, such as silicon oxide, silicon nitride, and silicon oxynitrides.

The bonding of the two thin support and nucleation layers either directly one against the other or in the presence of at least one layer of intermediate bonding material may be carried out by molecular bonding.

Advantageously, before step d), the method consists in epitaxially growing the epitaxially grown layer so as to form a fine nucleation layer on said thin nucleation layer.

Said fine nucleation layer may be produced by metal organic chemical vapor deposition (MOCVD), by hydride vapor phase epitaxy (VHPE), by molecular beam epitaxy (MBE), by cathode sputtering deposition, by epitaxial lateral over growth (ELOG) or pendeo-epitaxy.

Advantageously, the method comprises the additional step of eliminating the thin support layer, the thin nucleation layer, and possibly the layer or layers of dielectric material remaining integral with the epitaxially grown layer.

The epitaxially grown layer may be produced from a wide band gap semiconductor material, for example gallium nitride or cubic silicon carbide.

The epitaxially grown layer may be of sufficient thickness to constitute a self-supported layer.

According to a particular embodiment of the invention, the thickness of the epitaxially grown layer obtained at the end of step d) is insufficient to be self-supporting, and the method comprises the following additional steps carried out between steps d) and e) and consisting in:

- applying a first layer of metal onto said thin epitaxial layer and a second layer of metal onto a third substrate termed the "acceptor substrate",
- placing the two layers of metal in contact with each other and bonding them together,

to obtain the thin epitaxial layer transferred onto said acceptor substrate at the end of step e).

In a second aspect, there is provided a method of fabricating an epitaxy support substrate intended to be used for the fabrication of an epitaxially grown layer in particular for applications in the fields of optics, optoelectronics.

According to the invention, this method comprises the following steps consisting in:

i) implanting atomic species within a first substrate termed the "support substrate" to define therein a zone of weakness which demarcates a thin layer termed the "thin support layer" from the remainder of said substrate and along which said thin film and said remainder are detachable;

ii) transferring onto the free surface of said thin support layer a thin nucleation layer of a nature which is suitable for subsequent epitaxial growth of said epitaxially grown layer, by implementing a bonding interface between the two, in order to obtain said epitaxy support substrate.

Advantageously, step ii) of transferring the thin nucleation layer is carried out as above mentioned for stel b) of the method of fabricating an epitaxially grown layer.

Preferably, the bonding of the two thin support and nucleation layers is carried out as previously mentioned.

Finally, In a third aspect, there is provided an epitaxy support substrate intended to be used for the fabrication of an epitaxially grown layer in particular for above mentioned applications.

Said substrate comprises at least:

- a first substrate termed "support substrate" comprising a zone of weakness which demarcates a thin layer termed the "thin support layer" from the remainder of said substrate and

- a thin nucleation layer the nature of which is suitable for subsequent epitaxial growth of said

epitaxially grown layer, said nucleation layer being bonded to said support film either directly or by means of at least one layer of intermediate bonding material, such as a dielectric material.

Other characteristics and advantages of the invention become apparent from the following description made with reference to the accompanying drawings which provide non-limiting indications of possible implementations.

In the drawings:

- Figures 1 to 9 are diagrams representing the different successive steps of a first implementation of the method of the invention; and
- Figures 10 to 14 are diagrams representing the different successive steps of a second implementation of the method.

It should be noted that for simplification, the different layers shown in the figures are not drawn to scale particularly as regards their relative thicknesses.

The method of the invention uses a first substrate 1 termed the "support substrate" and a second substrate 2 termed the "nucleation substrate".

The support substrate 1 has a face 130 termed the "front face" intended to come into contact subsequently with the nucleation substrate 2 and an opposite face 110 termed the "back face".

Similarly, the nucleation substrate 2 has a front face 230 and an opposite back face 210.

The material constituting the support substrate 1 is selected to have good mechanical strength even at high temperatures which may reach 1300°C to 1400°C. Advantageously, currently commercially available bulk substrates are used.

Purely by way of illustration, it is possible to mention silicon, sapphire, polycrystalline silicon carbide, 6H or 4H monocrystalline silicon carbide,

gallium nitride (GaN), aluminum nitride (AlN), and zinc oxide (ZnO).

The nucleation substrate 2 is selected so that its nature and its lattice parameters allow subsequent epitaxial growth of the future layer of epitaxially grown material which is to be obtained and which has the reference numeral 6 or 6′ (see Figures 7 to 9 and 10 to 14).

As an example, the nucleation substrate 2 can be a semiconductor material such as gallium nitride (GaN), silicon (Si), silicon carbide (SiC), sapphire, diamond, gallium arsenide (AsGa), or aluminum nitride (AlN). However, this list is not limiting.

When an epitaxially grown layer of gallium nitride is to be formed, said nucleation substrate 2 is preferably selected from (111) silicon, silicon carbide (SiC), sapphire, or gallium nitride (GaN), and when the epitaxial layer is formed from cubic silicon carbide, the nucleation substrate 2 is preferably (001) silicon Si, or silicon carbide.

The two substrates 1 and 2 can be of identical or different natures.

An operation for implanting atomic species in the support substrate 1 and in the nucleation substrate 2 is then carried out, preferably on their respective front faces 130 and 230.

The term "atomic species implantation" means any bombardment of atomic, molecular, or ionic species which can introduce said species into a material with a maximum concentration of said species located at a depth that is determined with respect to the bombarded surface, in this case the surfaces 130 or 230. The molecular or ionic atomic species are introduced into the material with an energy that is also distributed about a maximum.

Atomic species implantation can be carried out, for example, using an ion beam implanter or a plasma immersion implanter.

Preferably, said implantation is carried out by ionic bombardment. Preferably, the implanted ionic species is hydrogen. Other ionic species can advantageously be used alone or in combination with hydrogen, such as rare gases (for example helium).

Reference should be made in this regard to the literature concerning the method known under the registered trade mark "Smart Cut".

Said implantation creates a zone of weakness 12 in the bulk of the source substrate 1 at a mean ion penetration depth. It divides the substrate 1 into two portions, namely a thin layer 13 termed below the "thin support layer" and the remainder 11 of said substrate.

The thin layer 13 extends between the zone of weakness 12 and its front face 130.

In similar manner, following implantation, the nucleation substrate 2 has a zone of weakness 22, a thin nucleation layer 23, and a remainder 21.

Optionally, and not shown in Figure 1, it is possible to form a layer of protective material on the front layer 130 of the substrate 1 and/or on the front face 230 of the substrate 2.

When such protective layers are present, atomic species implantation can be carried out through them, in particular by implantation over their respective free upper faces. Said layers may be sacrificial layers which act as masks and thus protect the substrates 1 and 2 during implantation. They can thus be eliminated after said implantation.

The parameters for the two atomic species implantations, i.e. the dose and the implantation energy, are selected as a function of the detailed criteria given below.

As shown in Figure 2, the support substrate 1 is then bonded onto the nucleation substrate 2 such that their respective thin layers 13 and 23 are located facing each other.

Advantageously, bonding is carried out by molecular bonding. Such bonding techniques are known to the skilled person and have been described, for example, in the work by Gösele, "Semiconductor wafer bonding", Sciences and Technology, Q.Y. Tong, U. Gösele, Wiley Interscience publications.

The bonding interface between the front face 130 of the support substrate 1 and the front face 230 of the support substrate 2 carries reference numeral 4.

However, it is also possible to carry out bonding between the thin layers 13 and 23 by means of at least one intermediate bonding layer formed on the front layer 130 of the substrate 1 and/or on the front face 230 of the substrate 2. Said layers can, for example, be the above-mentioned layers for protection during implantation or specific layers termed "bonding boosting layers" and carrying reference numerals 31 and 32. The bonding interface 4 is then the interface between the upper face 310 of the layer of material 31 and the upper face 320 of the layer of material 32.

As an example, said layers of material 31 and 32 can be formed from amorphous or polycrystalline silicon or from alumina (Al_2O_3).

Advantageously, said bonding boosting layers 31 and 32 can be layers of dielectric material selected, for example, from silicon oxide (SiO_2), silicon nitride (Si_3N_4), or silicon oxynitrides (Si_xO_yN). They may be identical in nature or different, and they have thickness in the range from a few tens of nanometers to about 1 micrometer (1 µm).

Said layers 31 and 32 can, for example, be obtained by plasma enhanced chemical vapor deposition,

(PECVD) or by oxidation of the substrate when it is formed from silicon or monocrystalline silicon carbide.

The two contacting surfaces are bonded after suitable surface preparation, for example and as is well known to the skilled person, by cleaning in known SC1 or SC2 type chemicals, by chemico-mechanical polishing, or by plasma or ultraviolet-ozone activation of one or both contacting surfaces. Ultraviolet-ozone activation is exposure to ultraviolet radiation in a gas environment so that the ultraviolet radiation interacts with the environment and generates ozone which then activates the surface. These various methods can also be combined.

The abbreviation "SC" corresponds to "standard clean". Cleaning product SC1 is based on liquid ammonia, hydrogen peroxide, and water, and SC2 is based on hydrochloric acid, hydrogen peroxide, and water.

Bonding can also be reinforced by thermal annealing. This annealing is adapted so that it has no influence on the zones of weakness 12 and 22 and does not cause detachment along those zones at this stage.

The aim of said treatment is to obtain strong bonding at the bonding interface 4 and to provide a "stiffening" effect which maintains the growth of defects formed at the zones of weakness 12 and 22 in a plane parallel to that of said bonding interface 4.

The remainder 21 of the nucleation substrate 2 shown in Figure 3 can then be detached by supplying energy of mechanical, thermal, optical, and/or chemical origin.

In this regard, it should be noted that the two above-mentioned atomic species implantation steps are carried out with dose and implantation energy parameters selected such that, combined with the nature of the pair of materials constituting the support substrate 1 and the nucleation substrate 2, the energy budget to be supplied to carry out subsequent

detachment of the remainder 11 along the zone of weakness 12 is greater than the cumulative energy budget supplied during all of the steps preceding detachment.

Advantageously, said parameters are selected so that the thermal budget to be supplied during detachment of the remainder 11 along the zone of weakness 12 is greater than the cumulative thermal budget supplied during all of the steps preceding said detachment and the steps for detaching the remainder 11 and the remainder 21 are carried out by heating.

Further, it should be noted that the strength of the bonding interface 4 is sufficient to tolerate said two detachments without unbonding.

Energy of thermal origin can be supplied by applying a suitable thermal budget, for example by heating the stack of layers shown in Figure 2 for a given time.

Energy of mechanical origin can be supplied, for example, by exerting bending and/or tensile stresses on the remainder 21 of the nucleation substrate 2, or by introducing a blade or a jet of fluid (liquid or gas), for example, at the zone of weakness 22. Shear or ultrasound forces may also be applied.

Mechanical stresses can also result from supplying energy of electrical origin (application of an electrostatic or electromagnetic field). They can also originate from thermal energy derived from applying an electromagnetic field, an electron beam, thermoelectric heating, a cryogenic fluid, a supercooled liquid, etc. They can also be of optical origin, consisting in applying a laser beam to the zone of weakness, light absorption by the material then providing sufficient energy to generate detachment along the zone of weakness.

Energy of chemical origin may be supplied by etching in a chemical product.

Advantageously, and as shown in Figure 4, it is possible to improve the surface quality of the free surface 231 of the layer 23 to limit its residual roughness observed after the step for detaching the remainder 21. This may prove useful in ensuring the success of the subsequent steps of the method, in particular deposition of a layer by epitaxial growth.

Said reduction in roughness can be achieved by different methods such as chemico mechanical polishing, thermal oxidation followed by deoxidation (in hydrofluoric acid, for example), plasma etching, or ion beam or ion cluster smoothing.

In the particular case in which the nucleation layer 23 is silicon onto which cubic silicon carbide is to be deposited by epitaxy, it is also possible to prepare the free surface 231 using the method described in European patent EP-A-1 288 346.

The thin layers 13 and 23 are of the order of 10 nanometers (10 nm) to about 10 micrometers (10 µm) thick, and preferably are in the range from a few tens of nanometers to 1.5 micrometers (1.5 µm).

In advantageous manner, one of the two thin layers 13 and 23 is much thicker than the other, i.e. five to twenty times thicker than the other, such that the free surface 231 is as flat as possible because of the absence of residual stresses inside the stack of layers shown in Figure 4.

Said stack of layers constitutes an epitaxy support substrate referenced 9.

The step shown in Figure 5 is optional.

It consists of forming a fine nucleation layer 5 on the thin nucleation layer 23 by epitaxy in order to obtain an epitaxy support substrate referenced 9'.

The two above-mentioned epitaxy support substrates 9 and 9' are advantageous in that the implantation made in substrate 1 is realized before the transfer of the

nucleation layer 23 so that the thin support layer 13 may be thicker as if it was done after said transfer.

Said layer 5 can be produced from the same material as that subsequently used to produce the epitaxially grown layer. It then serves as a seed for homoepitaxy, which improves the crystallographic quality of this layer.

Said fine layer 5 can also be used to compensate for differences in the expansion coefficients and lattice parameters existing between the nucleation layer 23 and the layer 6 which will subsequently be deposited by epitaxy.

As an example, and in particular for GaN, said fine layer 5 can be grown epitaxially by metal organic chemical vapor deposition (MOCVD) or by molecular beam epitaxy (MBE).

The fine nucleation layer 5 can also be obtained by hydride vapor phase epitaxy, HVPE, or by sputtering.

It is also possible to use epitaxial lateral over growth techniques, ELOG, or a technique known as "pendeoepitaxy" in which motifs are etched into the thin nucleation layer 23 before depositing the fine layer 5. Reference should be made to the article by Morgan et al, "Evaluation of GaN growth improvement techniques", Materials Science and Engineering B90 (2002), 201-205, for a description of these techniques.

The list of said techniques is not exhaustive.

The material used to produce the fine nucleation layer 5 can also be different from that used for the nucleation layer 23 and for the future epitaxially grown layer 6.

Regardless of the variant selected, i.e. whether or not the fine nucleation layer has been deposited, energy is supplied to the stack of layers which is sufficient to cause detachment of the remainder 11 along the zone of weakness 12, (see Figure 6).

In the particular variant comprising the nucleation layer 5, this supplied energy is greater than the cumulative energy budget applied during detachment at the zone of weakness 22 and during epitaxy of the fine nucleation layer 5.

In the variant without the nucleation layer 5, it is sufficient for said supplied energy to be greater than that supplied to carry out detachment along the zone of weakness 22 of the nucleation substrate.

Said detachment is carried out using the techniques described above for detachment along the zone of weakness 22.

Detachment isolates from the remainder 11 of the support substrate 1 a multilayered structure carrying general reference numeral 7' comprising the thin support layer 13, the thin nucleation layer 23, the fine nucleation layer 5 and, if present, the molecular bonding boosting layers 31 and 32. When the fine nucleation layer 5 is not present, the multilayered structure carries general reference numeral 7, as can be seen in Figure 6.

However, detachment is carried out by taking care not to move away the remainder 11 of the support substrate 1 from the structure 7 or 7' and in particular from the support layer 13. In other words, the thin support layer 13 stays in contact with the remainder 11. The two layers 11 and 13 remain "connected" because their facing surfaces, respectively 111 and 131, are very rough, which ensures sufficient friction to maintain the whole structure in place.

Further, the multilayered structures 7 or 7' and the remainder 11 of the support substrate 1 can be kept against each other by a clip, for example.

In a further variant, the stack of layers 7 or 7' and 11 can also be placed in an epitaxial reactor and detachment is then carried out, the susceptor (or support) however maintaining the thin layer 13 and the

remainder 11 in contact with each other as explained above. To this end, the susceptor can comprise a clip or a cavity with the same shape as the substrate, for example.

Said structure 7 is several hundred nanometers to about a few tens of microns thick; structure 7' is 200 nanometers (200 nm) to 100 micrometers (100 µm) thick; the fine nucleation layer 5 is two to ten times thicker than that of the structure 7. This range of thicknesses means that the multilayered structures 7 or 7' are mechanically stable during the subsequent step in which epitaxy is repeated in Figure 7.

As shown in Figure 7, the material intended to form the future epitaxially grown layer 6 to be obtained is deposited, either on the free surface 231 of the nucleation layer 23 or on the upper free face 50 of the fine nucleation layer 5, if present.

The epitaxially grown layer can be constituted by any material that can be deposited by epitaxy, for example and preferably large band gap semiconductor materials, in particular gallium nitride or cubic silicon carbide.

In a first variant, deposition is continued until the layer 6 reaches a thickness of at least 100 microns, whereupon it is self-supporting and constitutes a substrate which can in turn, for example, be used as a substrate for epitaxy.

Advantageously, said epitaxially grown layer 6 is obtained by a high growth rate epitaxial growth technique.

In the case of gallium nitride, a particularly suitable method is hydride vapor phase epitaxy, HVPE. Deposition is carried out at a temperature in the range 700°C to 1100°C.

In the case of cubic silicon carbide, a particularly suitable method is CVD carried out in a cold wall CVD reactor.

Since the layer 6 is substantially thicker than the multilayered structures 7 or 7′ by a factor of about 10 to 100, it can be considered that the influence of said multilayered structures on the epitaxially grown layer 6 is very small. This produces a very high quality layer of hetero- or homo-epitaxially grown material 6 since the support problems mentioned above in the analysis of the prior art are avoided.

Further, although it does not appear in the figures, the fine nucleation layer 5 is generally two to ten times thicker than the structure 7 on which it rests. As a result, the global structure 7′ can be considered to have the same properties as the material constituting said fine nucleation layer 5, which is advantageous for epitaxial growth of the material 6.

As shown in Figure 8, the remainder 11 of the support substrate 1 is move away from the thin support layer 13.

Finally, as shown in Figure 9, and advantageously, the multilayered structures 7 or 7′ are eliminated from the epitaxially grown layer 6, for example by chemical etching, reactive ion etching (RIE) or chemico-mechanical polishing or a combination of said techniques. This results in the epitaxially grown layer 6 alone or with the fine nucleation layer 5.

In the implementation described above, the nucleation layer 23 was obtained by forming a zone of weakness in the nucleation substrate 2. However, in a further variant, not shown in the figures, the thin nucleation layer 23 can be obtained in a different manner, by bonding a substrate 2 which does not have a zone of weakness onto the support substrate 1 with or without a dielectric layer 31 and 32, then by attacking the back face 210 of said substrate 2. Attack can be carried out either by chemical etching alone or by grinding followed by chemical attack and/or ionic

etching. That method is known to the skilled person as BESOI, which means "bond and etch back silicon on insulator".

A second variant of the method is described below with reference to Figures 10 to 14.

The first steps of this method are identical to those shown in Figures 1 to 4 of the first method and are not described again.

Energy is supplied to the stack of layers shown in Figure 4, in sufficient quantity to cause detachment of the remainder 11 from the support substrate 1 along the zone of weakness 12, (see Figure 10).

Said detachment is carried out using the techniques and energy conditions described in connection with the step shown in Figure 6.

Said detachment is therefore carried out taking care not to move the remainder 11 away from the support substrate 1 of the multilayered structure 7 which rests thereon.

A layer of material is then deposited by epitaxy, but its thickness is less than about 100 µm so that it is not self-supporting. Said epitaxially-grown thin layer is referenced 6'.

Deposition can be carried out by one of the techniques described in the first implementation or by metal organic chemical vapor deposition (MOCVD) or by molecular beam epitaxy (MBE).

Such a thin layer can constitute the active portion of a component and it must then be transferred to a third substrate or acceptor substrate which then becomes the mechanical support for the final component.

One example of a technique for transferring said epitaxially grown thin layer 6' is shown in Figure 11.

This uses the metal bonding technique. Reference should be made, for example, to US-6 335 263 for a description of this technique.

Said technique consists of depositing a first layer of metal 81 on the thin epitaxially grown layer 6′ and a second layer of metal 82 onto an acceptor substrate 8.

The metal layers 81 and 82 can also be replaced by multiple superimposed layers of metals and/or metal alloys.

Said metals are selected in order to obtain good bonding and/or good electrical conduction and/or to reflect light.

The metal layers 81 and 82 are then placed in contact with each other as shown in Figure 11 and the interface formed is bonded by treatment at a suitable temperature and pressure.

The remainder 11 is then moved away from the support substrate 1 of the thin support layer 13, (see Figure 13).

Finally, as shown in Figure 14, the multilayered structure 7 is eliminated until the epitaxially grown layer 6′ is obtained resting on the acceptor substrate 8 and capable of constituting a conducting structure.

In a variant, not shown in Figures 10 to 14, it is also possible to deposit the thin epitaxially grown layer 6′ on a fine nucleation layer 5 deposited on the thin nucleation layer 23 using the procedures described above for the implementation of the thick epitaxially-grown layer 6.

By means of the method of the invention, the nucleation layer 23 of the support 1 is detached before carrying out epitaxial growth of the thick layer 6 or of the thin layer 6′. A "pseudo-substrate" is thus created formed by the multilayered structure 7, 7′, which thus enables an epitaxially grown layer to be made without being subjected to the influence of the support substrate 1, and therefore without the drawbacks of the prior art. In other words, the layer 6 is rigorously flat, does not present cracks, has an

excellent crystalline quality, and its thickness is not limited.

Three examples of the method of the invention are given below.

EXAMPLE 1: Production of a thick epitaxially grown gallium nitride layer

Hydrogen was implanted into a [111] silicon nucleation substrate 2 through a silicon oxide layer 32 obtained by thermal oxidation.

Hydrogen was also implanted onto a support substrate 1 formed from polycrystalline silicon carbide (SiC) through a layer of silicon oxide 31 obtained by deposition.

The implantation conditions for the silicon nucleation substrate 2 were an energy of 120 kilo electron-volts (keV) and a dose of $5 \times 10^{16} H^+/cm^2$ and for the SiC support substrate 1, the energy was 95 keV and the dose was $6 \times 10^{16} H^+/cm^2$.

The silicon oxide layers 31 and 32 underwent chemico-mechanical polishing, CMP, to activate them and encourage their bonding by molecular bonding.

The remainder 21 of the [111] silicon layer was then detached by annealing at a temperature of 500°C for two hours. It should be noted that the thermal budget applied to ensure detachment in the silicon was less than that necessary to cause detachment in the silicon carbide so that detachment could not occur along the zone of weakness 12.

The structure obtained was then placed in a HVPE reactor and the remainder 11 of the support substrate was detached, but without moving away from the other portion of the stack of layers, just before a thick layer 6 of gallium nitride (GaN) was deposited. Said detachment was carried out by annealing at a temperature of 1000°C for 30 minutes.

The thickness of the GaN layer measured several hundred microns.

The remainder 11 was then moved away and the multilayered structure 7 was eliminated by chemical etching.

The resulting thick layer of GaN was of very good crystallographic quality.

EXAMPLE 2: Production of a thick epitaxially grown layer of monocrystalline cubic silicon carbide

Hydrogen was implanted into a monocrystalline [001] silicon nucleation substrate 2 through a silicon oxide layer 32 obtained by thermal oxidation.

Hydrogen was also implanted onto a support substrate 1 formed from polycrystalline silicon carbide (SiC) through a layer of silicon oxide 31 obtained by deposition.

The implantation conditions for the silicon nucleation substrate 2 were an energy of 120 keV and a dose of $5 \times 10^{16} H^+/cm^2$ and for the SiC support substrate 1, the energy was 95 keV and the dose was $6 \times 10^{16} H^+/cm^2$.

The silicon oxide layers 31 and 32 underwent chemico-mechanical polishing (CMP) to activate them and to boost bonding by molecular bonding.

The remainder 21 from the [001] silicon layer was then detached by annealing at a temperature of 500°C for two hours. It should be noted that the thermal budget applied to ensure detachment in the silicon was less than that necessary to cause detachment in the silicon carbide, so that detachment could not occur along the zone of weakness 12.

The upper surface 231 of the [001] silicon was then prepared by chemico-mechanical polishing and by sacrificial oxidation type heat treatment.

The structure obtained was then placed in a cold wall CVD reactor and a thick layer of cubic silicon carbide was grown epitaxially.

The remainder 11 of the SiC support substrate was detached, but without moving away from the other portion of the stack of layers, just before a thick

layer 6 of cubic silicon carbide (SiC) was deposited. Said detachment was carried out by annealing at a temperature of 1000°C for 30 minutes.

The thickness of the SiC layer measured several hundred microns.

The remainder 11 was then moved away and the multilayered structure 7 was eliminated by chemical etching.

The resulting thick layer of SiC was of very good crystallographic quality and had very little deformation or "bend" since there was no residual stress in the thick silicon carbide layer.

CLAIMS

1. A method of fabricating an epitaxially grown layer in particular for applications in the fields of optics, optoelectronics, or electronics, characterized in that it comprises the following steps consisting in:

a) implanting atomic species within a first substrate termed the "support substrate" to define therein a zone of weakness which demarcates a thin layer termed the "thin support layer" from the remainder of said substrate;

b) transferring onto the free surface of said thin support layer a thin nucleation layer of a nature which is suitable for subsequent epitaxial growth of said epitaxially grown layer, by implementing a bonding interface between the two;

c) detaching the remainder of the support substrate along the corresponding zone of weakness, but while maintaining the thin support layer in contact with said remainder;

d) growing said epitaxially grown layer by epitaxy on said nucleation layer; and

e) moving away the remainder of the support substrate from the thin support layer.

2. A method according to claim 1, characterized in that step b) of transferring the thin nucleation layer is carried out:

- by implanting atomic species within a second substrate termed the "nucleation substrate" to define therein a zone of weakness which demarcates said thin nucleation layer from the remainder of said substrate, then

- by bonding the two substrates one against the other, so that their respective thin support layer

and nucleation layer are located facing each other, and

- by detaching the remainder of the nucleation substrate along the corresponding zone of weakness;

the parameters for the two implantations being selected so that when combined with the nature of the pair of materials constituting said support substrate and said nucleation substrate, the energy budget to be supplied for subsequently carrying out detachment of the remainder long the zone of weakness is greater than the cumulative energy budget supplied during all of the steps preceding said detachment.

3. A method according to claim 2, characterized in that the parameters for the two implantations are selected so that, when combined with the nature of the materials constituting said support substrate and said nucleation substrate, the thermal budget to be supplied for subsequently carrying out detachment of the remainder along the zone of weakness is greater than the cumulative thermal budget supplied during all of the steps preceding said detachment and in that the detachment steps are carried out by heating.

4. A method according to claim 1 or claim 2, characterized in that the remainder of the nucleation substrate or the remainder of the support substrate is detached by application of additional energy of mechanical, optical, and/or chemical origin.

5. A method according to claim 1, characterized in that step b) of transferring the thin nucleation layer is carried out:

- by bonding a second substrate termed the "nucleation substrate" onto said thin support layer, by reducing the thickness of said substrate from its back face by lapping and/or chemical etching and/or ionic etching to obtain said thin nucleation layer.

6. A method according to any preceding claim, characterized in that bonding the two thin support and nucleation layers is carried out by means of at least one layer of intermediate bonding material .

7. A method according to claim 6, characterized in that said layer of intermediate bonding material is made of dielectric material.

8. A method according to claim 7, characterized in that the dielectric material is selected from silicon oxide, silicon nitride, and silicon oxynitrides.

9. A method according to any preceding claim, characterized in that bonding the two thin support and nucleation layers either directly one against the other or in the presence of at least one layer of intermediate bonding material is carried out by molecular bonding.

10. A method according to any preceding claim, characterized in that before step d), the method consists in epitaxially growing the epitaxially grown layer so as to form a fine nucleation layer on said thin nucleation layer.

11. A method according to claim 10, characterized in that said fine nucleation layer is produced by metal organic chemical vapor deposition (MOCVD), by hydride vapor phase epitaxy (VHPE), by molecular beam epitaxy (MBE), by cathode sputtering deposition, by epitaxial lateral over growth (ELOG) or pendeo-epitaxy.

12. A method according to any one of the preceding claims, characterized in that it comprises the additional step of eliminating the thin support layer, the thin nucleation layer, and possibly the layer or layers of dielectric material remaining integral with the epitaxially grown layer.

13. A method according to any one of the preceding claims, characterized in that the epitaxially grown layer is produced from a wide band gap semiconductor material.

14. A method according to claim 13, characterized in that the epitaxially grown layer is formed from gallium nitride.

15. A method according to claim 13, characterized in that the epitaxially grown layer is formed from cubic silicon carbide.

16. A method according to any one of the preceding claims, characterized in that the epitaxially grown layer is of sufficient thickness to constitute a self-supported layer.

17. A method according to any one of claims 1 to 15, characterized in that the thickness of the epitaxially grown layer obtained at the end of step d) is insufficient to be self-supporting and in that the

method comprises the following additional steps carried out between steps d) and e) and consisting in:

- applying a first layer of metal onto said thin epitaxially grown layer and a second layer of metal onto a third substrate termed the "acceptor substrate",

- placing the two layers of metal in contact with each other and bonding them together, to obtain the thin epitaxially grown layer transferred onto said acceptor substrate at the end of step e).

18. A method of fabricating an epitaxy support substrate intended to be used for the fabrication of an epitaxially grown layer in particular for applications in the fields of optics, optoelectronics, or electronics, characterized in that it comprises the following steps consisting in:

i) implanting atomic species within a first substrate termed the "support substrate" to define therein a zone of weakness which demarcates a thin layer termed the "thin support layer" from the remainder of said substrate and along which said thin film and said remainder are detachable;

ii) transferring onto the free surface of said thin support layer a thin nucleation layer of a nature which is suitable for subsequent epitaxial growth of said epitaxially grown layer, by implementing a bonding interface between the two, in order to obtain said epitaxy support substrate.

19. A method according to claim 18, characterized in that step ii) of transferring the thin nucleation layer (23) is carried out:

- by implanting atomic species within a second substrate termed the "nucleation substrate" to

define therein a zone of weakness which demarcates said thin nucleation layer from the remainder of said substrate, then

- by bonding the two substrates one against the other, so that their respective thin support and nucleation layers are located facing each other, and

- by detaching the remainder of the nucleation substrate along the corresponding zone of weakness;

the parameters for the two implantations being selected so that when combined with the nature of the pair of materials constituting said support substrate and said nucleation substrate, a subsequent detachment of the remainder along the zone of weakness is possible, provided that the energy budget supplied to do so is greater than the cumulative energy budget supplied during all of the steps preceding said detachment.

20. A method according to claim 18, characterized in that step ii) of transferring the thin nucleation layer is carried out:

- by bonding a second substrate termed the "nucleation substrate" onto said thin support layer;

- by reducing the thickness of said substrate from its back face by lapping and/or chemical etching and/or ionic etching to obtain said thin nucleation layer.

21. A method according to anyone of claims 18 to 20, characterized in that bonding the two thin support and nucleation layers is carried out by means of at least one layer of intermediate bonding material, such as a dielectric material.

22. A method according to claim 21, characterized in that the dielectric material is selected from silicon oxide, silicon nitride, and silicon oxynitrides.

23. A method according to anyone of claims 18 to 22, characterized in that bonding the two thin support and nucleation layers either directly one against the-other or in the presence of at least one layer of intermediate bonding material is carried out by molecular bonding.

24. A method according to anyone of claims 18 to 23, characterized in that it comprises the steps of forming a fine nucleation layer on said thin nucleation layer.

25. A method according to claim 24, characterized in that said fine nucleation layer is produced by metal organic chemical vapor deposition (MOCVD), by hydride vapor phase epitaxy (VHPE), by molecular beam epitaxy (MBE), by cathode sputtering deposition, by epitaxial lateral over growth (ELOG) or pendeo-epitaxy.

26. An epitaxy support substrate intended to be used for the fabrication of an epitaxially grown layer in particular for applications in the fields of optics, optoelectronics, or electronics, characterized in that it comprises at least:
- a first substrate termed "support substrate" comprising a zone of weakness which demarcates a thin layer termed the "thin support layer" from the remainder of said substrate and
- a thin nucleation layer the nature of which is suitable for subsequent epitaxial growth of said epitaxially grown layer, said nucleation layer being bonded to said support film either

directly or by means of at least one layer of intermediate bonding material, such as a dielectric material.

27. An epitaxy support substrate according to claim 26, characterized in that said nucleation layer is covered with a fine nucleation layer.

28. An epitaxy support substrate according to claims 26 or 27, characterized in that the support substrate is made of a material selected from silicon, sapphire, polycrystalline silicon carbide, 6H or 4H monocrystalline silicon carbide, gallium nitride (GaN), aluminum nitride (AlN), and zinc oxide (ZnO).

29. An epitaxy support substrate according to anyone of claims 26 to 28, characterized in that the nucleation layer is made of a material selected from gallium nitride (GaN), silicon (Si), silicon carbide (SiC), sapphire, diamond, gallium arsenide (AsGa), and aluminum nitride(AlN).