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D E C I S I O N
of 22 April 2004

Case Number: T 0288/02 - 3.4.3

Application Number: 97106589.1

Publication Number: 0793281

IPC: H01L 33/00

Language of the proceedings: EN

Title of invention:
Electro-magnetic transducers

Applicant:
MINNESOTA MINING AND MANUFACTURING COMPANY, et al

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 84, 123(2), 54, 56

Keyword:
"Definition of individual product features in a product claim by process features"
"Novelty (yes)"
"Inventive step (yes)"

Decisions cited:
T 0150/82

Catchword:
-

Case Number: T 0288/02 - 3.4.3

D E C I S I O N
of the Technical Board of Appeal 3.4.3
of 22 April 2004

Appellant: MINNESOTA MINING AND MANUFACTURING COMPANY
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 7 September 2001 refusing European application No. 97106589.1 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: R. K. Shukla
Members: E. Wolff
J. P. B. Seitz

Summary of Facts and Submissions

I. This is an appeal from the decision of the examining division, posted 7 September 2001, to refuse European patent application Nr. 97 106 589.1, a divisional application of European patent application 91 307 650.1 in accordance with Article 76 EPC, for lack of novelty in the light of document

D3 Patent abstracts of Japan, vol. 13, no. 137 (C582), 5 April 1989 & JP 63-A-303889 A, 12 December 1988, and

D3a which is an English translation of JP 63-A-303889.

The examining division concluded that document D3 discloses a process for forming a pn junction which is exactly the same as the process disclosed in the application in suit. Moreover, since the starting products and the growth process were strictly identical, the layers grown had to be identical; if there were differences, then the application did not disclose the invention sufficiently for it to be performed by a skilled person.

The decision further stated that, should the objection of lack of novelty show itself to be unfounded, then the following two documents would, each on its own, deprive the invention as claimed of an inventive step:

D1 Migita et al: "P-type conduction of ZnSe highly doped with nitrogen by metalorganic molecular beam epitaxy", Journal of Crystal growth, vol. 101, no. 1/4, 1 April 1990, pages 835-840;

D2 Take et al: "P-type conductivity control of ZnSe highly doped with nitrogen by metalorganic molecular beam epitaxy", Applied Physics letters, vol. 56, no. 20, 14 May 1990, pages 1989-1991.

During examination, the appellant had submitted, inter alia, the following documents to support the arguments in favour of novelty of the claimed invention and the presence of an inventive step:

D6 Migita et al., Journal of Crystal Growth, 138 (1994) pp. 391-396
(submitted as Exhibit A)

D7 Ho et al., Applied Physics Letters **66**(9), 27 February 1995, pp. 1062-1064
(submitted as Exhibit B)

D8 Kamata et al., Applied Physics Letters **63**(24), 13 December 1993, pp. 3353-3354
(submitted as Exhibit C)

D9 Wolk et al., Applied Physics Letters **63**(20), 15 November 1993, pp. 2756-2758
(submitted as Exhibit D)

D10 "Doping of nitrogen acceptors into ZnSe using a radical beam during MBE growth"; K. Ohkawa et al., Journal of Crystal Growth **111** (1191) pp. 797-801
(submitted as Exhibit G)

II. The appellant filed a notice of appeal and paid the appeal fee on 8 October 2001. The statement setting out the grounds of appeal was filed on 14 January 2002, together with a new main request and auxiliary requests 1 and 2. The statement of grounds was accompanied by the following document

D11 Copy of the certificate of the award of the Rank prize for Opto-Electronics on 30th November 1993 (submitted as Exhibit H)

III. On 22 March 2004, in response to a communication accompanying the summons to oral proceedings, the appellant filed a new main request and three new auxiliary requests and submitted the following document

D12 S.M. Sze, "Physics of Semiconductor Devices", Wiley, 1981, pp. 686-688

On 24 March 2004 the appellant submitted further arguments in support of the application and submitted the following documents

D12a S.M. Sze, "Physics of Semiconductor Devices", Wiley, 1981, front page, bibliographic data to document D12

D13 Excerpts of J. Gutowski et al., "Optical Properties of ZnSe epilayers and films", Review Article, phys. stat. sol. (a), **120**, 11, 1990, pp. 11-12, 38-43, 52-59

IV. Oral proceedings took place on 22 April 2004. During the oral proceedings, the appellant filed a new main request. The new main request comprises independent claim 1 and dependent claims 2 and 3, and a description adapted to match these amended claims.

V. Claim 1 of the request reads as follows:

"1. An electromagnetic transducer comprising

a conductive nitrogen doped p-type IIB-VIA semiconductor film obtainable by molecular beam epitaxy using a free radical source to generate atomic nitrogen radicals from gaseous nitrogen, the film having a net acceptor concentration greater than about $5x10^{15}$ cm^{-3} and exhibiting an electrical resistivity less than 15 ohm-centimeters,

the transducer having a room temperature (300K) electroluminescent spectrum with a maximum intensity at wavelengths of less than 550 nanometers, and being useful in light emitting diodes and lasers."

VI. The arguments put forward by the appellant an be summarized as follows:

(a) At the priority date of the application in suit, despite long and extensive efforts to find suitable materials, dopants, growth techniques and doping techniques, no substantial progress had been made by anyone towards achieving semiconductor lasers and LEDs capable emitting light in the blue-green region of the spectrum. Although high levels of incorporation of dopant species in IIB-VIA semiconductors had been obtained, the effective dopant concentrations achievable with p-type dopants such as lithium and nitrogen were well below the levels required to obtain working devices.

(b) It was the appellant's achievement to find that p-type doping of IIB-VIA semiconductor crystals at doping levels sufficiently high to enable device fabrication could be obtained by using molecular beam epitaxy in combination with providing nitrogen in the form of free atomic radicals from gaseous nitrogen.

(c) Document D3/D3c describes a method of forming II-VI semiconductor devices by molecular beam epitaxy, in which radicals of nitrogen molecules are used as the source of the p-type dopant with the option of using radicals of nitrogen atoms or molecules such as ammonia (NH_3) also being mentioned. However despite the reference to atomic radicals, at the time of the priority date of the invention, a skilled person would not have been set by document D3/D3c on the path to the claimed invention. Firstly, Document D3/D3c was just one of a great number of documents describing the many efforts that were being made at the time to obtain IIB-VIA semiconductor devices capable of emitting light in the blue or blue-green region of the spectrum. Secondly, four years after the date of publication of document D3/D3c, the same team of authors published a document, which shows that their approach had proved unsuccessful. Thirdly, at the time the appellant made the invention in suit, the invention made an immediate impact as demonstrated, inter alia, by the award to the inventors of the Rank Prize for Optoelectronics.

(d) As regards the prior art documents D1 and D2, these relate to the growth of IIB-VIA compound semiconductors by means of metal-organic growth processes. Metal-organic processes leave behind in the finished products traces of carbon from the organic compounds employed in the growth process, which can be used to distinguish between devices manufactured by those processes and those of the claimed invention which are produced by molecular beam epitaxy. The "*very weak whitish-blue emission*" to which document D1 refers is not at all useful for practical devices which need more than a very weak emission to operate, and need to operate at room temperature where the luminescent efficiency is only about 20% of that at 77K, the temperature at which the luminescence was observed.

(e) A combination of the teachings of document D1 or document D2 with the technique described in document D3 would also not have led towards the claimed invention in view of the use in documents D1 and D2 of a process which combines metal-organic molecular beam epitaxy (MOMBE) with the use of ammonia (NH3) as source for the nitrogen dopant. The invention in suit uses neither of these techniques.

Reasons for the Decision

1. The appeal is admissible.

2. *Amendments (Article 123(2) EPC*

Claim 1 of the main request differs from the originally filed claim 1 in several respects.

(a) The original claim 1 claimed an electromagnetic transducer comprising a p-type layer and an n-type layer and the electrical connections to these layers, while claim 1 of the request refers only to the p-type layer and its properties. However, the Board accepts that it is clear both from the description in general and from selected passages of the description in particular (column 2, lines 39 to 43 and column 12, lines 17 to 27 of the application as published), that the invention lies in the provision of a doped p-type IIB-VIA semiconductor layer, and that therefore the reference to the n-type layer and the electrical connections may be omitted without contravening Article 123(2) EPC.

(b) The application as published discloses without reference to the doping efficiency specified in the originally filed claim 1, that the p-type layer is doped such that the net acceptor concentration is greater than about $5x10^{15}cm^{-3}$ and exhibiting an electrical resistivity of less than 15 ohm centimetres (column 2, lines 39 to 43), thereby providing the basis for the amended wording of claim 1.

(c) It is also disclosed in the description that a free radical source is used to generate atomic nitrogen radicals from gaseous nitrogen (e.g., published application, column 8, lines 24 to 29), that the p-doped film of the transducer has a net acceptor concentration which is greater than $5x10^{15}cm^{-3}$ and providing a room temperature (300K) electroluminescent spectrum with an intensity maximum at a wavelength below 550nm (column 3, lines 35 to 42, column 4, lines 9 to 17 and lines 43 to 51), and that it is useful in light emitting diodes and laser diodes (column 7, lines 30 to 34). Accordingly, the subject matter of the corresponding features of claim 1 was already disclosed.

(d) The Board is therefore satisfied that the amendments which distinguish claim 1 of the request from claim 1 as originally filed do not introduce subject matter which goes beyond the contents of the originally filed application.

3. *Clarity (Article 84, second sentence)*

3.1 Claim 1 of the main request is a product claim which, by stating that the film "*is obtainable by molecular beam epitaxy using a free radical source to generate atomic nitrogen radicals from gaseous nitrogen*", attempts to define some of the features of the product in process terms.

3.2 According to the established case law of the Boards of Appeal, product-by-process claims are permissible if the product cannot be adequately characterised by its composition or other verifiable parameters (see, for example, T 150/82). By implication, it is therefore equally permissible to define individual product features in a product claim in terms of process features. For a product-by-process claim and equally for the definition of a product feature in terms of process features, the process itself must, of course, be adequately defined.

3.3 The features of the product which are defined by the process features in claim 1 are the electrical and chemical characteristics of the material which are obtained by growing the material by molecular beam epitaxy and by doping the material with atomic nitrogen radicals.

3.4 Metal-organic crystal growth processes, whether metal-organic vapour phase deposition (MOVPE) or metal-organic molecular beam epitaxy (MOMBE), utilise as starting material organic compounds such as, e.g. DMZ (dimethylzinc - see document D1, p. 835, right-hand column) which include carbon and other elements forming part of the organic compound. As explained by the appellant, metal-organic crystal growth processes always contain unwanted carbon-based residues left behind by the organic starting materials, in contrast to material grown by MBE (molecular beam epitaxy) which, by virtue of the pure starting materials employed, does not contain any carbon-based impurities. Therefore, materials grown by MBE could always be distinguished from those grown by MOMBE or other metal-organic crystal growth techniques. The Board accepts the appellant's argument the presence or absence of carbon based residues enables semiconductor material grown by MBE to be distinguished from semiconductor material grown by growth techniques which use metal-organic compounds as their starting materials.

3.5 Claim 1 further requires that doping is performed by means of "*a free radical source to generate atomic nitrogen radicals from gaseous nitrogen*". Thus, doping is performed by a technique which does not rely on organic starting materials.

3.5.1 Also, the process feature that doping of the material is performed "*using a free radical source to generate atomic nitrogen radicals from gaseous nitrogen*" serves to claim the high uncompensated doping levels of p-type nitrogen doping of the transducer, which is required to obtain the claimed characteristics of the transducer that the room temperature (300K) electroluminescent spectrum with a maximum intensity at wavelengths of less than 550 nanometres is "*useful in light emitting diodes and lasers.*"

3.6 On the basis of the foregoing arguments, the Board accepts that material which "*is obtainable by molecular beam epitaxy*" and in which doping of the material is performed "*using a free radical source to generate atomic nitrogen radicals from gaseous nitrogen*" displays the verifiable product features that the grown material is free of carbon-based impurities and has the required effective p-type doping concentrations levels to make the transducer useful in light emitting diodes and lasers.

3.7 The Board further accepts that in the present case there is no other concise way of defining the material. As the claimed process features can be associated with identifiable product features, the Board is satisfied that independent claim 1 is clear, as required by Article 84, second sentence EPC.

4. *Novelty (Article 54 EPC)*

4.1 The complete text of the translation D3a used by the examining division was not on file. The Board therefore introduced Document D3c, which is a full translation of the corresponding published Japanese Patent application JP63-A-303889 and was furnished by the appellant in connection with the above-mentioned earlier European patent application.

4.2 Document D3, Japanese patent application 91 307 650.1, discloses, according to its Japanese patents abstract and its translation D3c, a process of growing II-VI semiconductor compounds using molecular beam epitaxy, in which a beam of nitrogen radicals is projected onto the substrate during crystal growth. The radical beam contains an impurity element such as N, with an "*excited species being generated in the gas such as* N_2".

4.2.1 The examining division concluded from the corresponding text of the translation D3a that the claimed invention lacked novelty because the process disclosed in document D3 to grow and dope the material was identical to the one described in the application, so that the semiconductor layers grown by the claimed process and by the process described in document D3 would necessarily have the same characteristic parameters.

4.2.2 However, although alternative sources of nitrogen such as nitrogen atoms and nitrogen-containing molecules such as ammonia (NH_3) are mentioned in passing - "*and nitrogen molecule radicals (N_2°) or nitrogen atom radicals (N°) are irradiated.*" (page 4, lines 8 and 9) and that they "*can be radicals of nitrogen atom or molecules containing nitrogen atom, for example ammonia (NH_3)*" (page 5, lines 8 and 9) - in the process described in detail in document D3 as having been carried out, the only source of nitrogen were, in fact, nitrogen molecule radicals (see, e.g., page 4, lines 23 to 25 referring to the "*nitrogen molecule radical beam 25a*"). There is thus clearly no teaching in document D3 which would inevitably lead to a transducer having the features which are claimed in claim 1 of the main request.

4.2.3 It is to be observed that there is also neither a reference to a device in document D3 (except for a general remark that the method enables p-type II-VI semiconductor films to be made with have a large range of applications (D3c, page 5, bottom of the page)), nor is there any mention of characteristic features of a device such as carrier concentration, resistivity and electroluminescence maxima.

4.2.4 For the foregoing reasons, the Board concludes that the invention claimed in claim 1 of the main request is new with regard to document D3/D3c.

4.3 Documents D1 and D2 relate to materials for manufacturing blue LEDs and lasers (D1, page 835, first few lines of the left-hand column) and blue light emitting devices (D2, page 1989, first few lines of the left-hand column), respectively.

4.3.1 The devices disclosed in document D1 and D2 each consist of

(a) a pn junction device (see Figure 5 in each case)

(b) with a first contact

(c) a nitrogen-doped p-type II-VI film

(i) with a net acceptor concentration greater than $5x10^{15}cm^{-3}$ and a

(ii) resistivity of less than 15Ωcm

(d) an n-type layer coupled to establish a pn-junction, and

(e) a second contact electrically connected to the n-type layer (via the GaAs substrate).

4.3.2 Each of documents D1 and D2 reports the growth of nitrogen p-doped materials with low resistivity. The resistivity achieved in document D1 is 1Ωcm at a carrier concentration of $5x10^{17}$ cm^{-3} (page 835, left-hand column, second paragraph), or 10^{19} cm^{-3} (page 836, section 3.1, and page 839, section 4, first paragraph), while in document D2 the resistivity is 0.57 Ωcm at a carrier concentration of $5.6x10^{17}cm^{-3}$. The dopant source in both cases is ammonia (NH_3), the growth technique Metal-Organic Molecular Beam Epitaxy (MOMBE), and the low resistivity is attributed to the relatively high temperature of the substrate during growth (D1 and D2, Figure 4 and accompanying text). Document D2 acknowledges the effect of carrier compensation by contrasting the hole concentration ($1.2 \times 10^{16} cm^{-3}$ with the nitrogen concentration of $10^{19} cm^{-3}$ (page 1990, right hand column, end of first paragraph).

4.3.3 Both document D1 and document D2 relate to semiconductor materials in which the p-doped layer is obtained by MOMBE (document D1, page 835, right-hand column under the heading "2. Experimental", and document D2, p. 1989, left-hand columns, second paragraph), that is, by a process employing metal-organic starting materials. As discussed in paragraph 3.6 above, the process feature that the "*conductive nitrogen doped p-type IIB-VIA semiconductor film obtainable by molecular beam epitaxy using a free radical source to generate atomic nitrogen radicals from gaseous nitrogen*" serves to distinguish the layers grown in this manner on account of the absence of carbon-based residues, from layers produced from metal-organic starting materials. The Board accepts for these reasons that the claimed transducer is distinguishable over transducers fabricated using the metal-organic growth techniques disclosed in each of documents D1 and D2.

4.3.4 For the foregoing reasons, the Board concludes that the invention claimed in claim 1 of the main request is new with regard to each of the documents D1 and D2.

5. *Inventive step (Article 56 EPC)*

5.1 Document D3, according to its translation D3c (see point 4.1 above), discloses an apparatus and a method for producing thin films of semiconductor materials such as ZnSe by means of MBE. A radical beam source (22) produces a radical beam containing an impurity element such as nitrogen which is projected onto the substrate (17) simultaneously with the irradiation of the molecular beams (13a and 14a). Although document D3 does not provide any characterisation of devices incorporating such materials, the processes employed in document D3 are of the same kind as the processes in terms of which product features are defined in claim 1. Document D3 accordingly represents the nearest prior art.

5.1.1 Given this closest prior art, the object of the invention is to provide an electromagnetic transducer comprising a p-type IIB-VIA semiconductor film which is useful in light emitting diodes and laser diodes operating at wavelengths of less than 550nm.

5.1.2 Document D3/D3c undoubtedly refers to atomic nitrogen radicals as a possibility ("*... nitrogen gas is used as the impurity raw material, and nitrogen molecule radicals (N_2°)or nitrogen atom radicals (N°) are irradiated ...*" (page 4, lines 7 to 9), and "*… for this invention, a nitrogen molecule radical was used, but in addition to this it can be radicals of nitrogen atoms and molecules containing a nitrogen atom, for example ammonia (NH_3).*" (page 5, lines 7 to 9). However, the Board accepts the appellant's argument that atomic nitrogen radicals together with nitrogen containing molecular radicals such as NH_3 are mentioned merely in passing, while in the process described as having actually been carried out in practice the dopant was provided only in the form of molecular nitrogen radicals (page 4, lines 12 to 29).

5.1.3 The Board also considers plausible the appellant's argument that against the background of a flood of different publications the skilled person would, other than in hindsight, have had no reason to consider document D3/D3c as being relevant any more than many of the other publications that appeared at the time. The appellant drew to the Board's attention the contemporary document D13, a review article which lists more than 200 publications that relate to obtaining light emission in the blue-green region of the spectrum in II-VI semiconductors. Summarized in tabular form are some 40 acceptor and acceptor-exciton complexes in ZnSe films involving a variety of acceptors such as Li, N, Cu, etc., with semiconductor materials grown by a variety of different growth techniques (MBE, LPE, MOCVD) and on different substrates (page 41, table 5). Despite listing several cases in which MBE is used as growth process in combination with nitrogen as dopant, there is nothing in document D13 which would have indicated to the skilled person that a combination of MBE growth technique and the use of atomic nitrogen radicals as p-dopant source would ultimately result in a practically useful device.

5.2 There can, in the Board's view, be no doubt that the authors of document D3/D3c failed to appreciate that selecting a dopant source other than nitrogen molecular radicals would be the key to achieving the desired high-level nitrogen p-type doping of IIB-VIA semiconductors which would render them useful for fabricating devices such as lasers and light emitting diodes that operate at wavelengths less than 550nm, just as much as those authors failed to appreciate that the use of ammonia (NH_3) radicals as source of the nitrogen dopant would not lead to the required high doping efficiencies because of the compensation of p-type doping by the hydrogen present in the process (see, for example documents D7, D8 and D9, all published after the priority date of the above mentioned earlier European patent application; in document D7, excess hydrogen causing compensation is present as a by-product of the MOVPE process - document D7, page 1062, left-hand column, lines 17 to 25, and page 1063, right-hand column, third-last line to page 1064, left-hand column, line 2; in document D8, hydrogen passivation is considered "*the most likely origin of nitrogen acceptor compensation*" - page 3353, lefthand column, lines 13 to 16; and according to document D9, the "*formation of a N-H complex is believed to be responsible for the passivation of N acceptors in ZnSe*" - page 2756, left-hand column, first paragraph, last three lines).

5.2.1 Additionally, as evidenced by document D10 which was published by the authors of document D3 some 4 years after the date of the latter, there is no indication anywhere that the authors at any time considered pursuing the options of using dopant sources other than molecular nitrogen radicals ("*The source materials are elemental Zn, Se and* N_2 *or* NH_3 *gas for the nitrogen radical beam",* page 797, right-hand column, under the heading "*Experimental procedure*"); on the contrary, in document D10 the authors admit the failure of their technique by reporting that "*Activation of N doped in ZnSe was as low as 2%. More work will be needed to enhance the activation of N*" (Document D10, page 800, under the heading "5. Conclusions").

5.2.2 For these reasons the Board concludes that the skilled person would, at the time, have failed to realise that the passing reference in document D3 to atomic nitrogen radicals could, in combination with MBE as crystal growth technique, have lead to the claimed practically useful IIB-VIA semiconductor transducer emitting in the blue-green region of the spectrum.

For the foregoing reasons the Board concludes that the invention as claimed in claim 1 of the main request is not obvious in the light of document D3 alone.

5.3 Documents D1 and D2 disclose semiconductor layers grown by MOMBE and doped with NH_3 (ammonia) as the source of the nitrogen dopant (see paragraphs 4.3.1 to 4.3.3 above). It follows that neither document D1 nor document D2 could have presented the skilled person with any indication that it is the combined use of MBE with nitrogen atomic radicals as dopant which makes possible the fabrication of a transducer that is practically useful in laser diodes or light emitting diodes having a room temperature electroluminescent spectrum with a maximum intensity at wavelengths of less than 550nm.

5.3.1 As for the disclosure in document D1 that "*at 77K, very weak whitish-blue emission is observed from the junction ...*" (page 839, left-hand column, third paragraph), the Board accepts the appellant's argument that the general decrease in luminescence efficiency with temperature as shown in document D12 (page 687, last paragraph) would mean that the "*very weak bluish white emission*" observed at 77K would result in an extremely weak and almost unobservable emission at room temperature (300K) and thus not be useful in light emitting diodes and laser diodes as required by claim 1 of the main request. In comparison, Figures 4 and 5 of the application show the luminescence spectra at 77K and 300K, respectively of semiconductor material according to the claimed invention. Although the units of intensity in these Figures are arbitrary units, the graph of Figure 5 in particular must be interpreted in the context of the application as a whole as illustrating room temperature intensity peaks at wavelengths less than 550nm, which are sufficient to be useful in light emitting diodes and lasers.

5.3.2 It follows that neither document D1 nor document D2 contain any indication how their teaching or the teaching of document D3 would need to be modified in order to arrive at a device as claimed in claim 1 of the application in suit.

5.3.3 For the foregoing reasons, the Board concludes that claim 1 of the main request involves an inventive step as required by Article 56 EPC having regard to documents D2 or D2 whether on their own or in combination with document D3.

6. In the Board's judgement, for the reasons given, claim 1 of the main request is novel as required by Article 54 EPC and involves an inventive step as required by Article 56 EPC.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first Instance with the order to grant a patent with the following documents:

   – Claims 1 to 3, and pages 2, 2a, 3to 7 of the description filed during the oral proceedings.

   – Figures 1, 2A, 2B, 3A, 3B, 4A, 4B, 5 and 6 as originally filed

The Registrar: The Chairman:

D. Meyfarth R. K. Shukla