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Datasheet for the decision of 04 April 2008

T 1195/06 - 3.3.06 Case Number:

Application Number: 98400082.8

Publication Number: 0853970

IPC: B01D 53/22

Language of the proceedings: EN

Title of invention:

An improved process for separation and recovery of perfluorocompound gases

Patentee:

L'AIR LIQUIDE, Société Anonyme pour l'Etude et l'Exploitation des Procédés Georges Claude

Opponent:

Solvay Fluor GmbH

Headword:

Carbon sieve membrane/AIR LIQUIDE

Relevant legal provisions:

Relevant legal provisions (EPC 1973):

EPC Art. 54, 56

Keyword:

- "Substantial procedural violation (no)"
- "Novelty (yes)"
- "Inventive step (yes): technical problem to be formulated taking into account the technical field of application expressly mentioned in the claims - no incentive for trying a known method of separation with expectation of success"

Decisions cited:

Catchword:

See points 3.2. and 3.5 of the reasons.



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Boards of Appeal

Chambres de recours

Case Number: T 1195/06 - 3.3.06

DECISION of the Technical Board of Appeal 3.3.06 of 04 April 2008

Appellant: L'AIR LIQUIDE, Société Anonyme pour l'Etude

(Patent Proprietor) et l'Exploitation des Procédés Georges Claude

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Representative: Vesin, Jacques

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Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted 07 July 2006 revoking European patent No. 0853970 pursuant

to Article 102(1) EPC 1973.

Composition of the Board:

Chairman: P.-P. Bracke
Members: L. Li Voti

A. Pignatelli

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Summary of Facts and Submissions

- I. The present appeal is from the decision of the Opposition Division to revoke the European patent no. 0 853 970, concerning a process for separating and recovering perfluorocompound gases, which patent was granted with a set of 47 claims, claim 1 of which reading as follows:
 - "1. A process to recover at least one perfluorocompound gas from a gas mixture originating from a semiconductor manufacturing process, comprising the steps of a) providing feed flow stream of said gas mixture comprising at least one perfluorocompound gas, said perfluorocompound gas being defined as a compound comprising C, S and/or N atoms wherein all or all but one hydrogen have been replaced by fluorine, and at least one carrier gas, said gas mixture being at a first pressure and a first temperature;
 - b) providing a first membrane having a feed side and a permeate side and exhibiting preferential permeation of the carrier gas, having selectivity SEL, defined as D_cS_c/D_pS_p greater than 1.0, wherein,
 - $\ensuremath{D_{\text{p}}}$ is the mobility selectivity of a perfluoro compound gas
 - S_{p} is the solubility selectivity of the perfluoro compound gas
 - $D_{\rm c}$ is the mobility selectivity of a carrier gas $S_{\rm c}$ is the solubility selectivity of the carrier gas;
 - c) contacting the feed side of said membrane with said gas mixture;

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- d) withdrawing from the feed side of said membrane as a first non-permeate stream at a pressure which is substantially equal to said predetermined pressure a concentrated gas mixture comprising essentially the at least one perfluorocompound gas,
- e) withdrawing from the permeate side of said membrane as a first permeate stream a depleted gas mixture consisting essentially of the at least one carrier gas, and wherein said first membrane is a carbon sieve membrane or zeolite coated or zeolite filled membrane."

Dependent claims 2 to 47 relate to particular embodiments of the claimed process.

II. In its notice of opposition the Opponent sought revocation of the patent on the grounds of Article 100(a) EPC 1973.

The Opponent referred *inter alia* to the following documents:

- (1): Ullmanns Encyclopedia of Industrial Chemistry, 5. edition (1990), pages 189 to 207;
- (2): Separation Science and Technology, 18(8), (1983), pages 723 to 734; and
- (5): US-A-4685940.

In the communication appended to the summons to oral proceedings the Opposition Division informed the parties that only the inventive step of the subject-matter of claim 1 would have to be discussed during oral proceedings since no other grounds of opposition had been cited and that any submission filed after the deadline of 28 April 2006 might be discarded.

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During the oral proceedings before the Opposition Division the Opponent requested the introduction of a new ground of opposition under Article 100(b) EPC 1973 and raised for the first time a novelty objection based on the disclosure of document (5).

- - the new ground of opposition, based on Article 100(b) EPC 1973, submitted by the Opponent for the first time during oral proceedings, was not admissible since it had been filed very late and was not relevant for the maintenance of the patent as granted;
 - the ground of opposition concerning lack of novelty of the subject-matter of claim 1 over document (5), though having been submitted for the first time during oral proceedings, was admitted into the proceedings since it was relevant for the maintenance of the patent;
 - however, document (5) did not contain any disclosure of either a separation method involving the feeding of a gas mixture comprising a perfluorocompound (PFC) and a carrier gas to a carbon sieve membrane or a separation method wherein the gas mixture fed originates from a semiconductor manufacturing process;
 - therefore, the claimed subject-matter was novel over the disclosure of document (5).

As regards the inventive step of the claimed subjectmatter, the Opposition Division found that - 4 - T 1195/06

- the description of the patent in suit suggested that the claimed process was also applicable to gas mixtures not originating from a semiconductor manufacturing process; therefore, even though claim 1 required that the treated gas mixtures originates from a semiconductor manufacturing process, the technical problem underlying the patent in suit had to be formulated in more general terms as the provision of an alternative method for recovering PFCs from any gas mixture containing them;
- as disclosed in document (1), it was well known to the skilled person that membranes were suitable means for separating two gases; therefore, it was obvious for the skilled person to try a gas separation by means of membranes as an alternative to the known methods for recovering PFCs from a gas mixture such as burning or adsorption;
- moreover, document (5) taught that carbon sieve membranes were suitable for separating a PFC such as sulphur hexafluoride from inert gases such as helium or nitrogen;
- therefore, the skilled person would have tried the membranes of document (5), which had necessarily the selectivity required in claim 1, as an alternative to the known separation methods for recovering PFCs from a gas mixture;
- the subject-matter of claim 1 thus lacked an inventive step.

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IV. Appeal was filed against this decision by the Patent Proprietor (hereinafter Appellant).

The Appellant submitted *inter alia* the following document with the statement of the grounds of appeal:

(14): Mat.Res.Soc.Symp.Proc., vol. 344 (1994): "PFC Concentration and Recycle" by G.M. Tom et al., pages 267 to 272.

Moreover, it requested the reimbursement of the appeal fee and the referral of an important question of law to the Enlarged Board of Appeal.

With a letter of 11 February 2008 the Appellant submitted several auxiliary requests.

With the fax of 3 April 2008 the Respondent (Opponent) withdrew its opposition and communicated that it would not take part any longer to the proceedings.

Oral proceedings were held before the Board on 4 April 2008 in the presence of the Appellant only.

During oral proceedings the Appellant withdrew all the auxiliary requests submitted with the letter of 11 February 2008 as well as its requests for the reimbursement of the appeal fee and for a referral of an important question of law to the Enlarged Board of Appeal.

V. The Appellant submitted orally and in writing inter alia that

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- the Opposition Division had informed the parties in the communication appended to the summons to oral proceedings that only the inventive step of the subject-matter of claim 1 would have to be discussed during oral proceedings since no other grounds of opposition had been cited; moreover, the Division noted that any submission filed after the deadline of 28 April 2006 might be discarded;
- however, during the oral proceedings which were held on 31 May 2006, the opposition division allowed the Opponent to discuss at length a new ground of opposition based on Article 100(b) EPC 1973 and a new objection concerning lack of novelty of the claimed subject-matter;
- this amounted to a substantial procedural violation since the opposition division had stated in the communication appended to the summons that only the inventive step of claim 1 would have to be discussed during oral proceedings;
- the new ground of opposition under Article 100(b) EPC 1973 was not admissible;
- the claimed subject-matter was novel over document (5) inter alia because this document did not concern the treatment of a gas mixture originating from a semiconductor manufacturing process;
- the closest prior art was represented by document (14) concerning an adsorption method for separating and recovering PFCs from a gas mixture originating from a semiconductor manufacturing process;

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- the technical problem underlying the invention thus was not the recovering of a relatively pure PFC stream from any venting of PFCs but the recovering of PFCs from a mixture of gases originating from a semiconductor manufacturing process in an energetically efficient and reliable way compared to the process of document (14);
- since the membranes selected in claim 1 can separate CF₄ from the carrier gases, which separation cannot be satisfactorily carried out by means of the adsorption method disclosed in document (14), and the PFCs are recovered according to the claimed process at their initial pressure without requiring a change of pressure and a recompression as in the method of document (14), thereby saving energy, the claimed invention solved the underlying technical problem;
- moreover, the prior art did not suggest that a separation by means of a membrane could be used in the technical field of the semiconductor manufacturing processes, for example in the treatment of the effluent stream of an etching step or of a chemical vapour deposition step or of the reactor cleaning step, as a suitable alternative to the method of separation of document (14);
- in fact, document (5) illustrated only the separation of pure gases, e.g. sulphur hexafluoride and inert gases; therefore, the skilled person could not have predicted on the basis of this teaching if the same separation would have been possible in the treatment of gas mixtures originating from a semiconductor

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manufacturing process, which mixtures are not simply mixtures of a PFC and a carrier gas in a predetermined ratio but are extremely variable mixtures of unknown composition and unpredictable volume, containing PFCs, carrier gases and PFCs reaction products;

- therefore, the skilled person would not have had any incentive for trying the type of technology described in document (5) in order to solve the technical problem underlying the invention;
- thus, the claimed subject-matter involved an inventive step.
- VI. The Appellant requests that the decision under appeal be set aside and that the patent be maintained as granted.

Reasons for the Decision

- 1. Procedural issues
- 1.1 The Appellant submitted that the Opposition Division had committed a substantial procedural violation since it had stated in the communication appended to the summons to oral proceedings that at the oral proceedings only inventive step would have to be discussed and, to the contrary, it allowed the Opponent to discuss at length during oral proceedings a new ground of opposition based on Article 100(b) EPC 1973 and a new objection concerning lack of novelty of the claimed subject-matter (see point V above).

1.2 The Board notes that the Opponent raised during oral proceedings for the first time a new ground of opposition and a new objection concerning lack of novelty of the claimed subject-matter. Therefore, the Opposition Division, despite of what had been previously communicated in writing to the parties, had to let expose the Opponent its arguments in order to preserve its right to be heard before deciding upon the admissibility of the new grounds of opposition.

The fact that the new raised facts had been discussed at length is not a criterion of relevance for judging whether a procedural violation has been committed.

As it results from the minutes of the oral proceedings, both parties received sufficient time for expressing their arguments before the Opposition Division decided upon the admissibility of the new grounds.

1.3 The Board thus finds that the Opposition Division acted correctly during oral proceedings and applied correctly its discriminating power upon the admissibility of the new grounds of opposition.

Therefore, the Board has no reason for departing from the finding of the department of the first instance that the new ground of opposition under Article 100(b) 1973, raised for the first time by the Opponent during the oral proceedings before the Opposition Division, is inadmissible but that the new ground of opposition concerning lack of novelty of the claimed subjectmatter has to be considered admissible (see point III above).

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2. Novelty

The Board notes that the wording of the process claim 1, by mentioning explicitly that the gas mixture treated originates from a semiconductor manufacturing process (see point I above), requires as an essential feature that the gas mixture treated is one exiting a semiconductor manufacturing process.

The fact that the description of the patent in suit refers to the possibility of applying a similar separation method to other processes (page 4, lines 39 to 40 and page 12, lines 11 to 12) cannot thus be used for construing the claim as extending to these other processes which are not mentioned in the wording of claim 1.

As regards the novelty of the claimed subject-matter over the disclosure of document (5), the Board thus finds that this document does not disclose the treatment of a gas mixture originating from a semiconductor manufacturing process. This fact has not been contested.

Therefore, the claimed subject-matter is novel over the cited prior art.

- 3. Inventive step
- 3.1 The invention relates to a process to separate and recover perfluorocompound gases from a gas mixture originating from a semiconductor manufacturing process (see page 2, lines 5 to 8 of the patent in suit).

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As explained in the description of the patent in suit, the semiconductor industry uses perfluorocompound gases in admixture with carrier gases, for example inert gases, in various etching steps and chamber cleaning steps of the semiconductor manufacturing process (page 2, lines 12 to 16).

Since a reduction of the emission of perfluorocompound gases in the atmosphere is considered to be beneficial to the environment, various methods for decomposing or recovering such gases have been proposed. However, the decomposition processes lead mostly to the formation of hydrofluoric acid, which must also be abated. Therefore, the recovery of PFCs is considered to be a more environmentally responsible approach (page 2, lines 19 to 41).

Different methods based on the adsorption or on the low temperature trapping of PFCs have been suggested (page 2, lines 24 to 26 and 40 to 42).

One of these methods concerns the use of a dual bed adsorber based on activated carbon, wherein the beds adsorb the PFCs and not the carrier gases which can thus be vented; moreover, according to this method, one of the beds is in the adsorption mode while the other is regenerated and when the system is switched, for example, from the first to the second adsorber bed, the first bed is evacuated by means of a vacuum pump and the effluent is recompressed in order to recover the separated PFCs (page 2, line 53 to page 3, line 2).

However, this known method does not allow the separation of CF_4 which is then rejected with the vent gases; moreover, the absorber beds are very sensitive

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to moisture and any trace of water present in the feed (page 3, lines 2 to 4).

As to the technical problem underlying the invention, even though the description of the patent in suit refers to the possibility of applying the separation method described not only to a gas mixture originating from a semiconductor manufacturing process but also to other processes (page 4, lines 39 to 40 and page 12, lines 11 to 12), it cannot be ignored that the invention is defined by the wording of the claims, which requires that the treated gas mixture is not any gas mixture containing PFCs and carrier gases but one originating from a semiconductor manufacturing process.

Therefore, the technical problem underlying the invention indicated in the description cannot be interpreted, as decided by the department of first instance (see point III above), to relate in more general terms to the provision of an alternative method for recovering PFC compounds from any gas mixture containing them, thereby ignoring the technical field of application expressly mentioned in the claims.

The Board finds, to the contrary, that the technical problem underlying the patent in suit is formulated in the description as the provision of an alternative environmentally sound process for concentrating and recovering PFCs from a gaseous stream originating from a semiconductor manufacturing process which is effective even with important or extreme variations of flows or concentration and can be used with any feed (page 3, lines 21 to 24; 28 to 31; page 5, lines 9 to 12).

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3.3 Document (5), though relating to the use of specific membranes in a process for separating gases (column 1, lines 5 to 9), does not deal with the above mentioned technical problem and does not regard explicitly the recover of PFCs from an effluent stream originating from a semiconductor manufacturing process.

To the contrary, document (14) describes a process using a dual adsorber bed similar to that described in the description of the prior art in the patent in suit (see point 3.1 above) and concerns the provision of an environmentally friendly separation process for recovering PFCs from a gas mixture exiting a semiconductor manufacturing process (see page 267, abstract).

Therefore, the Board finds that document (14) is the most suitable starting point for evaluating inventive step, as submitted by the Appellant in writing and during oral proceedings.

The process of document (14) differs from the claimed subject-matter insofar as it does not involve the use of membranes which permeate preferentially carrier gases but adsorber beds which adsorb preferentially PFCs.

3.4 The technical problem underlying the invention can thus be defined as suggested in the patent in suit as the provision of an alternative environmentally sound process for concentrating and recovering PFCs from a gaseous stream originating from a semiconductor manufacturing process which is effective even with

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important or extreme variations of flows or concentration, can be used with any feed and is energetically more efficient than the method of document (14).

As explained by the Appellant during oral proceedings and indicated in the patent in suit, the selected membranes can separate CF₄ from inert gases, which separation cannot be satisfactorily carried out by means of the adsorption method disclosed in document (14) (see also page 269, second and third line below the figure), are not sensitive to moisture and the PFCs can be recovered at the same pressure at which they are fed to the membrane without the need of recompressing them as in the method of document (14), thereby saving energy (see also page 3, lines 28 to 39 and 50 to 54; page 5, lines 16 to 28; page 6, lines 22 to 39).

Therefore, the Board is convinced that the technical problem underlying the invention has been successfully solved by means of a process having the features of claim 1.

3.5 It was well known to the skilled person at the priority date of the patent in suit that it was possible to separate gases by means of membranes (see document (1), page 203, paragraph 3.3, line 1 to page 204, left column, line 9); however, as submitted by the Appellant, it was also known in the technical field of membranes that the behaviour of a mixture of components within a membrane cannot be generally predicted from the known behaviour of single components (see, for example, document (1), page 205, left column lines 4 to 7).

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Document (5), as well as document (2) which is of a similar content, teach that specific carbon membranes are able to separate SF6, i.e. a perfluorocompound gas, from carrier gases such as N_2 , O_2 , He (see document (5), column 2, line 55 to column 3, line 30; table 1, experiments 10 and 11; table 2, experiments 7 to 9 and table 3, lines 58 to 60; and document (2), page 731, lines 15 to 17 below table 2; page 732, lines 7 to 8 of the passage entitled "Comparison with Polymer Membranes"; page 733, table 3, last two lines). However, this teaching concerns the separation of single pure gases and is based on the measurement of the behaviour of single components (see document (5), column 5, lines 56 to 63; column 6, lines 8 to 10; and document (2), page 726, lines 1 to 2 of the passage entitled "Permeability System"). Furthermore, document (5) concerns only the separation of a mixture of two gases of different molecule size (column 2, lines 63 to 67) and does not concern the separation of such gases in admixture with other reaction products as necessarily present in the effluent stream of a semiconductor manufacturing process, which, as submitted by the Appellant in writing and during oral proceedings and suggested in document (14), are not simply a mixture of a PFC and a carrier gas in a predetermined ratio but is an extremely variable mixture of unknown composition and unpredictable volume containing PFCs, carrier gases and PFCs reaction products (see e.g. document (14), page 267, lines 6 to 10 of the passage entitled "Introduction").

Therefore, the Board finds that the skilled person could not have predicted on the basis of the teaching of documents (5) or (2) and of his common general

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knowledge if the membranes used in document (5) or (2) would have been efficient for separating a gas mixture originating from a semiconductor manufacturing process.

Furthermore, it has to be noted that even though the selectivity of carbon membranes was known from documents (5) and (2), which are about 10 years older than the priority date of the patent in suit, a separation process involving the use of a membrane had not been taken into consideration by the semiconductor manufacturing industry up to the priority date of the patent in suit as explained in the patent with reference to a symposium hold in June 1994 (see page 2, line 24 to page 3, line 4).

Therefore, the Board concludes that the skilled person would have not found any incentive in the prior art for trying the separation method taught in documents (5) or (2) as alternative to the method of document (14) with the expectation of successfully solving the technical problem underlying the invention.

Therefore, the subject-matter of claim 1 involves an inventive step.

For the same reasons, the dependent claims involve an inventive step.

Order

For these reasons it is decided that:

The decision under appeal is set aside.

The patent is maintained as granted.

The Registrar:

The Chairman:

C. Vodz P.-P. Bracke