METHOD FOR EPIREADY SURFACE TREATMENT ON SIC THIN FILMS

Field of the invention and prior art

The present invention relates in general to the treatment of semiconductor materials intended for microelectronics and/or optoelectronics applications.

In particular, it relates to a method of preparing the surface of a thin film of thickness in the range 1 nanometer (nm) or a few tens of nm to 100 nm or a few hundred nm, for example 400 nm or 500 nm.

In particular, the invention relates to a film of monocrystalline silicon carbide with a view to performing epitaxial growth thereon.

The film may be a silicon carbide film transferred onto a further material (silicon, monocrystalline or polycrystalline SiC covered with an oxide or other film: deposited oxide, nitride, etc).

In particular, the invention is applicable to SiC substrates, for example of polytype 4H, which are used for epitaxial growth with a view to fabricating electronic power components.

In order to obtain good quality epitaxy, the starting surface must be free of defects and must be as smooth as possible.

A thin layer transfer method is known, in particular for transferring thin SiC films, and is known as the "Smart-Cut" method (or substrate fracture method); it is described, for example, in the article by A. J. Auberton-Hervé et al entitled "Why can Smart-Cut change the future of microelectronics?", Int Journal of High Speed Electronics and Systems, Vol. 10, no. 1, 2000, pp 131-146. After fracture, that method leaves roughness of about 5 nm rms (root mean square), which is not very compatible with epitaxial growth.

That roughness has to be reduced to about 1 nm to 2 nm rms by applying thermal oxidation type treatments (known as annealing) and/or ion etching. However, those

techniques cannot produce the desired final roughness (0.1 to 0.2 nm rms).

The annealing step does not consume sufficient material to significantly reduce the roughness since thermal SiC oxidation is very slow, especially on the silicon face.

Further, chemical-mechanical polishing (CMP) of SiC is difficult to carry out since the chemical reactivity of the polished surfaces is low compared with materials such as silicon. In addition, the removal rate is very low, of the order of 10 nm per hour, as opposed to 50 nm per minute for silicon polishing.

Further, the mechanical hardness of SiC is extremely high and the use of "diamond" abrasives or certain abrasives that are known for polishing silicon may result in scratches.

Thus, it is difficult to use an abrasive which can produce a sufficiently high removal rate without creating said scratches and defects. Hence, SiC polishing methods are often very lengthy (several hours) and abrasives based on diamond particles cannot produce a roughness of less than 1 nm rms.

For these two reasons, SiC polishing techniques are very particular. Currently, few SiC substrate polishing methods are known.

United States document US-A-5 895 583 describes a successive polishing method: several steps are necessary to remove work hardened zones generated by each polishing step. That method uses abrasives based on diamond-containing particles of decreasing diameters.

French patent application No. 02/09869 dated 2nd August 2002 describes a method employing a mixture of abrasives (diamond/silica) that can produce roughness compatible with molecular bonding.

Techniques other than polishing exist to produce low roughness: the majority thereof are based on bombarding the surface with ions from a plasma (RIE) or a beam (for

example gas cluster ion beam), a technique described in US-2002 0014407. Those techniques are of interest as regards the removal rates, but the surface condition is often too rough for epitaxy and in particular, the surface cannot be smoothed.

Thus, there is a problem with developing a novel method of treating or preparing the surface of a film, in particular a silicon carbide film.

A further problem is to find a method of treating films, in particular silicon carbide films, that can produce low roughness, and/or that can produce a sufficient removal rate without creating scratches or defects.

There is also a further problem, namely to develop a method of treating films, in particular silicon carbide films, which can produce low roughness, preferably of less than 15 angstroms (Å) or 10 Å rms or 5 Å rms or 1 Å rms, compatible with epitaxial growth.

Description of the invention

The invention provides a method of preparing the surface of a wafer of material, the method comprising:

- a step of annealing in an oxidizing atmosphere;
- a step of polishing with an abrasive based on particles of colloidal silica.

The combination of these two steps can produce a satisfactory surface condition, in particular in the case of silicon carbide.

The annealing step can produce roughness of less than or of the order of 20 Å rms. It can be carried out at a temperature in the range 1100°C to 1300°C, for a period in the range 1.5 hours (h) to 2.5 h, for example.

After the annealing step, a surface deoxidation step may be carried out, for example of the type employing a chemical such as hydrofluoric acid. In the case of ion etching, an RCA (SC1, SC2) type chemical cleaning step may be carried out after the annealing step.

Polishing is, for example, carried out with a colloidal SYTON W30 type colloidal silica and with a polishing head rotating at a speed in the range 10 revolutions per minute (rpm) to 100 rpm.

A chemical cleaning step can be carried out after polishing, for example using hydrofluoric acid.

Finally, an ion etching step can also be carried out, for example before the annealing step.

Brief description of the Figures

- Figures 1A and 1B are diagrams of polishing apparatus.

Detailed description of implementations of the invention

An implementation is described below, relating to the silicon face of a SiC film. It should be recalled that SiC is a polar material and thus comprises two faces constituted by different atoms (a silicon face and a carbon face).

Said thin film is, for example, obtained by a substrate fracture method ("Smart-Cut") such as that described in the above-cited article by A. J. Auberton-Hervé et al.

Firstly, heat treatment is carried out on said thin film in an oxidizing atmosphere, for example at a temperature in the range 100°C or 1150°C to 1300°C for a period in the range 1 h to 3 h. This annealing step in an oxidizing atmosphere can produce roughness of the order of 2 nm rms. One example of a device for carrying out said annealing step is described in "Thermal and Dopant Processes", Chapter 4, Advanced Semiconductor Fabrication Handbook, ICE, 1998.

The treated surface can be deoxidized by chemical etching, for example using 10% hydrofluoric acid.

Subsequently, CMP (chemical-mechanical polishing) is carried out, for example using an IC1000 pad (distributed by RODEL, compressibility = 3%) and an abrasive based on

colloidal silica SYTON W30 (or LuDox) type particles (pH = 10.2, viscosity = 2 megapascal.seconds (mPs.s), mean particle size = 125 nm, containing 30% by weight of SiO_2).

A polishing head 10 into which the substrate 12 to be polished is inserted, is shown in Figure 1A. Figure 1B shows the polishing head, the substrate 12 to be polished, and also a plate 16 and a polishing pad 14. A liquid abrasive is injected into the head, for example via a side conduit 18. A pressure 20 and a movement represented by an arrow 22 are applied to the head 10 to carry out polishing.

Optionally, chemical cleaning using hydrofluoric acid can prevent crystallization of the abrasive on the surface.

Such a method can produce a surface with roughness that enables it to be used for good quality homoepitaxy (SiC on SiC epitaxy), and optionally also heteroepitaxy (AlN, AlGaN or GaN on SiC).

In an example relating to a thin film of 4H type SiC (obtained by the "Smart-Cut" method):

- an annealing step was carried out in an oxidizing atmosphere (2 h at 1150°C, for example), followed by surface deoxidation in 10% HF;
- followed by polishing the surface by CMP. Polishing was carried out under the following conditions:
  - using a rotating polishing plate onto which a polishing head had been applied, also rotary, the speed of rotation being of the order of 60 rpm (this speed can be in the range 10 rpm to 100 rpm); a pressure of 0.75 bars (which can be in the range 0.1 bar to 1 bar) was applied to the head;

- the pad used was a "hard" type IC1000 pad distributed by RODEL, with a slurry which was a SYTON W30 type colloidal silica.

The polishing time was 15 minutes (min) to 30 min.

The roughness obtained after polishing was of the order of 3 Å rms.

Final cleaning was carried out using deionized water with a 10% HF bath for 10 minutes.

Table I below summarizes the results obtained for thin SiC films under different conditions.

I	II	III	IV	V (nm)	VI
1	None			5.02	
2	Ion etching + anneal 1150°C, time: 2h			3.02	
3	Ion etching + anneal 1150°C, time: 2h	30 min/70 rpm/0.75b	UR 100/ glansox	0.583	soft pad
4	Anneal 1150°C, time: 2h	30 min/60 rpm/0.75b	UR 100/ glansox	1.246	soft pad
5	Anneal 1150°C, time: 2h + ion etching			1.12	
6	Anneal 1150°C, time: 2h			2.54	annealing sufficient to reduce roughness
7	Anneal 1300°C, time: 1h			1.64	
8	Anneal 1150°C, time: 2h	15 min/25 rpm/0.6 b	IC1000/ syton	0.267	Fairly slow speed of rotation
9	Anneal 1150°C, time: 2h	30 min/60 rpm/0.75 b	IC1000/ syton	0.101	
10	Anneal 1150°C, time: 2h	15 min/60 rpm/0.75 b	IC1000/ syton	0.155	worn pad
11	Anneal 1150°C, time: 2h	15 min/60 rpm/0.75 b	IC1000/ syton	0.064	new pad

TABLE I

In the table, column I indicates the test number and column II shows the nature of the treatment carried out prior to CMP polishing. Tests 2 and 3 underwent ion etching followed by annealing at 1150°C for two hours;

for test n° 5, the treatment was annealing at 1150°C for two hours followed by ion etching.

For tests 4, 6 and 8 to 10, only annealing at 1150°C for two hours was carried out.

Column III gives the conditions for carrying out CMP polishing: time, rotation speed, applied pressure.

Column IV shows the nature of the pad and the abrasive mixture.

Column V shows roughness measurements over a surface area of 5 micrometers (μm) × 5 μm.

Any comments are shown in column VI.

This table shows that the combination of an annealing step followed by polishing can substantially reduce the roughness of the initial film to less than 2 nm rms (tests 3-5 and 7-11), 1.5 nm (tests 3-5, 8-11), 1 nm rms (tests 3, 8-11), 0.5 nm rms (tests 8-11), or 0.1 nm rms (test 11).

Thus, the invention can produce a silicon carbide film with a roughness of less than 2 nm rms, less than 1 nm rms, less than 0.5 nm rms, or less than 0.1 nm rms.

Use of prior ion etching, as in test n° 3, also improves this result.

The best results appear to be obtained with an IC1000 pad and with a Syton W30 abrasive solution.

Table II shows more detailed conditions concerning tests n° 10 and 11.

Test n° 10 was carried out using an "S107" plate while test n° 11 was carried out using an "S126" plate.

Table II compares roughness using plates S126 and S107.

Two types of measurement were carried out: scanning a certain surface area (column S, surface area indicated in square micrometers (μm^2)), and point measurements (column B, surface measurements indicated in μm × μm).

The last three columns show, in angstroms: roughness as a root mean square value (rms), mean roughness (Ra), and maximum roughness (Rmax)

The values shown in Table I for tests 10 and 11 respectively correspond to those shown in the third and seventh lines of Table II (rms column).

Comparison of roughness using plates S126 and S107					
Plate	S (μm^2)	B µm	rms (Å)	Ra (Å)	Rmax (Å)
S107	1 µm × 1 µm		0.97	0.77	14.7
		0.3 × 0.9	0.7	0.55	8.2
	5 µm × 5 µm		1.55	1.21	16.1
		3 × 1	1.38	1.06	12.1
S126	1 µm × 1 µm		0.37	0.28	7
		0.6 × 0.7	0.34	0.27	3
	5 µm × 5 µm		0.64	0.5	29.7
		1.5 × 4	0.31	0.25	4.9

TABLE II

The results shown in these tables indicate that the method of the invention can produce a surface that is ready for epitaxy ("epiready") on thin SiC films, using a rapid technique, which employs steps and machines that are standard in microelectronics. The smoother the SiC surface and the lower its roughness, the better the quality of the epitaxy, which can substantially increase the yield of electronic components produced on the thin film.

This surface preparation method of the invention, comprising an annealing step followed by polishing, can thus produce a good quality surface that is not rough and is smooth.

The example of a polytype 4H SiC substrate has been given, but the invention is also of application to a polytype 6H or 3C SiC substrate.

CLAIMS

1. A method of preparing the surface of a wafer of silicon carbide, the method comprising:

- a step of annealing in an oxidizing atmosphere so as to reduce roughness at the surface of the wafer;
- a step of polishing with an abrasive based on particles of colloidal silica.

2. A method according to claim 1, in which the annealing step is carried out at a temperature in the range 1000°C to 1300°C.

3. A method according to claim 1 or claim 2, in which the annealing step is carried out for a period in the range 1 h to 2.5 h.

4. A method according to any one of claims 1 to 3, further comprising, after the annealing step, a step of deoxidation of the surface or an RCA (SC1, SC2) type chemical cleaning step in the case of ion etching.

5. A method according to claim 4, in which the deoxidation step is carried out in hydrofluoric acid.

6. A method according to any one of claims 1 to 5, in which after polishing, a chemical cleaning step is carried out.

7. A method according to claim 6, in which the cleaning step employs hydrofluoric acid.

8. A method according to any preceding claim, in which the material is a semiconductor material.

9. A method according to any preceding claim, in which polishing is carried out with a SYTON W30 type colloidal silica.

10. A method according to any preceding claim, in which polishing is carried out with a polishing head rotating at a rate in the range 10 rpm to 100 rpm.

11. A method according to the preceding claim, in which a pressure in the range 0.1 bar to 1 bar is applied to the polishing head.

12. A method according to any preceding claim, in which polishing is carried out for a period in the range 15 minutes to 30 minutes.

13. A method according to any preceding claim, in which polishing is carried out with an IC1000 type polishing pad.

14. A method according to any preceding claim, further comprising an ion etching step.