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D E C I S I O N
of 22 May 2001

Case Number: T 0042/97 - 3.4.3

Application Number: 92103395.7

Publication Number: 0501492

IPC: H01L 21/306

Language of the proceedings: EN

Title of invention:

Method for cleaning semiconductor products

Applicant:

OKMETIC OY

Opponent:

-

Headword:

-

Relevant legal provisions:

EPC Art. 54, 56, 113

EPC R. 67

Keyword:

"Novelty (yes) - claimed feature disclosed in the prior art as not suitable for its function stated in the claim"

"Inventive step (yes)"

"Procedural violation (no)"

Decisions cited:

T 0182/90, T 0300/89

Catchword:

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Boards of Appeal

Chambres de recours

Case Number: T 0042/97 - 3.4.3

D E C I S I O N
of the Technical Board of Appeal 3.4.3
of 22 May 2001

Appellant: OKMETIC OY
Sinimäentie 12
FI-02630 Espoo (FI)

Representative: Zipse + Habersack
Wotanstrasse 64
D-80639 München (DE)

Decision under appeal: Decision of the Examining Division of the
European Patent Office posted 9 August 1996
refusing European patent application
No. 92 103 395.7 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: R. K. Shukla
Members: M. Chomentowski
M. J. Vogel

Summary of Facts and Submissions

- I. European patent application No. 92 103 395.7 (Publication No. 0 501 492) contained only one independent claim reading as follows:

"1. A method for cleaning a semiconductor product of particles accumulated on its surface as well as of metallic and organic contamination, characterized in that the washing of the semiconductor product is carried out with an acid-water solution, with a dilution ratio between $1:10^6$ - $1:10^3$, advantageously between $1:10^5$ - $1:10^4$."

According to dependent claim 6, the acid is hydrofluoric acid (HF).

- II. The application was refused by a decision of the examining division dated 9 August 1996 on the ground of lack of novelty of the subject-matter of claim 1 having regard to document

D1: Applied Physics Letters, vol. 57, no. 7, 13 August 1990, pages 676 to 677.

The examining division reasoned essentially as follows:

Document D1 discloses a method for cleaning a silicon substrate whereby the substrate is cleaned in a 0.05% hydrofluoric acid-water solution, i.e. a dilution ratio of $1:2 \times 10^3$ which falls within the claimed range $1:10^6$ - $1:10^3$.

The applicant's argument that document D1 is not

concerned with the cleaning of a semiconductor substrate but describes the epitaxial growth of Si films on a HF treated Si substrate as a function of such a pre-treatment, was not considered as convincing because claim 1 is directed to a method **suitable for** cleaning a semiconductor product of particles accumulated on its surface as well as of metallic and organic contamination and, since the known method, which is also cleaning a semiconductor product, uses the same cleaning solution, it must necessarily be suitable for removing particles accumulated on the surface of the substrate as well as metallic and organic contamination.

Moreover, the applicant's argument that the teaching of document D1 would lead the skilled person away from selecting the 0.05% HF solution was not considered as convincing because, although the document specifically indicates the use of three different solutions, namely a 49%, 5% and a 0.05% HF solution, whereby the 5% HF solution is mentioned as providing the best epitaxial layer, this does not alter the fact that a cleaning method using the 0.05% HF solution as such is known from document D1.

In the decision, there were also additional comments concluding that the subject-matter of dependent claim 6, mentioning a hydrofluoric acid solution, lacked novelty, and that the other acid solutions specified in the dependent claims 2 to 5 were well known acids, which were to be considered as alternatives for HF. The skilled person, in his normal experimental practice, would verify the effectiveness of the highly diluted acid solutions for cleaning a semiconductor product, so that the methods of the

claims 2 to 5 lacked an inventive step.

III. The applicant lodged an appeal against this decision on 21 October 1996 paying the appeal fee on the same day. On 17 December 1996, he filed the statement setting out the grounds of appeal and a new description page 2a comprising an acknowledgement of document D1. The appellant requested that the decision under appeal be set aside and a patent be granted on the basis of the application documents on file, i.e.,

Description:

Pages 1 to 6, as filed;

Page 2a (to be inserted on page 2, between the first and second paragraph), filed on 17 December 1996;

Claims:

Nos. 1 to 6, as filed.

Moreover, the appellant requested the reimbursement of the appeal fee because of an alleged procedural violation.

Oral proceedings were requested auxiliarily.

IV. With an official communication issued on 19 March 2001, the Board of Appeal informed the appellant that, in view of his arguments, it appeared that the subject-matter of claim 1 was new and also involved an inventive step, so that a patent could be granted on this basis. The appellant's arguments concerning the alleged procedural violation were however not considered as convincing.

The appellant was asked to clarify whether the oral

proceedings were requested in the event that the request for the refund of the appeal fee was to be rejected.

V. With his letter dated 17 April 2001, the appellant withdrew his request for oral proceedings.

VI. The appellant provided the following arguments in support of his requests:

Novelty

Object of document D1 is to find a method of HF-treatment of a Si substrate before low-temperature epitaxial deposition of Si films to avoid the building of surface oxide during the wet cleaning. Indeed, in this document, a 0.05% HF acid was used. However, the use of a very broad range of HF concentrations, between 0.05% and 49%, was only for scientific purpose, to prove the efficiency of HF solutions in different concentrations. Yet, the document (see in particular page 677, last paragraph) does not give the person skilled in the art a technical teaching to use a 0.05% HF solution as a cleaning liquid. It is stated in document D1:

"In conclusion, it was found that there were impurities, such as carbon, oxygen, fluorine, and chlorine" at the interface between low-temperature (900°C) deposited silicon films and 49% HF- and 0.05% HF-treated substrates. These impurities could not be detected for the 5% HF-treated sample and in this case Si films were grown epitaxially."

This conclusion of document D1 says to the skilled

person to use a 5% HF cleaning solution, and that a 0.05% solution is not a suitable cleaning solution. Therefore, a HF cleaning solution in the range 1×10^{-6} to 1×10^{-3} is not disclosed by document D1, and the subject-matter of claim 1 is thus new.

Inventive step

Starting from document D1, with successful removal of impurities for the 5% HF-solution and unsuccessful results for in particular the 0.05% HF-solution, and taking into account that it is generally known that very highly diluted acid solutions are less effective for obtaining good cleaning results, it was surprising to choose a very highly diluted acid solution for obtaining good cleaning results, as has been done in the invention. The results of measurements in the present application show that a low particle content as well as a high lifetime of the minority carriers are obtained by the method of the invention. Therefore, having regard to the state of the art, the claimed method involves an inventive step.

Reimbursement of the appeal fee

The applicant received a first communication from the examiner stating that the subject-matter of claim 1 lacked novelty. In response to this communication, the applicant filed a response wherein he presented his appreciation of the document D1. The applicant also requested an interview in case the examiner would not follow the statement of the applicant.

In response to this submission, the application was refused. Therefore, the applicant had no chance to

learn the evaluation of the applicant's arguments by the examiner.

The European examination proceeding is a proceeding based on good faith on both sides to effect a fair, fast and effective procedure. The good faith demands that one party learns about the reaction of the other party to its own arguments. This was not possible in the present case since immediately after filing the applicant's response to the first communication the application was refused. Therefore, the request for reimbursement of the appeal fee is justified.

Reasons for the Decision

1. The appeal is admissible.
2. *Novelty*
 - 2.1 Present claim 1 concerns a method for cleaning a semiconductor product of particles accumulated on its surface as well as of metallic and organic contamination, characterized in that the washing of the semiconductor product is carried out with an acid-water solution, with a dilution ratio between **1:10⁶** - **1:10³** advantageously between **1:10⁵** - **1: 10⁴**.

In accordance with the usual interpretation of the term "for", in the expression "A method for cleaning a semiconductor product", the claimed method is **suitable for** cleaning a semiconductor product of particles accumulated on its surface as well as of metallic and organic contamination, characterized in that the

washing of the semiconductor product is carried out with an acid-water solution, with a dilution ratio between $1:10^6$ - $1:10^3$ advantageously between $1:10^5$ - $1:10^4$.

According to dependent claim 6, the employed acid is hydrofluoric acid (HF).

2.2 Three methods are known from document D1 (see the whole document; see in particular page 676, right-hand column, second paragraph, second to third line); each of these methods comprises a treatment of the semiconductor product, which is carried out with an HF-water solution and which is part of a wet cleaning procedure.

Document D1 (see page 676, right-hand column, first and second paragraph) is presented as a study in order to examine the cause of polycrystal growth in more detail whereby residual elements, mentioned as interface impurities, at the interface between grown films and HF-treated substrates are measured by spectrometry. It is thus derivable from document D1 (see the sentence bridging pages 676 and 677) that the results of the measurements of the interface impurities and the growth of an epitaxial layer on the HF-treated substrate, as opposed to polycrystalline growth, are an indication for an efficient treatment.

It is to be noted in this respect that it is generally known to people skilled in the art that particles accumulated on the surface of a substrate are detrimental to the epitaxial growth of e.g. silicon on such a substrate and can result in the growth of inter alia a polycrystalline layer.

2.2.1 In a first wet cleaning procedure of document D1, a silicon substrate was dipped in a 5% HF acid for 22 seconds. Silicon films were then grown on a 5% HF treated substrate and were found to be epitaxial. Also, impurities such as carbon, oxygen, fluorine and chlorine could not be detected at the interface of the deposited silicon film and the 5% HF treated substrate (see page 676, right-hand column, Figure 1(b)).

Therefore, a 5% HF acid clean results in desorption of contaminants from the HF-treated surface and, at least with respect to the epitaxial growth following the HF treatment of substrates, as giving "good results" in the sense of document D1 (see the sentence bridging pages 676 and 677 and the last sentence of the document and of the abstract).

However, there is no indication whatsoever in document D1 concerning cleaning a semiconductor product of **metallic contamination**, i.e., it is not derivable from the document that this known method **is suitable for** cleaning a semiconductor product of particles accumulated on its surface **as well as of metallic** and organic **contamination**.

It is to be noted in this respect that in the method of the present application (see in particular page 3, second paragraph; see also page 4, second paragraph and page 6, Example 4) (i) particle contents for particles larger than 0.3 μm , (ii) lifetime (which can reveal metallic impurities such as copper) and (iii) iron content are measured and the measurement results are shown.

In any case, a 5% HF solution is outside the range of

dilution ratios of $1:10^6$ - $1:10^3$ of present claim 1.

Therefore, the cleaning method employing 5% HF acid solution does not anticipate the subject-matter of present claim 1, and this has not been contended either in the decision under appeal.

2.2.2 Document D1 (see the whole document) also discloses a treatment of the semiconductor product, which is carried out with an HF-water solution with a 49% HF concentration.

It is derivable from document D1 (see the last paragraph of the document; see also Figure 1(a) and the corresponding text) that impurities such as carbon, oxygen, fluorine and chlorine, which were measured at the interface between low-temperature (900°C) deposited silicon films and 49% HF-treated substrates were detected, and in this case the Si films which were grown were in the form of polycrystalline films.

Thus, at least with respect to the epitaxial growth following the HF treatment of substrates, an HF concentration of 49% can be derived as giving inadequate results in the sense of document D1 (see the sentence bridging pages 676 and 677).

In any case, an HF concentration of 49% (i.e. 49×10^{-2}), as in this other known method, is outside the range of dilution ratios between $1:10^6$ - $1:10^3$ of present claim 1, and this has not been disputed either.

2.2.3 Document D1 also discloses washing of a semiconductor product with an acid-water solution, with a dilution ratio of **0.05%**, which indeed falls within the range of **1:10⁶ - 1:10³** specified in the claim.

The appellant has submitted that since it is specified in the last paragraph of document D1 that it was found that there were impurities, such as carbon, oxygen, fluorine, and chlorine at the interface between low-temperature (900°C) deposited silicon and 49% and **0.05%** acid treated surface and since it was derivable from the document that this was detrimental to epitaxial growth of silicon films, this was to be interpreted by the person skilled in the art as meaning that this particular method of document D1 is **not suitable** for cleaning a semiconductor product of particles accumulated on its surface as well as of metallic and organic contamination.

The following is to be noted in this respect:

It is derivable from document D1 that the three disclosed methods differ only in the concentration of the HF solution and that the measured concentrations of residual elements (shown in Figures 1(a) to 1(c)) and the form of the deposited silicon film, i.e. epitaxial or polycrystalline, is dependent on the concentration of HF solution. It is also directly and unambiguously derivable that the treatment with a 0.05% or with a 49% HF solution does not result in the removal of the contaminants, and that removal of contaminants is obtained only with the 5% HF concentration.

Moreover, there is no derivable definite teaching from

document D1 about removal of impurities or desorption of contaminants following a **0.05%** or a 49% acid treatment.

It is also to be noted that there is no information derivable from document D1 about removal of **metallic contamination**.

2.2.3.1 Therefore, the appellant's argument that document D1 does not give the person skilled in the art a technical teaching to use a 0.05% HF solution as cleaning liquid and that, consequently, the document does not anticipate the presently claimed method, is convincing.

The further prior art documents are less relevant.

Therefore, the subject-matter of present claim 1 does not form part of the state of the art and, consequently, it is new in the sense of Article 54 EPC.

3. *Inventive step*

3.1 As set forth here above, the relevant teaching from document D1 is that **impurities** such as carbon, oxygen, fluorine and chlorine, **could not be detected** at the interface between low-temperature (900°C) deposited silicon films and **5% HF**-treated substrates, that in this case **Si films were grown epitaxially**, and that this is to be understood as being the result of desorption of contaminants from the treated surface of the semiconductor substrate. Removal of contaminants by the treatment with a **0.05%** acid solution is not

derivable from the document.

- 3.2 As convincingly argued in the statement setting out the grounds of appeal (see page 4, second paragraph), starting from document D1, with "good results" for the 5% HF-solution and "bad results" for the 0.05% HF-solution, and taking into account that it is generally known that very highly diluted acid solutions are less effective for obtaining good cleaning results, it was surprising to choose a very highly diluted acid solution for obtaining good cleaning results, as has been done in the invention.

The further prior art documents are less relevant.

- 3.3 Therefore, the subject-matter of present claim 1 involves an inventive step in the sense of Article 56 EPC, so that a European patent can be granted on this basis (Art. 52(1) and 97(2) EPC).

4. *Reimbursement of the appeal fee*

It is the established case law of the boards of appeal (see e.g. **T 182/90** OJ 1994, 641; cf. point 4 of the reasons) that it was **not** a substantial procedural violation within the meaning of Rule 67 EPC if a request to have an interview with the primary examiner was ignored; it was at the examiner's discretion to decide whether to conduct such informal discussions in accordance with the Guidelines, bearing in mind the particular circumstances of the case (see Guidelines, C-VI, 4.4 and 6, and **T 300/89**, OJ 1991, 480; cf. in particular point 9.2 of the reasons).

In support of his contention that Article 113 EPC has

been violated, the appellant has not provided any argument that in the present case the decision of the examining division was based on grounds or evidence on which he had not had an opportunity to present his comments.

The fact that the applicant had no chance to learn the evaluation of the applicant's arguments by the examiner before the decision is not relevant in the present case since, as set forth here above, there is no indication that the decision of the examining division was based on grounds or evidence on which he had not had an opportunity to present his comments.

Therefore, the appellant's request for reimbursement of the appeal fee is rejected (Rule 67 EPC).

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the examining division with the order to grant a patent on the basis of the following patent application documents:

Description:

Pages 1 to 6, as filed;
Page 2a (to be inserted on page 2, between the first and second paragraph), filed on 17 December 1996;

Claims:

Nos. 1 to 6, as filed.

3. The request for the reimbursement of the appeal fee is rejected.

The Registrar:

The Chairman:

L. Martinuzzi

R. K. Shukla