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D E C I S I O N
of 11 October 2000

Case Number: T 0547/95 - 3.4.3

Application Number: 89105425.6

Publication Number: 0335313

IPC: H01L 21/306

Language of the proceedings: EN

Title of invention:
Method of manufacturing semiconductor device and apparatus for use in practicing the method

Applicant:
KABUSHIKI KAISHA TOSHIBA

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step (no) - ranges of parameters accessible through routine experimentation"

Decisions cited:
-

Catchword:
-

Case Number: T 0547/95 - 3.4.3

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D E C I S I O N
of the Technical Board of Appeal 3.4.3
of 11 October 2000

Appellant: KABUSHIKI KAISHA TOSHIBA
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 9 February 1995 refusing European patent application No. 89 105 425.6 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: R. K. Shukla
Members: G. L. Eliasson
M. J. Vogel

Summary of Facts and Submissions

I. European patent application No. 89 105 425.6 was refused in a decision of the examining division dated 9 February 1995. The ground for the refusal was that the subject matter of claims 1 to 12 lacked an inventive step having regard to the prior art documents

D1: Solid State Technology, December 1985, pages 51-59;

D3: EP-A-0 052 227; and

D4: US-A-4 605 479.

II. The appellant (applicant) lodged an appeal on 10 April 1995, paying the appeal fee the same day. A statement of the grounds of appeal was filed on 9 June 1995 together with new claims 1 to 12. Oral proceedings were requested in case the Board intended to dismiss the appeal.

III. In response to a communication annexed to summons to oral proceedings, the appellant filed with the letter dated 11 September 2000 new claims 1 to 12.

IV. At the oral proceedings held on 11 October 2000, the appellant requested that the decision under appeal be set aside and a patent be granted on the basis of the following documents:

Claims:	1 to 12 as filed with the letter dated 11 September 2000
Description:	Pages 1 to 17 as originally filed
Drawings:	Sheets 1/6 to 6/6 as originally filed.

V. Independent claims 1 and 7 read as follows:

"1. A method of manufacturing a semiconductor device, comprising the steps of

removing a native silicon oxide film formed on a silicon layer of an intermediate semiconductor device by using active species derived form a mixed gas of a gaseous halogen compound and gaseous oxygen, under the condition that the ratio Y of flow of said gaseous oxygen to that of the mixed gas and the temperature T (°C) of the intermediate semiconductor device both are controlled so as to satisfy the condition

$$Y \geq -0.13\ T + 106.3,$$

and to make the ratio of the etching speed of said silicon layer and the etching speed of said silicon oxide film not exceeding the value of 5, for keeping a difference between the etching speeds of said silicon layer and said silicon oxide film as small as possible; and

subsequently forming a covering film on said silicon layer with said silicon oxide film removed therefrom and without exposing said silicon layer to the atmosphere after removal of said silicon oxide film and prior to formation of said covering film."

In the last paragraph of the above wording, the spelling of "silicion" in "silicion oxide" has been corrected by the Board to read "silicon".

"7. An apparatus for forming a covering film on a silicon surface of an intermediate semiconductor device, comprising

a casing (10; 3-4, 3-10; 8-1, 8-8; 10-1, 10-2) for housing an intermediate semiconductor device (11; 3-3, 3-14; 8-2, 8-10; 10-12, 10-13);

an introduction tube (17) for introducing a flow of mixed gas comprising gaseous oxygen and a gaseous halogen compound into a discharge chamber (14; 3-6; 8-7; 10-7) connectable to said casing, for generating an active species capable to perform etching of a native silicon oxide film formed on the silicon layer of said intermediate semiconductor device when disposed in said casing;

first regulating means (21; 3-21; 8-21; 10-21) for regulating the ratio of the flow of gaseous oxygen to that of the gaseous halogen compound;

heating means (13; 3-5; 8-4; 10-9) for heating the intermediate semiconductor device when disposed in said casing;

second regulating means (22; 3-22; 8-22; 10-22) for regulating the temperature of said intermediate semiconductor device when disposed in said casing and heated by said heating means; and

cover film forming means for forming a cover layer on the silicon layer of said intermediate semiconductor device when disposed in said casing;

wherein said first regulating means and said second regulating means are operable for cooperatively controlling both the ratio Y of flow of said gaseous oxygen to that of the mixed gas and the temperature T (°C) of the intermediate semiconductor device when disposed in said casing, so as to satisfy the condition

$$Y \geq -0.13T + 106.3,$$

and to make the ratio of the etching speed of said silicon layer and the etching speed of said silicon oxide film not exceeding the value of 5, for keeping a difference between the etching speeds of said silicon layer and said silicon oxide film as small as possible."

In the above wording, the spelling of "menas" in "forming menas" has been corrected by the Board to read "means".

VI. The appellant presented essentially the following arguments in support of his request:

(a) In contrast to the method of claim 1 of the application in suit, document D1 does not relate to the removal of a native oxide film from a silicon surface, since document D1 discloses a substrate pretreatment step of using a CF_4/O_2 glow discharge which itself produces a 20 Å thin oxide layer on the silicon surface (cf. D1, page 55, last paragraph).

(b) Starting from document D1, a skilled person would not have any reason to seek an etching process having a ratio of the etching speed of silicon to the etching speed of silicon oxide less than 5, since document D1 does not give any indication that a small value of the above-mentioned ratio would be desirable.

(c) A skilled person faced with the object of improving the substrate pretreatment step of document D1 in such a manner that all native oxide is removed, would not consider document D3, since the latter document is not related to the removal

of native oxide. Instead, document D3 relates to finding a method of etching of silicon nitride using a CF_4/O_2 gas mixture where the etch rates of silicon oxide and polysilicon should be kept as low as possible (cf. D3, page 8, lines 8 to 15).

(d) Even assuming the hypothetical situation that a skilled person had combined the teaching of document D1 with that of document D3, the resulting method would still fail to meet the condition "$Y \geq -0.13T + 106.3$". Thus, in order to arrive at a method falling within the scope of claim 1, the skilled person would have to investigate further as to how to vary the parameter values disclosed in document D3. Such an investigation cannot be regarded as obvious to the skilled person.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is therefore admissible.

2. *Inventive step*

The main issue in the present appeal is whether the subject matter of claim 1 involves an inventive step or not.

2.1 Document D1 discloses the selective deposition of tungsten on silicon using low pressure CVD. Prior to deposition, native oxide is removed from the silicon surface (cf. D1, pages 55 and 56, "Substrate Pretreatment") For the removal of the native oxide, a dry etch of an "oxygen-rich mixture" of CF_4 and O_2 is used. The dry etching process was found to produce a

2 nm thin oxide layer on the exposed silicon. This oxide layer however turned out to have a negative effect on the quality of the subsequently deposited tungsten film (cf. D1, Figure 11). It is furthermore stressed in document D1 that the cleaning/treating of the silicon surface prior to deposition of W should be carried out in-situ (cf. D1, page 56, "Substrate Pretreatment", last sentence).

2.1.1 The appellant argued that document D1 is not related to the removal of a native oxide film, since the method disclosed therein produced a residual oxide film rather than removing an oxide film (cf. point VI(a) above).

In this connection, in the Board's view, document D1, not withstanding the fact that a thin residual oxide film is produced by the etching treatment described therein, is concerned with a preclean treatment involving etching using a gas mixture of CF_4 and O_2, as the application in suit. Moreover, it is stressed in document D1 that a silicon surface free of oxide is required in order to obtain optimal conditions for the selective growth of tungsten (cf. page 55, "Substrate Pretreatment", second paragraph; Figure 9; page 56, paragraph bridging both columns). Contrary to the submission of the appellant, therefore, document D1 is relevant in the consideration of inventive step of the claimed subject matter.

2.2 The method of claim 1 thus differs from that of document D1 in that the ratio Y of flow of oxygen to that of the mixed gas and the temperature T of the intermediate semiconductor device are both controlled so as to satisfy the condition

$Y \geq -0.13\ T + 106.3$;

and that the ratio of the etching speed of silicon and the etching speed of silicon oxide is not exceeding the value of 5.

In document D1, the only detail about the etching process is that the mixture of CF_4 and O_2 is "oxygen-rich".

2.3 As is apparent from Figure 10 of document D1, the dry etching process used in document D1 does not produce a satisfactory result. It is however pointed out in document D1 that an optimum preclean treatment would be one resulting in no oxide on the surface just prior to the tungsten deposition and that such a method of cleaning in situ prior to CVD of tungsten must be developed for shallow junction, micrometer-sized feature devices and beyond (cf. D1, page 56, paragraph bridging both columns).

Therefore, the objective technical problem addressed by the present invention is to find process parameters of the CF_4/O_2 etching process for satisfactorily removing native oxide on an exposed silicon surface to be covered with a tungsten film. Also, in case of shallow junction devices or devices with micrometer-sized features, it would be evident to provide a preclean treatment that would attack or remove the exposed silicon as little as possible.

Moreover, the CF_4/O_2 etching process is described in document D1 as a "pre-deposition cleaning step" (cf. D1, page 55, "Substrate Pretreatment", first paragraph). Thus, considering a "cleaning" process to be basically non-destructive, the skilled person would in this context interpret "pre-deposition cleaning step" to mean a process of removing contaminants, such as native oxide, from a silicon surface while, at the same time, ensuring that the silicon surface to be

cleaned remains as intact as possible after the cleaning process. Contrary to the submissions made by the appellant (cf. item IV(b) above), therefore, the Board finds that the skilled person would in this situation look for an etching process where the ratio of the etching speed of silicon and the etching speed of silicon oxide is small.

2.4 Document D3 discloses a method of dry etching silicon oxide using CF_4 and O_2 (cf. page 7, line 8 to page 8, line 16). Using the flows of 100 SCCM of O_2 and 18.8 SCCM of CF_4 at a substrate temperature of 150°C, the etch rates of 10 nm/min for silicon oxide and 9 nm/min for polysilicon are observed (cf. D3, page 7, lines 12 to 23; page 8, lines 6 to 9). It is furthermore taught in document D3 that the process parameters such as power, pressure, temperature and CF_4/O_2 flow ratio all may be adjusted to change the etch rates, although some recommendations are given as to preferred ranges of the parameters (cf. D3, page 9, line 16 to page 10, line 3).

2.5 A skilled person faced with the task of improving the pretreatment process involving etching used in document D1 would therefore in the Board's view consider the teaching of document D3 to optimize the process parameters. Although document D3 primarily relates to etching of silicon nitride and not of native silicon oxide, as correctly pointed out by the appellant (cf. point VI(c) above), the method of document D3 is non-selective with respect to silicon nitride, silicon oxide and polysilicon (cf. e.g. abstract), and concerns the same gas mixture of CF_4 and O_2 as in document D1. Since the method of document D3 succeeds in obtaining almost equal etch rates for silicon oxide and silicon, the skilled person would in the Board's view have sufficient incentive to consult document D3 in order to optimize the process of document D1. Moreover, as

document D1 does not disclose any process parameters except for the statement that the mixture of CF_4 and O_2 was "oxygen-rich", the skilled person has to consult other documents for finding suitable process parameters for a plasma etching process using CF_4 and O_2.

2.6 Thus, applying the process parameters disclosed on page 7, line 8 to page 8, line 16 of document D3 to the pre-deposition cleaning step of document D1, one arrives at a process where the ratio of etch rate of silicon to that of silicon oxide is 9/10=0.9, i.e. a value well below 5. The flow rates (100 SCCM of O_2 and 18.8 SCCM of CF_4) gives the ratio Y = 100/118.8 = 84.2%. Using the value of T (150°C) in the right-hand side of claimed condition $Y \geq -0.13 T + 106.3$, one arrives at a value of 86.8%.

The parameters of document D3 thus do not fulfil the condition $Y \geq -0.13 T + 106.3$ although the ratio of etch rates lies within the claimed range.

2.7 It is known in the art of dry etching that the parameters have to be adjusted for each reaction chamber, since a set of process parameters, such as temperature and gas flow, which has been used successfully in a particular process chamber cannot be expected to produce satisfactory result in another process chamber without some adjustment of the process parameters. Thus, a skilled person following the teaching of document D3 would consider the values given in document D3 as a starting point for finding optimum process parameter using a given reaction chamber at his disposal. In the present case, a relatively small change of the parameters given in document D3 would result in fulfilment of the condition "$Y \geq -0.13 T + 106.3$". Therefore, contrary to the argument by the appellant under point VI(d), the claimed range of parameters is considered to be within the ranges

accessible through routine experimentation, i.e. without employing any inventive skills, and that such routine experiments have to be carried out in order to find proper etching conditions using a given etching apparatus.

2.8 For the above reasons, in the Board's judgment, the subject matter of claim 1 does not involve an inventive step within the meaning of Article 56 EPC. The patent application therefore does not meet the requirements of Article 52(1) EPC.

3. Since claim 1 does not meet the requirements of Article 52(1) EPC, it is therefore not necessary to consider the patentability of the independent claim 7.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

Beglaubigt/Certified Geschäftsstelle
Certifiée conforme Registry/Greffe
München/Munich 20. NOV. 2000

D. Spigarelli R. K. Shukla