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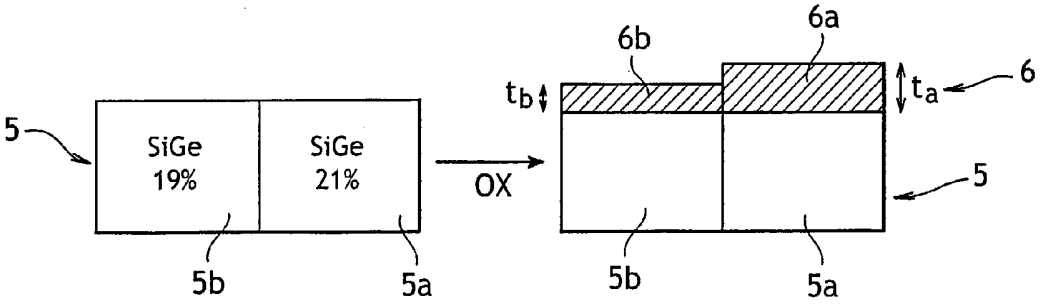
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(54) Title: METHOD FOR DETERMINING THE COMPOSITION OF A LAYER



(57) Abstract: The invention proposes a method for determining the composition of a layer, said layer being of a composed semiconductor material which comprises at least two elements and has an oxidation rate related to the concentration within said material of an element among said elements, said layer having a main surface, said method comprising the steps of: oxidizing the main surface of said layer for forming an oxide layer on said main surface; measuring the thickness of said oxide layer on said main surface; determining, from said thickness measurement, the concentration, within the composed material of said layer, of said element whose concentration is related to the oxidation rate. Application to the determination of a variable Ge concentration within a SiGe layer.

WO 2006/010996 A1

METHOD FOR DETERMINING THE COMPOSITION OF A LAYER

BACKGROUND OF THE INVENTION

Technical field

The invention relates to the layer composition analysis techniques employed during the manufacturing of substrates for electronics, optics or optoelectronics.

The invention relates more precisely to a method for determining the composition of a layer being made of a composed semiconductor material which comprises at least two elements and has an oxidation rate related to the concentration within said material of an element among said elements.

Background art

The determination of the composition of a layer relates more precisely to the determination of the distribution within said layer (for instance a silicium-germanium SiGe layer) of an element concentration (Ge concentration).

A wafer map cartography can thus be established, representing for instance said element concentration (Ge concentration) in a plurality of points within said layer (SiGe layer).

Indeed, and taking a SiGe layer as an example, the concentration of germanium may not be uniform within the layer (that is the Ge concentration may not be the same in all points of said SiGe layer).

Several techniques are known for determining the composition of a layer made of a composed semiconductor material comprising different elements.

These techniques include X-ray diffraction, Raman Spectroscopy, SIMS (Secondary Ion Mass Spectrometry), RBS (Rutherford BackScattering), AES (Auger Electron Spectroscopy), XPS (X-ray Photoelectron Spectroscopy), Ellipsometry.

The above mentioned techniques can hardly be used in the fabrication facilities for determining the uniformity of an element concentration (say Ge) all over a layer comprising said element (say a SiGe layer).

Indeed, X-ray diffraction is a time-consuming technique which necessitates several minutes for a single point to be analyzed.

Raman Spectroscopy, RBS, AES and XPS are also time-consuming techniques which cannot further be used in a clean room environment where the manufacturing of wafers comprising such a layer takes place.

SIMS is a destructive technique: a layer analysed with this technique cannot be reused for the manufacturing of a substrate.

Contrary to the preceding techniques, ellipsometry is a non destructive optical technique which can be used in production.

However ellipsometry is not adapted for analysing a layer on top of a complex and/or a thick structure.

There is thus a need for a non destructive and non time-consuming technique which could be usable in a clean room environment.

It has been proposed in "Determining the thickness and composition of SiGe heterostructures using an optical microscope", R.S Ferguson, K. Fobelets, M.M. Ahmad, D.J. Norris and J. Zhang, in Semiconductor Science and Technology 18, 390-392 (2003), a technique for determining the Ge composition in each SiGe layer of a SiGe heterostructure made of several SiGe layers having different thicknesses and Ge concentrations.

A two step process is disclosed in this document allowing the measurement of the thickness and the Ge composition of the different SiGe layers.

The first step is a beveling step intended for enlarging the layers so as make them visible under an optical microscope.

This step is a destructive one as a beveled layer cannot be used afterwards for the manufacturing of a substrate.

The second step is an oxidation step of the multilayer SiGe heterostructure so as to oxidize each SiGe layer.

It has indeed been reported that the oxidation of a SiGe layer strongly depends on the Ge concentration within the SiGe layer, in particular in the following documents:

- W.S. Liu, E.W. Lee, M-A. Nicolet, V. Arbet-Engels, K.L Wang, N.M. Abuhadba and C.R. Aita, in Journal of Applied Physics 71, 4015 (1992);
- S.K. Kang, D.H. Ko, K.C. Lee, T.W. Lee Y.H. Lee, T.H. Ahn, I.S. Yeo, S.H.Oh and C.G. Park, "Wet oxidation behaviours of polycristalline $Si_{1-x}Ge_x$ films", Journal of Vacuum Science and Technology A 19, 1617-22 (2001);
- A. Terrasi, S. Scalese, M. Re, R. Rimini, F. Iacona, V. Raineri and F. La Via, "Thermal Oxidation of Si (001) single crystal implanted with Ge ions", Journal of Applied Physics 91, 6754-60 (2002).

As a result of a low temperature oxidation of the multilayer SiGe heterostructure, a monotonic oxidation rate versus Ge concentration can be achieved.

Hence a different thickness of oxide is grown for each SiGe layer, dependent upon its Ge concentration.

The oxide thickness of each SiGe layer can be estimated from its color, as observed under an optical microscope.

It is thus possible, using a color scheme (for instance calibrated for different Ge concentrations and oxidation parameters), to estimate the Ge concentration of each SiGe layer of the SiGe heterostructure.

However the measuring accuracy is limited by the discrimination of the different colors.

Moreover if the oxide color depends on its thickness and its composition, it also depends upon the angle of incoming light.

This technique thus only provides an estimation of the Ge concentration.

Furthermore this technique cannot be used to measure the uniformity of Ge concentration within a SiGe layer. It is in particular impossible with such

a technique to establish a wafer map cartography representing the Ge concentration within a SiGe layer.

And, as it has already been mentioned, this technique is destructive, as the sample is bevelled and cannot be used after it has been analysed.

Hence there is a need for a method allowing the precise determination of the composition of a layer which in particular does not present the above mentioned drawbacks.

SUMMARY OF THE INVENTION

In order to fill the above mentioned need, the invention proposes a method for determining the composition of a layer, said layer being of a composed semiconductor material which comprises at least two elements and has an oxidation rate related to the concentration within said material of an element among said elements, said layer having a main surface, said method comprising the steps of:

- oxidizing the main surface of said layer for forming an oxide layer on said main surface;
- measuring the thickness of said oxide layer on said main surface;
- determining, from said thickness measurement, the concentration, within the composed material of said layer, of said element whose concentration is related to the oxidation rate.

Preferred but non restrictive forms of the method according to the invention are as follows.

- The oxidation step can be carried under conditions (notably at low temperature) chosen in order to avoid the segregation of said element whose concentration is related to the oxidation rate.
- The oxidation step can consist in a wet oxidation in an atmosphere charged with steam and may be carried, notably when the composed material is SiGe, at approximately 650°C during 10 minutes.
- The oxide layer having a free surface being the surface of the face of said oxide layer opposite said main surface, said thickness measurement

can consist in establishing a cartography of said oxide layer thickness over said oxide layer free surface.

- The thickness measurement can be realized by ellipsometry or reflectivity.
- The determination step can include the comparison of said thickness measurement to respective calibration thicknesses measured on oxide layers respectively grown on layers, made of said composed material and having known respective concentrations in said element whose concentration is related to the oxidation rate, by oxidation under the same oxidation conditions as those of said oxidation step.
- A cartography of the concentration within said layer of said element whose concentration is related to the oxidation rate can be determined from said comparison.
- The method can further comprise the steps of measuring the optical index of said oxide layer and calculating, from said optical index measurement, the composition of said layer in said element whose concentration is related to the oxidation rate.
- The method can further comprise the comparison of the compositions calculated from said optical index measurement and thickness measurement and the evaluation, from said comparison, of the strain repartition within said layer.
- The method can further comprise, prior to the oxidation step, a preliminary cleaning step of said main surface for preparing said main surface of said layer to be oxidized.
- The method can also further comprise a final oxide stripping step for removing said oxide layer formed during said oxidation step.
- Said composed material can be silicium-germanium SiGe.

BRIEF DESCRIPTION OF THE DRAWINGS

Other characteristics, purposes and advantages of the invention will appear to the reading of the following detailed description, with respect to the annexed drawings, given as non restrictive examples, in which:

- figures 1a and1b show various steps of a method according to a possible embodiment of the invention;
- figure 2 is a sketch representing the variation in oxide thickness according to the Ge concentration within a SiGe layer;
- figure 3 shows the oxide thickness grown on a SiGe material as a function of the Ge concentration.

DETAILLED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The invention relates to a method for determining the composition of a layer being made of a composed semiconductor material which comprises at least two elements and has an oxidation rate related to the concentration within said material of an element among said elements.

For the needs of this description, SiGe is taken as an example, but it has to be understood that the invention is in no way limited to this composed material and extends to any type of composed semiconductor material presenting the above mentionned properties.

A preferred embodiment of the method according to the invention will now be described below, which starts, with reference to figure 1a, from a wafer 1 consisting of a substrate 2 and, on the surface of said substrate 2, of a SiGe layer 3.

As non restrictive examples, such a susbstrate 2 may for instance be a silicon support substrate, or may comprise a silicon support substrate and a SiGe buffer layer having a concentration in germanium progressively increasing, or may comprise a silicon support substrate and an oxyde layer made of silicon oxide (also called silica or SiO_2).

The SiGe layer 3 has a main surface 7 which is the surface of the free face of said SiGe layer 3 opposite said substrate 2.

The invention aims at determining the composition of said SiGe layer 3, and more precisely, at determining the distribution within said SiGe layer 3 of the Ge concentration (or still said differently, at measuring the Ge concentration uniformity within the SiGe layer 3, and that on the whole of said wafer 1).

The method according to a preferred embodiment of the invention comprises a first step of oxidizing said SiGe layer 3 for forming an oxide layer 4 on top on said main surface 7 of said SiGe layer 3.

An important factor in oxidation processes involving Ge is its segregation behaviour. During oxidation of a SiGe layer, the germanium present in said layer tends to be pushed into regions thereof which are distant from the oxide layer being formed. Thus, germanium is observed to be segregated at the SiGe/oxide interface.

However, the germanium segregation phenomenon may be minimized, and the diffusion of Ge towards the surface limited, in particular by carrying out the oxidation under appropriately chosen conditions, notably at low temperature.

According to a preferred embodiment, wet oxidation is carried out by heating the wafer 1 in an atmosphere charged with steam at low temperatures so as to avoid the Ge segregation.

For instance, said oxidation step is thus carried at approximately 650°C for 10 minutes.

Figure 1b represents the wafer 1 after it has been oxidized. An oxide layer 4 is thus present on top of said SiGe layer 3.

More precisely, a composite SiGe oxide ($SiGeO_2$) layer 4 is formed on said main surface 7 of said SiGe layer 3.

As a result of the low temperature oxidation of the SiGe layer 3, a monotonic oxidation rate versus Ge concentration is achieved, as shown on figure 3.

Hence a particular thickness of oxide is grown, dependent upon the Ge concentration distribution within the SiGe layer 3.

More precisely, the oxide layer 4 presents a variable thickness according to a variable Ge concentration within the SiGe layer 3.

Let's consider the following caricatural example which schematically illustrates the growth of different thicknesses of oxide.

With reference to figure 2, a SiGe layer 5 comprises two halves 5a, 5b of different compositions, for instance a first half 5a having a Ge concentration of 21%, and a second half 5b having a Ge concentration of 19%, and a main surface 8.

After the oxidation step (represented by an arrow OX in figure 2), an oxide layer 6 is grown on the main surface 8 of said SiGe layer 5.

Said oxide layer 6 is made of two parts: a first part 6a grown on top of the first half 5a of the SiGe layer, the other part 6b grown on top of the second half 5b of the SiGe layer.

As the oxidation rate of each half 5a, 5b is related to the Ge concentration within said halves, said first and second parts 6a, 6b of said oxide layer 6 have different thicknesses t_a, t_b.

The thickness t_a of the first oxide part 6a on top of the first half 5a of the SiGe layer having a Ge concentration of 21% is thus more important than the thickness t_b of the second oxide part 6b grown on top of the second half 6 having a Ge concentration of 19%.

And, as mentioned above, these thicknesses t_a and t_b are directly correlated to the Ge concentration within each halves 5a, 5b of the SiGe layer 5.

And for instance, the measurement of the difference t_a-t_b in the oxide thickness of the oxide layer helps determining the Ge concentration variation between the halves 5a, 5b of the SiGe layer 5.

This very simplified example shows that it is possible, by measuring the thickness of the oxide layer in different points, to characterize the Ge concentration distribution within said SiGe layer 3.

More precisely, a legitimate assumption is made according to which the Ge concentration is uniform in-depth within the SiGe layer.

Returning to the description of the method according to a possible embodiment of the invention, said method comprises a second step of measuring the thickness of said oxide layer 4.

Said oxide layer 4 has a free surface 9 which is the surface of the face of said oxide layer 4 opposite said SiGe layer 3.

More precisely, this second step consists in establishing a cartography of said oxide layer 4 thickness, all over said oxide layer free surface 9.

Said second step can be realized using well known thickness measurement techniques, such as for instance reflectivity or ellipsometry.

As said oxide thickness in a point is directly correlated to the Ge concentration on the level of said point, a cartography can thus be established, representing said element concentration (Ge concentration) in a plurality of points within said layer (SiGe layer).

With the in-depth uniformity assumption, the Ge concentration is considered as being the same for all the points within the SiGe layer which are beneath a point where a thickness measurement is realized.

The thickness cartography allows establishing a 2D Ge concentration cartography which is, under said assumption, valid for all depths within the SiGe layer.

For this purpose, the method according to a possible embodiment of the invention comprises a third step of determining, from said thickness measurement, the Ge composition within the SiGe layer.

In order to calculate the Ge concentration within the SiGe layer, said method makes use of a preliminary calibration step whereby different calibration thicknesses are measured on oxide layers respectively grown on SiGe layers having known respective Ge concentrations by oxidization under the same oxidation conditions as those of said oxidation step (such as in particular a given temperature and a given duration).

Thus, such a calibration step makes it possible to have a preliminary knowledge of the Ge concentration according to the oxide thickness grown under given oxidation conditions.

Using said calibration step and said thickness measurement step, it is thus possible to determine the Ge concentration within the SiGe layer analysed by the method according to the invention.

Of course, it has to be noted that the concentration measurement accuracy is directly related to the thickness measurement accuracy.

The method according to the invention is thus particularly precise.

In particular, it has been determined that a variation of 1% of the Ge concentration within a SiGe layer results in a difference of 45 Angstroms in the oxide thickness.

Ellipsometry allows the measurement of thickness variation as low as 5 Angstroms. It is thus estimated that, thanks to the method according to an embodiment of the invention, a minimum distinguishable Ge concentration variation is approximately 0.11%.

Determinination of the strain repartition within the analyzed layer

It has however to be noted that the oxide thickness grown on a SiGe layer may also be influenced by the strain of said SiGe layer.

If such an influence is particularly limited, and can thus be ignored, when said SiGe layer is predominantly relaxed (having for instance a relaxation above 90%), said influence may have to be taken into consideration when said SiGe layer is not predominantly relaxed (as it is the case in particular when said SiGe layer presents important strain variations).

Hence the thickness measurement may depend at the same time on the Ge concentration and on the strain within the SiGe layer.

According to another embodiment, the method according to the invention may further comprise the step of measuring the optical index of the oxide layer 4 (see figure 1b) and the step of determining, from said optical index measurement, the Ge concentration within the SiGe layer 3.

Indeed, the optical index measurement of the oxide layer makes it possible to determine precisely the Ge concentration within said oxide layer.

For this purpose, preliminary calibrations linking the oxide optical index to its composition may be used for determining said Ge concentration.

As a variation in the Ge composition of the SiGe layer 3 results in a variation in the Ge composition of the oxide layer 4 grown on the main surface 7 of said SiGe layer 3, it is thus possible to determine, from said oxide layer optical measurement, the Ge concentration distribution within the SiGe layer 3.

Ellipsometry may be the technique used for measuring the optical index of the oxide layer

As the SiGe layer strain does not influence the optical index measurement, the Ge concentration distribution determined thanks to this optical technique is really accurate.

By comparing the Ge compositions determined from both the optical index and thickness measurements, it is possible to determine up to what point the strain influences the oxide thickness.

Hence, it is possible from said comparison to discriminate the effects of the composition and the strain on the oxide thickness and thus to evaluate the strain repartition within said layer.

Cleaning step

According to an advantageous embodiment of the method according to the invention, said method comprises a preliminary step consisting, before the oxidation step, in a cleaning step for preparing said main surface of the SiGe layer to be oxidized.

Said cleaning step advantageously helps increasing said SiGe layer sensitivity to oxidation.

Said cleaning step may for instance consist in a standard HF cleaning step or a standard RCA cleaning step.

Oxide stripping step

According to another embodiment, the method may also comprise a oxide stripping step for removing the oxide layer formed during said oxidation step.

Said oxide stripping step is preferentially carried out in a traditional way. For this purpose, the substrate can be plunged during a few minutes in a solution of hydrofluoric acid to 1% or 10% for example.

The method according to the invention finally allows obtaining a composition cartography of the analysed layer, and that on the whole of a wafer which comprises on its top said analysed layer.

The method according to the invention is also non destructive; the wafer comprising the analysed layer may be used after its composition has been analysed and the oxide layer removed.

Indeed, only a small portion (typically a few hundreds of Angstroms) of the analysed layer is consumed during the oxidation step.

Moreover, as the composition measurement accuracy is determined by the thickness measurement accuracy, the thickness of the consumed portion of the analysed layer can be varied, and even minimized.

The determination of the composition of the layer according to the invention can further be carried within a clean room environment.

The invention may for instance be utilized for characterizing the Ge composition and/or the strain of a SiGe layer epitaxially grown on a Si substrate.

However the invention is in no way limited to this application and may of course be employed whenever a layer composition and/or strain analysis has to be realized.

THE CLAIMS

1. A method for determining the composition of a layer (3, 5), said layer being of a composed semiconductor material which comprises at least two elements and has an oxidation rate related to the concentration within said material of an element among said elements, said layer having a main surface (7, 8),
said method comprising the steps of:

- oxidizing the main surface (7, 8) of said layer (3, 5) for forming an oxide layer (4, 6) on said main surface;
- measuring the thickness (t_a, t_b) of said oxide layer (4, 6) on said main surface (7, 8);
- determining, from said thickness measurement, the concentration, within the composed material of said layer (3, 5), of said element whose concentration is related to the oxidation rate.

2. The method according to the preceding claim wherein the oxidation step is carried according to conditions chosen in order to avoid the segregation of said element whose concentration is related to the oxidation rate.

3. The method according to the preceding claim wherein the oxidation step consists in a wet oxidation in an atmosphere charged with steam.

4. The method according to any of the two preceding claims wherein the composed material is silicium-germanium SiGe and the oxidation step is carried at approximately 650°C during 10 minutes.

5. The method according to any of the preceding claims wherein said oxide layer (4) has a free surface (9), said free surface (9) being the surface of the face of said oxide layer (4) opposite said main surface (7),

and said thickness measurement consists in establishing a cartography of said oxide layer thickness over said oxide layer free surface (9).

6. The method according to the preceding claim wherein said thickness measurement over said oxide layer free surface (9) is realized by ellipsometry or reflectivity.

7. The method according to any of the preceding claims wherein the determination step includes the comparison of said thickness measurement to respective calibration thicknesses measured on oxide layers respectively grown on layers, made of said composed material and having known respective concentrations in said element whose concentration is related to the oxidation rate, by oxidization under the same oxidation conditions as those of said oxidation step.

8. The method according to the preceding claim wherein said oxidation conditions include a given temperature and a given duration.

9. The method according to any of the two preceding claims wherein a cartography of the concentration within said layer of said element whose concentration is related to the oxidation rate is determined from said comparison.

10. The method according to any of the preceding claims further comprising the steps of measuring the optical index of said oxide layer (4, 6) and determining, from said optical index measurement, the concentration, within the composed material of said layer (3, 5), of said element whose concentration is related to the oxidation rate.

11. The method according to the preceding claim, wherein said composition calculation is made on the basis of preliminary calibrations linking the oxide optical index to the oxide composition.

12. The method according to any of the two preceding claims further comprising the comparison of the compositions determined from both said optical index measurement and thickness measurement and the evaluation, from said comparison, of the strain repartition within said layer (3, 5).

13. The method according to any of the preceding claims further comprising, prior to the oxidation step, a preliminary cleaning step of said main surface (7, 8) for preparing said main surface of said layer (3, 5) to be oxidized.

14. The method according to any of the preceding claim further comprising a final oxide stripping step for removing said oxide layer (4, 6) formed during said oxidation step.

15. The method according to any of the preceding claims wherein said composed material is silicium-germanium SiGe.

16. The method according to the preceding claim wherein, during said step of measuring the thickness of the oxide layer, a difference of 45 Angstroms in the oxide thickness is measured for a variation of 1% in the Ge concentration within said SiGe layer.

17. A method for determining the composition of a layer (3, 5), or both the composition and the strain of said layer (3, 5), said layer being of a composed semiconductor material which comprises at least two elements and has an oxidation rate related to the concentration within said material

of an element among said elements, said layer having a main surface (7, 8), said method comprising the steps of:

- oxidizing the main surface (7, 8) of said layer (3, 5) for forming an oxide layer (4, 6) on said main surface;
- measuring the thickness (t_a, t_b) of said oxide layer (4, 6) on said main surface (7, 8);
- and
  - the step of determining, from said thickness measurement, the concentration, within the composed material of said layer (3, 5), of said element whose concentration is related to the oxidation rate;
  - or the steps of:
    - determining, from said thickness measurement, the concentration, within the composed material of said layer (3, 5), of said element whose concentration is related to the oxidation rate;
    - measuring the optical index of said oxide layer (4, 6);
    - determining, from said optical index measurement, the concentration, within the composed material of said layer (3, 5), of said element whose concentration is related to the oxidation rate;
    - comparing the composition determined from said optical index measurement to the composition determined from said thickness measurement;
    - evaluating, from said comparison, the strain repartition within said layer (3, 5).

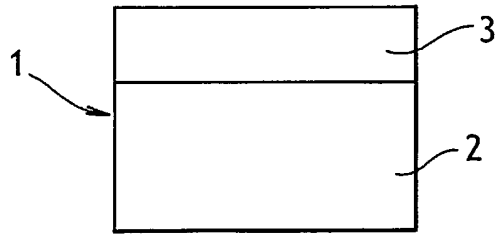


FIG.1a

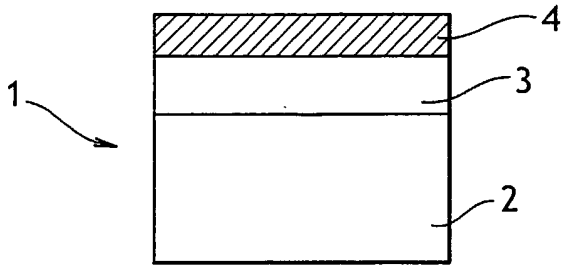


FIG.1b

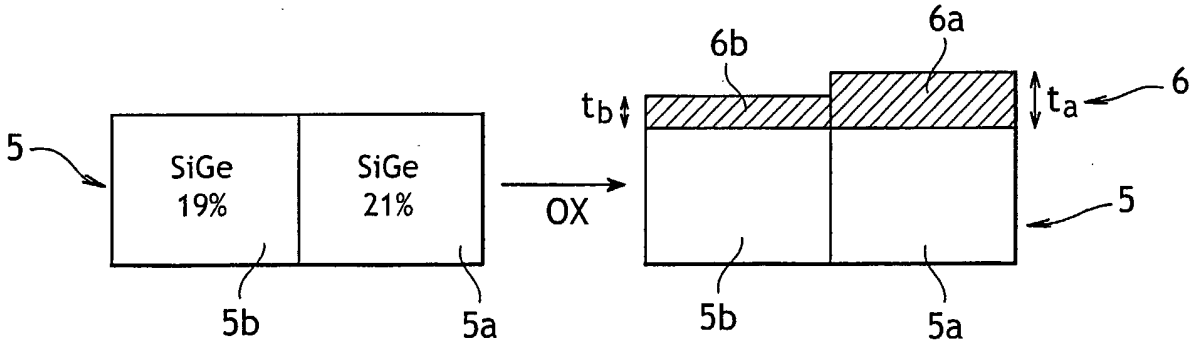


FIG.2

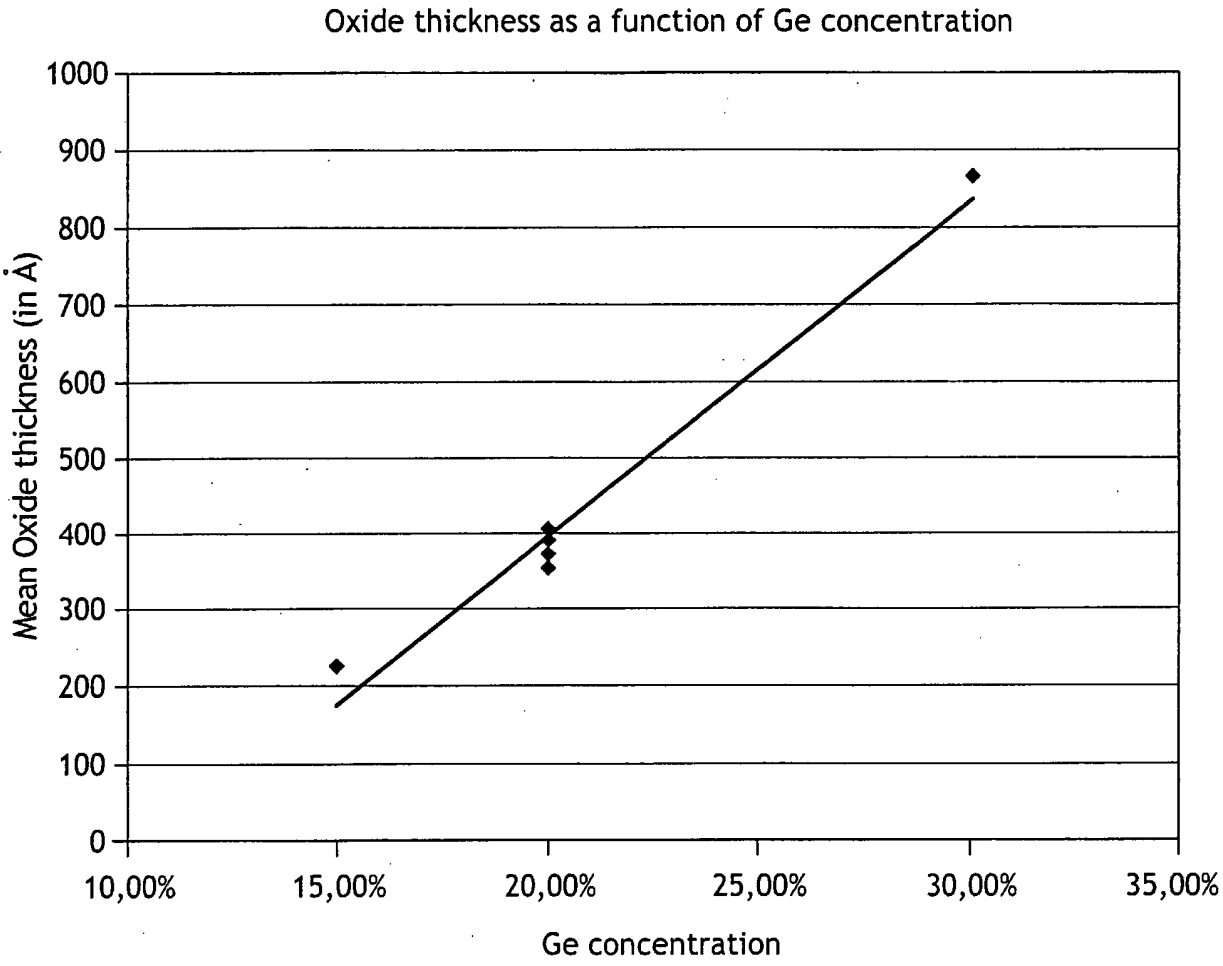

Oxide thickness as a function of Ge concentration
Mean Oxide thickness (in Å)
1000
900
800
700
600
500
400
300
200
100
0
10,00%
15,00%
20,00%
25,00%
30,00%
35,00%
Ge concentration

FIG.3

INTERNATIONAL SEARCH REPORT

PCT/IB2004/002606

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 H01L21/66

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 H01L G01N

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, INSPEC, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	FERGUSON R S ET AL: "Determining the thickness and composition of SiGe heterostructures using an optical microscope" SEMICONDUCTOR SCIENCE AND TECHNOLOGY IOP PUBLISHING UK, vol. 18, no. 4, 1 April 2003 (2003-04-01), pages 390-392, XP002324595 ISSN: 0268-1242	1-3,7,8, 15
Y	the whole document	4-6, 9-11,13, 14,16

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"A" document defining the general state of the art which is not considered to be of particular relevance

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