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D E C I S I O N
of 23 October 2002

Case Number: T 0643/99 - 3.4.3

Application Number: 92914380.8

Publication Number: 0548368

IPC: H01L 21/205

Language of the proceedings: EN

Title of invention:

Compound semiconductor and manufacturing method therefor

Applicant:

Mitsubishi chemical corporation

Opponent:

-

Headword:

Selective growth/MITSUBISHI

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (no)"

Decisions cited:

-

Catchword:

-



Case Number: T 0643/99 - 3.4.3

D E C I S I O N
of the Technical Board of Appeal 3.4.3
of 23 October 2002

Appellant:

MITSUBISHI CHEMICAL CORPORATION
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Representative:

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Decision under appeal:

Decision of the Examining Division of the
European Patent Office posted 2 February 1999
refusing European patent application
No. 92 914 380.8 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. 92 914 380.8 was refused in a decision of the examining division dated 2 February 1999. The grounds for the refusal was that the claims according to the main request and first to fifth auxiliary requests did not meet the requirements of Articles 82 and 123(2) EPC. The claims according to the sixth auxiliary request were considered as not involving an inventive step (Articles 52(1) and 56 EPC) having regard to the prior art documents

D5: Patent Abstracts of Japan, vol. 13, no. 453 (E-831) 11 October 1989 & JP-1-175 727; and

D6: DE-A-28 30 081.

II. The appellant (applicant) lodged an appeal on 12 April 1999, paying the appeal fee the same day. A statement of the grounds of appeal was filed on 14 June 1999 together with new claims.

III. In its communication accompanying summons to oral proceedings, the Board supplied an English translation of document D5 (in the following referred to as document D5a), and informed the appellant of its provisional opinion that the application did not appear to meet the requirement of inventive step having regard to the prior art documents D5 and D6.

In response, the appellant filed with the letter dated 23 September 2002 new claims 1 to 3 forming an auxiliary request.

IV. At the oral proceedings held on 23 October 2002, the appellant requested that the decision under appeal be set aside and a patent be granted on the basis of the following requests:

Main request

Claims 1 to 3 as filed on 14 June 1999 with the statement of the grounds of appeal with the deletion of line 8 of claim 1;

Auxiliary request 1

Claims 1 to 3 as filed with the letter dated 23 September 2002 with the deletion of line 8 of claim 1.

V. Claim 1 according to the main request corresponds to claim 1 of the sixth auxiliary request considered in the decision under appeal and reads as follows:

"1. A process for vapor phase selective growth of an aluminum-containing III-V compound semiconductor thin film on a substrate, using a halogen element-free hydride and a halogen element-free organometallic compound as raw materials to be grown, a protective film on an area of said substrate where said growth shall not occur, and a halide gas and/or halogen gas free from mother elements of the compounds to be grown, wherein said halide gas and/or halogen gas are added to a reaction atmosphere at a flow rate that enables a selective mode to be maintained during growth of the semiconductor thin film,

characterized in that

said organometallic compound is trialkylaluminum and said halide gas and/or halogen gas is hydrogen chloride gas, and the molar ratio R of hydrogen chloride to trialkylaluminum, and the molar ratio X_{Al} of aluminum in the elements of group III of the aluminum-containing III-V compound satisfy the following relations:

$$0 < R \leq 50, \text{ when } 0.2 < X_{Al} < 0.4$$

$$0 < R \leq 10, \text{ when } 0.4 < X_{Al} \leq 1."$$

Claims 2 and 3 are dependent claims.

VI. Claim 1 according to the auxiliary request differs from claim 1 according to the main request in that it additionally contains the following feature at the end:

"and said III-V compound semiconductor film is doped in such a way as to provide carrier concentration of $1 \times 10^{16} \text{ cm}^{-3}$ or more."

VII. The reasons given in the decision under appeal with regard to inventive step can be summarized as follows:

- (a) Document D6, which discloses metal-organic vapor phase epitaxial growth (MOVPE) of $\text{Ga}_{1-x}\text{Al}_x\text{As}$, is considered the closest prior art. The claimed method differs from that of document D6 only in that the process is a selective growth, whereas in document D6, a blanket deposition is carried out.
- (b) Selective growth using MOVPE is well-known in the art and document D5 discloses that HCl has to be added in order to obtain selective growth of InP. Since document D5 does not disclose any process parameters for $\text{Ga}_{1-x}\text{Al}_x\text{As}$, the skilled person would

as a first attempt use the parameters disclosed in document D6. Since the parameters disclosed in document D6 fall within the claimed range, they would result in selective growth of $Ga_{1-x}Al_xAs$.

VIII. In support of inventive step, the appellant presented essentially the following arguments:

- (a) Document D5 and not D6 should be considered the closest prior art, since it relates to *selective* growth of a III-V semiconductor compound using vapor phase epitaxy using a silicon oxide film as a protective film. Document D6, on the other hand, is not related to selective growth.
- (b) The examples disclosed in document D5 relate to selective growth of InP. As demonstrated in a comparative experiment carried out by the appellant using the process parameters corresponding to a value of the molar ratio R equal to 0.05 employed in document D5, it is evident that the molar ratio in document D5 is too low for preventing $Ga_{1-x}Al_xAs$ polycrystals from forming on the protective film.
- (c) Document D6 is not concerned with selective growth of $Ga_{1-x}Al_xAs$, but rather with increasing the growth speed of $Ga_{1-x}Al_xAs$ layers which are blanket deposited. In order to increase the growth speed, it is taught in document D6 to add HCl. Therefore, a skilled person seeking to adapt the method of document D5 for selective growth of $Ga_{1-x}Al_xAs$ would not consider the teaching of document D6 relevant, as it is not concerned with selective growth.
- (d) As to the auxiliary request, document D5 is concerned with selective growth of Fe-doped InP to produce a highly resistive material. Therefore,

the skilled person seeking to produce highly conductive semiconductor layers would not consider document D5 at all.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is therefore admissible.
2. *Inventive step - Main request*

The only issue in the present appeal is inventive step having regard to the prior art documents D5 and D6.

- 2.1 The application in suit relates to metal-organic vapor phase (MOVPE) selective growth of aluminum-containing III-V semiconductor thin films, such as $\text{Ga}_{1-x}\text{Al}_x\text{As}$, where the molar ratio x of aluminum in the group III elements is greater than 0.2. Before the growth takes place, a protective film is formed on a substrate exposing selected areas of the substrate. During selective growth, the semiconductor thin film is formed only on the exposed areas of the substrate and not on the protective film.

According to the application in suit, in the MOVPE technique it is particularly difficult to achieve selective growth of aluminum-containing III-V semiconductor thin films with higher aluminum content. The undesired consequence is the formation of semiconductor polycrystals on the protective film (cf. application as published, page 2, lines 10 to 31).

The application solves the above problem by adding a small amount of HCl to the reaction mixture in the claimed proportions, which will inhibit the undesired

growth of semiconductor polycrystals on the mask layer. Very small amounts of HCl do not induce selective growth, whereas too much HCl inhibits the activation of dopants, and eventually stops the growth altogether (cf. Figures 7 and 8; page 3, lines 6 to 48).

- 2.2 Document D5 discloses selective organometallic vapor phase epitaxial growth (MOVPE) of InP on an InP substrate using a protective film 206 made of silicon oxide as mask (cf. Figure 2; D5a, page 5, last paragraph to page 6, last paragraph). The reaction gases are $\text{In}(\text{CH}_3)_3$ and PH_3 .

A halide gas such as HCl is added at the upstream side of the substrate in the reaction chamber in order to prevent polycrystalline InP from growing over the protective film, i.e. to ensure selective growth (cf. D5a, page 4, "Problems intended to be resolved..." and "Means for resolving the problems"). In addition to inhibiting growth of InP on the protective layer, the presence of the halide gas also has the effect of making the surface of the InP layer flat, thereby making it suitable for producing planar device structures.

The detailed example shows selectively grown Fe-doped InP acting as current-blocking layers in an InGaAsP/InP laser structure.

Although document D5 only describes selective growth of InP in detail, it is mentioned that selective growth of other III-V semiconductor compounds such as AlGaAs/GaAs would be possible as well (cf. D5a, page 7, third paragraph).

- 2.3 Document D6, which was considered closest prior art in the decision under appeal, discloses deposition of $\text{Ga}_{1-x}\text{Al}_x\text{As}$ using MOVPE where trimethyl gallium (TMG),

trimethyl aluminum (TMA) and arsene (arsenic hydride) are used as raw material for the deposition (cf. page 10, line 4 to page 15, line 19). The deposition is blanket deposition on the entire substrate, i.e. the growth is not selective.

$Ga_{1-x}Al_xAs$ layers grown by MOVPE are generally not of very high quality mainly due to the presence of carbon impurities (cf. D6, page 6, line 9 to page 7, line 15). A halogen such as HCl is therefore added in the method of document D6 for increasing the quality of the layers (cf. page 8, lines 13 to 18). It is also noted that the addition of HCl increases the deposition speed in a cold-wall growth system (cf. page 8, lines 19 to 26).

Several Examples with different deposition parameters for growing $Ga_{1-x}Al_xAs$ layers with mirror-like surfaces are disclosed in document D6: In Example 4, $Ga_{0.21}Al_{0.79}As$ was grown using 1.22 cm³ HCl and 2.0 cm³ TMA, which corresponds to a molar ratio $R = 0.61$ (cf. page 11, lines 8 to 14). In Examples 6, and 10, $Ga_{0.70}Al_{0.30}As$ layers were grown using a molar ratio R equal to 3.9. In Example 10, the layer was doped with silicon at a concentration of 2×10^{18} cm⁻³ (page 13, lines 10 to 26 and page 15, lines 4 to 19). The other Examples disclose growth of layer having an aluminum content $x < 0.2$.

2.4 In the decision under appeal, the examining division considered document D6 to represent the closest prior art. The appellant contended that document D5 and not document D6 should be considered the closest prior art, since it relates to *selective growth* of a III-V semiconductor using MOVPE. It follows from the discussion of the prior art documents D5 and D6 above

that document D5 relates to selective growth of InP, a III-V semiconductor material as in the application in suit. The Board therefore considers document D5 to be the closest prior art.

2.5 The method of claim 1 according to the main request differs from that of document D5 in that (i) selective growth of an aluminum-containing III-V compound semiconductor film is obtained, whereas in document D5, the only embodiment shows selective growth of InP, which is a III-V compound semiconductor free from aluminum; (ii) trialkylaluminum is used as source gas for aluminum; and (iii) the ratio R of hydrogen chloride to trialkylaluminum and the molar ratio X_{Al} of aluminum in the elements of group III of the semiconductor satisfy $0 < R \leq 50$ when $0.2 < X_{Al} < 0.4$ and $0 < R \leq 10$ when $0.4 < X_{Al} \leq 1$.

2.6 In view of the above differences, the objective technical problem addressed relates to providing an MOVPE method for selective growth of an aluminum-containing III-V compound semiconductor, such as $Ga_{1-x}Al_xAs$, and having an aluminum content x within the range $0.2 < x \leq 1$.

2.7 It is suggested in document D5 that the method described therein can be used for selective growth in the GaAs/GaAlAs system (cf. D5a, page 7, third paragraph). Thus, given the above suggestion, the skilled person faced with the problem of providing a MOVPE method for selective growth of an aluminum-containing III-V compound semiconductor would have to modify the method of document D5 appropriately for growing $Ga_{1-x}Al_xAs$. To this end, he would consider document D6 disclosing a MOVPE method for the growth of $Ga_{1-x}Al_xAs$ compound semiconductor to be highly relevant.

In the MOVPE process disclosed in document D6 for growing $Ga_{1-x}Al_xAs$ layers, the compounds TMG, TMA, and arsene are used as raw materials (cf. e.g. Example 4). Moreover, as stated in paragraph 2.3 above, HCl gas is added to improve the quality of the $Ga_{1-x}Al_xAs$ layer and increase the growth rate.

Examples 4, 6, and 10 of document D6 disclose deposition parameters for growth of $Ga_{1-x}Al_xAs$ layers with the aluminum content x within the range $0.2 < x \leq 1$. For $x = 0.79$, the value of R is 0.61 (cf. Example 4), and for $x = 0.30$, R is 3.9 (cf. Examples 6 and 10), i.e. both values of R are within the claimed range covered by the claimed method.

2.8 In this context, the appellant argued that only document D5 discloses selective growth, and therefore, the skilled person would only consider the concentration of HCl disclosed in document D5 and not the concentrations disclosed in document D6 (cf. item VIII(b) above). Since document D6 is not concerned with selective deposition but is mainly concerned with increasing the deposition speed of a non-selective deposition method, the appellant argued that the skilled person would not consider this document as a source of information regarding the concentration of HCl required for obtaining selective growth (cf. item VIII(c) above).

2.8.1 The above argument is not convincing for the following reasons:

Firstly, document D5 discloses process parameters which are only relevant for selective growth of InP, and it would be regarded fortuitous if the growth parameters disclosed in document D5 would provide satisfactory results for $Ga_{1-x}Al_xAs$ as well.

Secondly, and more importantly, document D6 is primarily concerned with improving the quality of $\text{Ga}_{1-x}\text{Al}_x\text{As}$ layers grown using MOVPE. This task is accomplished by adding HCl to the raw materials TMG, TMA, and arsene. Although the appellant correctly observes that document D6 reports an increase of the deposition speed due to the addition of HCl, this is presented as an additional benefit in addition to the main task of improving the quality of the $\text{Ga}_{1-x}\text{Al}_x\text{As}$ layers (cf. D6, page 8, lines 19 to 16; page 15, lines 20 to 33). Due to the stringent requirements in the semiconductor industry on factors such as the surface morphology and the level of defects and impurities of semiconductor materials, the skilled person would consider high quality of the grown layers to be of crucial importance, since otherwise, the method cannot be used for producing high-performance GaAs/ $\text{Ga}_{1-x}\text{Al}_x\text{As}$ semiconductor devices. Moreover, although document D6 is not concerned with *selective growth*, the selective growth requires the growth of a high-quality layer in exposed areas at a reasonable rate, so that the disclosure in document D6 relating to molar concentration of HCl would be regarded as relevant by the skilled person in selective growth of $\text{Ga}_{1-x}\text{Al}_x\text{As}$.

For the foregoing reasons, in the Board's judgement, the subject matter of claim 1 according to the main request does not involve an inventive step within the meaning of Article 56 EPC.

3. *Inventive step - auxiliary request*

- 3.1 With respect to the method according to the main request, the method of claim 1 according to the auxiliary request further specifies that the aluminum-containing III-V compound semiconductor is doped to have a carrier concentration of $1 \times 10^{16} \text{ cm}^{-3}$ or more. Such values of the carrier concentrations are

considered conventional in the art, as exemplified in document D6, where dopant concentrations of $2 \times 10^{18} \text{ cm}^{-3}$ (n-type) and $3 \times 10^{18} \text{ cm}^{-3}$ (p-type) are disclosed (cf. Example 10, page 15, lines 15 to 18). The layers disclosed in Example 10 in document D6 were furthermore produced having a value of R equal to 3.9 falling within the claimed range. Therefore, whenever a carrier concentration of $1 \times 10^{16} \text{ cm}^{-3}$ or more is required, the skilled person would be able to introduce a corresponding amount of dopants without employing inventive skills.

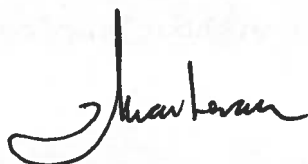
- 3.2 Although the appellant correctly pointed out that document D5 discloses the selective deposition of a Fe-doped InP layer, i.e. an InP layer doped to be semi-insulating or highly resistive (cf. item VIII(d) above), it is mentioned in document D5 that the teaching is independent from which dopant is used in the selectively grown semiconductor layer (cf. D5a, page 7, first paragraph). The skilled person would thus recognize that the teaching of document D5 is equally well applicable for the growth of n- or p-type semiconductor layers having high carrier concentrations.
- 3.3 Therefore, the subject matter of claim 1 according to the auxiliary request does not involve an inventive step within the meaning of Article 56 EPC for the same reasons as for the main request.

Order

For these reasons it is decided that:

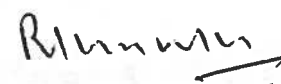
The appeal is dismissed.

The Registrar:



P. Martorana

The Chairman:


R. K. Shukla