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DECISION of 26 November 1998

Case Number: T 0193/95 - 3.4.1

Application Number: 88300572.0

Publication Number: 0276959

IPC: H01L 21/20

Language of the proceedings: EN

Title of invention: Process for producing crystal article

Applicant:

Canon Kabushiki Kaisha

Opponent:

-

Headword: Selective nucleation/CANON

Relevant legal provisions:

EPC Art. 87(1), 56, 113(1)

Keyword:

"Entitlement to priority (yes) - same invention in the priority application" "Inventive step (yes)" "Procedural violation (no)"

Decisions cited: T 0081/87, T 0296/93, T 0990/91, T 0248/92

Catchword:

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Case Number: T 0193/95 - 3.4.1

D E C I S I O N of the Technical Board of Appeal 3.4.1 of 26 November 1998

Appellant:

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Decision under appeal: Decision of the Examining Division of the European Patent Office posted 5 October 1994 refusing European patent application No. 88 300 572.0 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman:	G.	Davies
Members:	R.	K. Shukla
	G.	Assi

Summary of Facts and Submissions

- I. European patent application No. 88 300 572.0 filed on 25 January 1988 and claiming priority of 26 January 1987 from the prior Japanese patent application JP 14102/87, was refused by a decision of the examining division dated 5 October 1994. The only ground for the refusal was that the subject-matter of claim 1 lacked an inventive step in view of the following documents,
 - D1: EP-A-0241 204 (published on 14 October 1987);
 - D2: EP-A-0244 081 (published on 4 November 1987); and
 - D8: Research Report SSD86-63 of Society of Electrical Communication, pages 37 to 42 and English translation.

According to the decision, the invention as claimed in claim 1 of the application in suit was not entitled to the claimed priority date of 26 January 1987 and, consequently, documents D1 and D2 published before the filing date of 25 January 1988 of the application in suit were comprised in the state of the art according to Article 54(2) EPC. The reason given for the nonentitlement to the priority was that the text at page 10, line 9 to page 12, line 16 in the application in suit disclosed two different types of nuclei, namely "initial nuclei" having a radius smaller than a critical radius and "stable nuclei" having a radius larger than said critical radius, and that the invention as claimed defined essential process parameters in terms of a single stable nucleus. The priority document, on the other hand, did not make it clear which kind of nuclei were taken for measuring silicon nucleation density in Figure 5. Accordingly, it was not unambiguously derivable from the priority document that "nucleus" was a "stable nucleus" as in the application in suit.

II. The applicants lodged an appeal on 15 December 1994 paying the appeal fee on the same day, and filed a statement of the grounds of appeal on 15 February 1995 along with two sets of claims and the corresponding amended pages of the description, forming respectively the main request and the auxiliary request. The applicants also requested that the appeal fee should be refunded on the ground that a procedural violation had been committed by the examining division which had introduced a new document, Applied Physics Letters, volume 54, No. 26, pages 2648 to 2650 (hereinafter D9)

during the oral proceedings. Oral proceedings were requested in the event that the Board intended to dismiss the appeal.

III. Claim 1 of the Main Request has the following wording:

"A method for producing a plurality of silicon single crystal islets (6) in the surface of a recessed SiO_2 substrate (1), which method comprises:

establishing a deposition surface area (2) in each one of a plurality of the recesses (4) of the substrate (1), each said deposition surface area (2) having a property of nucleating silicon at a higher nucleation density than that of the bottom surface of each recess (4), and each being limited in area to a size which is suitable for forming thereon a single nucleus of silicon <u>from which a single crystal of silicon is to be</u> <u>grown</u>;

depositing silicon (1) and forming thereby, selectively on substantially each of the deposition surface areas (2), a single nucleus of silicon <u>from</u> <u>which a single crystal of silicon is to be grown</u>;

growing single crystal silicon (5) from each single nucleus and filling thereby each respective recess(4); and

subjecting the substrate (1,5) to rotary working whilst it is pressed against a polishing pad (9) provided on a rotary disc (8), whilst feeding from a polishing liquid source (10) a polishing liquid of ethylenediamine, pyrocatechol and water which reacts with silicon to form $Si(OH)_6^{2-}$ which is removed by the polishing pad (9) so that surplus single crystal silicon (5) is removed until polishing is automatically stopped by the SiO₂ surface which acts as a stopper."

In the above, amendments to the claim in relation to the wording of claim 1 forming the basis of the decision under appeal are underlined by the Board. Also, whereas according to line 1 of the above claim 1 of the main request, "A method for producing a plurality of silicon single crystal islets..." is claimed, claim 1 forming the basis of the decision concerned "A method <u>of deposition</u> for producing a plurality of silicon single crystal islets..." (emphasis added by the Board).

Claim 2 is dependent on claim 1.

- IV. The appellant submitted essentially the following arguments in support of his requests:
 - (a) The present application is entitled to the priority from a Japanese patent application No. 14102/87 filed in Japan on 26 January 1987, since the priority document and the present application both address the problem of providing electrically insulated islets of single crystal semiconductor material which have flat surfaces, and disclose the solutions which are the same in all essential aspects.
 - (b) Both the priority document and the present application contain sufficient information for the skilled adressee to understand the required relationship between the deposition surface area

and the growth of single crystal. According to the priority document and the application, the deposition surface area (the size of the nucleation surface) has to be limited in size, which may be "several μ m or less", to grow a single nucleus. The skilled addressee following the above teaching needs to carry out only routine trial and error experiments to determine the required size of the deposition surface area. Such routine experiments are described in the article, "Control of Grain Boundary Location by Selective Nucleation over Amorphous Substrates", T. Yonehara et al, Material Research Society Symposium Proc., Volume 106, 1988, pages 21 to 26 (hereinafter D10).

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(c) It is accepted that the text at page 10, line 9 to page 12, line 16 and Figure 4 in the application in suit are not in the priority document. The new information in the application in suit is, however, included only by way of explanation, and was already a matter of common knowledge before the claimed priority date. Figure 4 was reproduced from a text book, "Crystal Growth" by Akiya Okawa, the first edition of which was published on 25 October 1977 (hereinafter D11).

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- (d) From document D3 Journal of Electrochemical Society: Solid-State Science and Technology, Volume 127 (1980), no. 1, pages 194 to 202, it follows that before the priority date of the application in suit, observation of nucleation and the measurement of nucleation density was commonplace in the art, and that the size of nucleus observable by transmission electron microscope (TEM) and scanning electron microscope (SEM) was not less than 15 nm. As the critical radii at which a nucleus is a "stable" nucleus, are disclosed to be 0.4 nm and 1.5 nm for different deposition conditions in D3, it is evident that in the document the observations and measurements were based upon "stable" nuclei and not nuclei of less than critical radius. It is therefore inferred from this that the nucleation densities shown in Figure 5 of the priority document are those of "stable" nuclei.
- (e) The comparison drawn in paragraph II.2.2 of the decision under appeal between document D9 -Applied Physics Letters, vol. 54, No. 26, pages 2648 to 2650 and Figure 5 of the priority document is not valid, since the effect of HCl in the gas composition employed in the former was to etch nuclei on the nucleation site leaving only single surviving stable nucleus, whereas in the priority document the measurements were derived using a gas composition which did not include HCl.

- (f) Document D9 was introduced by the examining division for the first time in the oral proceedings. The Representative was not in a position to contact the applicants in Japan for their technical comments during the adjournment of the oral proceedings. This evidence to support new reasoning could not have been foreseen. A refund of the appeal fee is therefore requested.
- V. In a communication dated 18 November 1997, the Board informed the applicants of its provisional views that there was no procedural violation justifying the refund of the appeal fee.

Reasons for the Decision

1. The appeal is admissible.

2. Amendments

The wording of claim 1 forming the basis of the decision under appeal was considerably revised with respect to claim 1 as filed. In the decision under appeal no objections under Article 123(2)EPC were raised against these amendments. The Board has considered these amendments and is satisfied that they are allowable under the provision of Article 123(2) EPC.

With regard to the additional amendments in claim 1 of the main request now before the Board in relation to claim 1 forming the basis of the decision under appeal (which are underlined in point III above), it is stated in claim 1 as filed that the nucleation surface has a surface area which is "sufficiently small to the extent such only a single nucleus can grow" [sic]. The claim as filed further includes a step of "permitting a single crystal to grow by applying crystal growth treatment on to said nucleus". Thus, the amendments underlined above also have a basis in the application as filed.

Dependent claim 2 specifies that the deposition surface areas are established by a film of Si_3N_4 deposited by reduced pressure chemical deposition followed by plasma etching. In the application as filed there is a basis for the subject-matter of claim 2 (see, e.g. page 6, line 22 to page 7, line 7).

The description has been amended for consistency with the claims and to acknowledge documents D1 and D2.

The application as amended according to the main request thus complies with the requirement of Article 123(2) EPC.

3. Priority

The main issue in the present appeal is whether or not the application in suit including claim 1 according to the main request is entitled to the priority date of 26 January 1987 of the prior Japanese patent application JP 14102/87, and consequently, whether documents D1 and D2, published respectively on 14 October 1987 and 4 November 1987, i.e between the claimed priority date of 26 January 1987 and the filing date of 25 January 1988 of the application in suit, are comprised in the state of the art according to Article 54(2) EPC.

3.1 Pursuant to Article 87(1) EPC, a European patent application is entitled to a priority from a previous application only if it is in respect of the same invention as was disclosed in the priority application. Also, according to the established case law of the boards of appeal, the above requirement is met if all the essential elements of the invention, i.e features of the invention, are found in the priority document either in the form of express disclosure, or are unambiguously implied by the text of the priority document (see, e.g. T 81/87 OJ EPO 1990, 250; T 296/93). Also, according to the above decisions, the priority document must disclose the invention forming the subject-matter of the later application in such a way that a skilled person can carry it out.

> In the present case, therefore, the question arises whether the priority document discloses all the essential elements of the invention as claimed in claim 1 of the main request so that the invention could be carried out by a skilled addressee.

3.2 Claim 1 of the main request relates to a method for producing a plurality of silicon single crystal islets in the surface of a recessed SiO₂ substrate. In this method a deposition surface area having a property of nucleating silicon at a higher nucleation density than that of the bottom surface of the recess is

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established, the deposition surface area being limited in size which is suitable for forming thereon a **single nucleus of silicon**. Moreover, according to the invention, subsequent to the formation of a single nucleus of silicon on the deposition surface area, a single crystal, an islet, of silicon is grown from each single nucleus to fill the recess. The portions of silicon islets protruding above the SiO2 substrate are then removed by polishing.

According to the description at page 10, line 9 to page 12, line 16 and Figure 4 of the application in suit, of a general process for the deposition of a thin film, once a nucleus exceeds a critical size r_c, its free energy G decreases from a maximum value, and the nucleus becomes a "stable nucleus" and grows further to form an island. It is further stated at page 10, line 23 to page 11, line 1 that "in the basic description of the present invention herein below, "nucleus" unless otherwise specifically noted indicates the "stable nucleus"". Thus, it is evident that the term, "single nucleus" as used in claim 1, refers to a "stable single nucleus" which subsequently grows into a single crystal.

3.3 The priority document is concerned with the same process as the application in suit, i.e a process for producing a plurality of silicon single crystal islets in the surface of a recessed SiO₂ substrate (see e.g. pages 6 to 8 and Figures 1(A) to 1(c)). Moreover, both the priority document and the application in suit contain essentially the same description of the process, the only difference being that the application

in suit additionally contains the description at page 10, line 9 to page 12, line 16 and Figure 4, which describes a general process for the deposition of a thin film of a metal or semiconductor on an amorphous substrate, with a view to explaining the process according to the present invention, i.e. selective nucleation and epitaxial growth of a single crystal of silicon on a SiO, substrate. In particular, the text and Figure 4 show that the free energy of a single nucleus is maximum at a critical radius r and that at radii exceeding r_c , the free energy reduces, so that a "stable single nucleus" is formed when the radius exceeds r Also, it follows that the further growth of a single nucleus having a radius less than the critical radius r, i.e. "initial nucleus", would not be favoured due to the increase in the free energy associated with such a growth.

Document D11 cited by the applicant is an excerpt from a standard text book on crystal growth first published in October 1977, and explains the growth of a molecular layer on a flat substrate surface from vapour phase. It is evident from the discussion of the growth of a partial round molecular layer, on pages 3 and 4 and with respect to Figure 1 to 6, which is identical to Figure 4 of the application in suit, that the extra information in the application in suit concerning "stable nucleus" and "initial nucleus" was a matter of common knowledge before the claimed priority date. In the light of this common general knowledge, it is selfevident to a skilled addressee of the priority document that in the selective nucleation and epitaxial growth process from the vapour phase, the deposition surface

.../...

area is made sufficiently small so that a single nucleus, which is thermodynamically *stable*, is formed on the deposition surface area, since otherwise, a single crystal cannot be grown from the single nucleus.

Thus, contrary to the finding in the decision under appeal, in the Board's view, it is unambiguously derivable from the priority document that the nucleus is thermodynamically stable in that it has a size exceeding a critical radius, and is a single nucleus.

3.4 Figure 5 of the priority document and Figure 6 of the application in suit (and their corresponding descriptions) are identical, and show nucleation density of silicon on silicon nitride and silicon dioxide as a function of time. According to section II, paragraph 2.2 of the decision under appeal, the priority document does not disclose even implicitly which kind of nuclei, i.e. "initial" or "stable", are taken for the measurement of silicon nucleation density in Figure 5, so that the skilled person would determine the size of the deposition surface area in relation to any type of nuclei.

In connection with the above, prior art document D3 cited by the applicants is relevant. This document concerns nucleation of silicon by chemical vapour deposition on SiO_2 and Si_3N_4 substrates and describes on page 195 experimental results, in particular, nucleus density as observed using transmission electron microscopy (TEM) or scanning electron microscopy (SEM) as a function of time. It is evident from the disclosure in the right-hand column, lines 9 to 10 that

in the case of TEM, minimum size of the nucleus which can be detected is 15 nm. Also, from the disclosure on page 197, right-hand column and formula [2], it follows that the radius of the critical cluster (r_c of the nucleus) is of the order of 1.5 nm, and does not vary appreciably with the temperature or pressure. It is thus evident that, in document D3, the nucleus density observations and measurements are in respect of "stable nuclei". In view of the above, the Board finds the submission of the applicant (see point IV (d) above) plausible that the nucleus density shown in Figure 5 of the priority document was necessarily in respect of "stable" nuclei having radii exceeding r_c , since the smaller nuclei could not have been resolved by the known techniques.

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3.5 For the foregoing reasons, all the essential elements of the invention as claimed in the application in suit, and in particular the features requiring that (i) the deposition surface area has to be sufficiently small so that a single stable nucleus of silicon can grow and (ii) the deposition surface area has a property of nucleating silicon at a higher density of stable nuclei than that of the bottom surface of the recess, are unambiguously derivable from the priority document.

> Moreover, in the Board's view, the priority document contains sufficient information for a skilled addressee to perform the invention. In particular, following the teaching of the priority document in connection with the embodiments of Figures 1A to 1C, 4A to 4B, 6A to 6D and 7A to 7B, the skilled addressee has to reduce the size of the deposition surface area of $Si_{1}N_{4}$, which is

disclosed to be "several microns or less " on page 14, lines 7 to 9, and observe whether or not a single crystal of silicon is selectively formed on the deposition surface area. Such routine experiments would lead him to an appropriate size of the deposition surface area under given conditions of deposition for the selective growth of a single stable nucleus and the subsequent growth of a single crystal in a recess.

3.6 For the foregoing reasons, in the Board's judgement, the application in suit is in respect of the same invention as that disclosed in the priority document, and is therefore entitled to the claimed priority date of 26 January 1987.

4. Inventive step

Pursuant to Article 89 EPC, therefore, the application in suit has the filing date of 26 January 1987, so that documents D1 and D2 published after the above filing date are not comprised in the state of the art according to Article 54(2) EPC and cannot be taken into account in the consideration of inventive step.

Document D8 cited in the decision as a subsidiary document concerns selective polishing of silicon protruding above a substrate surface and merely describes a process having the features as set out in the last paragraph of claim 1.

There is no other prior art document on file which describes a method of selectively growing single crystals of silicon in recesses of a SiO, substrate, as set out in the first four paragraphs of claim 1.

In view of the above, the subject-matter of claim 1 involves an inventive step within the meaning of Article 56 EPC.

5. Procedural violation

For the reasons which follow, in the Board's judgement, the contested decision was in compliance with the requirement of Article 113(1), so that there was no procedural violation justifying the refund of the appeal fee.

(i) From point 2.1 and the first and second subparagraphs on page 5 of point 2.2 of the contested decision, it is apparent that in reaching the conclusion that the claimed priority could not be allowed, the examining division only relied on the contents of the priority document and the application in suit. In the decision, document D9 was relied upon as an additional evidence to support the contention that the primary document did not unambiguously disclose that the term, "nucleus" is used for a "stable nucleus" (see, the statement, "Furthermore, studies of silicon on page 6, line 2 ff of the decision). In the Board's view, the contested decision was thus based on evidence on which the applicant had had an opportunity to present its comments (T 990/91, point 3 of the "Reasons for the Decision").

(ii) Furthermore, it is apparent from the submissions regarding the procedural violation in the statement of the grounds of the appeal that the above document was in fact discussed during the oral proceedings, so that the representative was given an opportunity to present his comments on the relevance of this document. In the event that the representative considered that he needed to consult the applicant and that there was not enough time for such a consultation, he could have requested that the proceedings be continued in writing. However, it would appear that no such request was made by the representative, so that the examining division had no reason to assume that the representative was not in a position to comment on the relevance of the cited document (see T 248/92, point 2 of the "Reasons for the Decision").

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The case is remitted to the department of the first instance with the order to grant a patent on the basis of the following documents according to the main request:
 - Claims: 1 and 2 filed on 15 February 1995 with the letter dated 15 February 1995;
 - Description: pages 1 to 22 filed on 15 February 1995 with the letter dated 15 February 1995;

Drawings: sheets 1/9 to 9/9 as originally filed.

 The request for the refund of the appeal fee is refused.

The Registrar:

The Chairman:

М.	Beer	

G. Davies