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D E C I S I O N
of 1 July 1998

Case Number: T 0940/95 - 3.3.5

Application Number: 87302691.8

Publication Number: 0240270

IPC: C01B 23/00

Language of the proceedings: EN

Title of invention:
One-step process for purifying an inert gas

Patentee:
The BOC Group, Inc.

Opponent:
Praxair Technology, Inc.

Headword:
Purifying an inert gas/BOC

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step - no; obvious solution of a technical problem"

Decisions cited:
-

Catchword:
-

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number: T 0940/95 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 1 July 1998

Appellant: (Proprietor of the patent)
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Decision under appeal: **Decision of the Opposition Division of the European Patent Office posted 11 September 1995 revoking European patent No. 0 240 270 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: R. K. Spangenberg
Members: G. J. Wassenaar
M. B. Günzel

Summary of Facts and Submissions

I. The appeal is from the decision of the Opposition Division revoking European patent No. 0 240 270, which was granted in response to European patent application No. 87 302 691.8.

II. The decision under appeal was based on two sets of 16 claims, submitted as main and auxiliary request during oral proceedings before the Opposition Division. It was held that the subject-matter of claim 1 of both the main and the auxiliary request lacked an inventive step having regard to the state of the art (Article 56 EPC). In the decision under appeal, inter alia, the following documents were cited:

D2: Römpps Chemie-Lexicon 7th ed. 1974/1975, pages 2324, 2325 and 2909,

D10: Chemical Abstracts volume 104 (1986), abstract no. 21368g,

D12: Linde Union Carbide Leaflet "Inert gas purifier" 1984 (L-5680 83-D650 5M 9/84) and

D14: Tompkins "Chemisorption of Gases on Metals" (1978), pages 18 and 19.

III. With the statement of grounds of appeal, the Appellant submitted eight sets of claims, a main request and seven auxiliary requests. It was argued that it was not obvious to combine the teaching of D10 with any of the other cited documents so that the conclusion of the Opposition Division that the main request lacked an inventive step was wrong. During oral proceedings,

which were held on 1 July 1998, it was stressed that in the prior art it was regarded as necessary to use a two-step process with two different absorbents in order to remove all the contaminants from an inert gas for use in semiconductor manufacturing processes. In this respect reference was made to D12. The Appellant surprisingly discovered that with the absorbent used in the patent in suit all the contaminants, normally present in nitrogen from an air plant, could be reduced in one step to a level required by the semiconductor industry.

IV. The Respondent contested the Appellant's arguments. It was argued that the Opposition Division's conclusion also applied to the main request now on file. It was further argued that even the most restricted independent claim on file, claim 1 of the 7th auxiliary request, was still obvious in view of the cited prior art. In this respect reference was made to some further documents.

V. The Appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of his main request as filed with his Grounds of Appeal (claims 1 to 16 submitted on 15 January 1996). As auxiliary request, he requested that the patent be maintained on the basis of any of auxiliary requests 2 to 7 as filed with his Grounds of Appeal and in the order indicated therein.

The Respondent requested that the appeal be dismissed.

At the end of the oral proceedings, after deliberation, the decision was announced that the appeal was dismissed.

VI. Claim 1 of the main request reads as follows:

"A process for purifying an inert gas stream containing in total less than 1000 volumes per million of impurities comprising oxygen and one or more other impurities selected from carbon monoxide, carbon dioxide, hydrogen and water vapour, in which said inert gas stream is passed in a single step through a bed of particulate material comprising nickel in an amount of at least 5% by weight as elemental nickel, thereby concurrently to remove said oxygen and one or more of the other impurities from the inert gas stream, and a purified inert gas is withdrawn."

Claim 1 of the second auxiliary request differs therefrom in the additional requirement that the treatment takes place at a temperature in the range of 0° to 50°C.

Claim 1 of the third auxiliary request differs from claim 1 of the second auxiliary request in the additional requirement that the particulate material comprises both elemental nickel and nickel oxide.

Claim 1 of the fourth auxiliary request differs from claim 1 of the main request in the additional requirement that the inert gas stream to be purified comprises oxygen, carbon monoxide and hydrogen and that these impurities are removed from the inert gas stream together with any carbon dioxide and any water vapour impurity present therein.

Claim 1 of the fifth auxiliary request combines the features of claims 1 of the third and fourth auxiliary

request.

Claim 1 of the sixth auxiliary request combines the features of claims 1 of the second and fourth auxiliary request.

Claim 1 of the seventh auxiliary request combines the features of claims 1 of the third and sixth auxiliary request.

Reasons for the Decision

1. The appeal is admissible.

2. The only issue discussed in the appeal proceedings was inventive step. In view of the outcome of these appeal proceedings the Board considers it unnecessary to give a reasoned opinion on any other issue.

3. *Main request*

3.1 According to the patent in suit, the invention relates to a process for purifying an inert gas stream by removing impurities such as oxygen, hydrogen, carbon monoxide, carbon dioxide and water vapour to such an extent that the purified gas can be used in the semiconductor industry, in particular in the production of integrated circuits (page 2, lines 3 to 10).

3.2 The only prior art document on file disclosing a purifying process whereby apart from oxygen at least one other of the above-mentioned impurities is said to be removed in order to provide a purified inert gas suitable for highly sensitive processes in electronics

production is D12 (page 1, left column). The Board is, therefore, of the opinion that D12 represents the closest prior art. This document discloses an inert gas purifier which removes trace amounts of water, oxygen and carbon dioxide from an inert gas in a two-step process, whereby in a first step oxygen is removed from the stream as it combines with activated nickel in a first reactor, followed by feeding the gas stream from the nickel-containing reactor to a molecular sieve absorber bed where water vapour and carbon dioxide are removed. It is further indicated that in a regeneration process the nickel oxide formed by the reaction with oxygen is reduced to nickel, which proves that the first reactor contains metallic nickel (page 2, left column).

3.3 According to the patent in suit, the invention is concerned with solving the problem of providing a process which is able to purify in a single step an untreated inert gas stream containing in total less than 1000 volumes per million of impurities comprising oxygen and one or more other impurities selected from carbon monoxide, carbon dioxide, hydrogen and water vapour (page 2, lines 49 to 53).

Starting from D12, the Board regards the problem underlying the invention as providing a simplified process for producing a purified inert gas. This definition of the problem is clearly in agreement with the above-mentioned statement in the patent specification. According to claim 1, this problem is solved by using an inert gas stream containing in total less than 1000 volumes per million of impurities comprising oxygen and one or more other impurities selected from carbon monoxide, carbon dioxide, hydrogen

and water vapour, and passing said inert gas stream in a single step through a bed of particulate material comprising nickel in an amount of at least 5% by weight as elemental nickel.

According to the examples in the patent in suit, the impurities can be reduced to a level of less than 0.1 ppmv (parts per million by volume); ie comparable to the level mentioned in D12. Since the use of only one bed of particulate material can be regarded as a technical simplification the Board is satisfied that the process according to claim 1 solves the above-mentioned problem. It remains to be decided whether the claimed solution was obvious to the skilled person trying to solve the problem having regard to the prior art.

3.4 The inert gas purifier of D12 has been specially developed to cope with contamination problems such as pipeline contamination and leaks in a pipeline system, whereby air and moisture can be introduced into a pressurized inert gas stream (page 1, middle column). As also pointed out in the patent in suit, carbon dioxide and water vapour are not present as contaminants in nitrogen leaving the cryogenic air separation plant but are picked up by the inert gas stream during the handling and delivery thereof between the air separation plant and the end-use location (page 3, lines 11 to 14). Since the second absorber step in the process of D12 only serves to remove carbon dioxide and water vapour, it is obvious to a skilled person that if the inert gas to be treated does not contain the latter contaminants, as is within the scope of present claim 1, the second step is redundant. Thus for treating inert gases having no or only

insignificant traces of carbon dioxide and water vapour, as available from a cryogenic air separation plant, it is obvious to use only the reactor comprising the nickel metal.

3.5 Apart from the presence of metallic nickel, D12 does not disclose the composition of the bed particles in the reactor. In order to find a suitable particle composition, the skilled person will look into other recent documents relating to the purification of an inert gas stream with a bed of nickel containing particles. In a standard handbook for such a search, The Chemical Abstracts, he will find D10, an abstract of DD-A-224 229, which discloses a process for the removal of oxygen from inert gases by using a pelletized Ni-Al_2O_3 adsorbent at 273-423 K, having a total Ni content of 43-55% and a metallic Ni content of at least 30%. According to the example of D10, such adsorbent is capable of reducing the oxygen content of a nitrogen gas stream from 0.89 vol.% (8900 ppmv) to less than 2 ppmv. It is obvious to the skilled person that the adsorbent of D10 is suitable to reduce the oxygen content of nitrogen from a cryogenic air plant, having generally an oxygen content below 10 ppmv (see page 3, lines 14 to 17 of the patent in suit), to a much lower level.

The Board is therefore convinced that a skilled person, trying to solve the problem of removing impurities from nitrogen from a cryogenic air plant to a level which allows its use in highly sensitive processes in electronics production (D12, page 1, left column) in a simple way, will take into consideration passing the nitrogen through a bed of particles according to D10. If the nitrogen contains no or only negligible traces

of water vapour and carbon dioxide, as is generally the case with nitrogen from a cryogenic air plant, the skilled person would expect that passing the nitrogen through a bed of the particles according to D10 was sufficient to obtain a purified inert gas suitable for the purpose mentioned in D12. This obvious solution of the above-mentioned problem falls within the scope of present claim 1.

3.6 The Board cannot accept the Appellant's argument that, since the skilled person did not know that the particles according to D10 would remove other impurities than oxygen, he would not use such particles if he also wanted to remove impurities such as hydrogen and carbon monoxide. It belongs to the general knowledge of the skilled person that nickel also adsorbs hydrogen and carbon monoxide and that these components can be removed from an inert gas by contacting the gas with nickel; see the textbook passages D2 (in particular page 2324, right-hand column, lines 29 to 39 under "Nickel") and D14 (the sentence bridging pages 18 and 19). But even if, in the Appellant's favour, it is assumed that the skilled person was not aware that hydrogen and carbon monoxide would be adsorbed, the argument must fail. Said impurities are always present in nitrogen from a cryogenic air plant as indicated in the patent in suit (page 3, lines 2 to 7) and confirmed by the parties during oral proceedings. The Appellant also confirmed that no additional measures beyond the presence of nickel- containing particles are necessary to remove hydrogen and carbon monoxide together with oxygen from the inert gas stream. Their concurrent removal is thus the inevitable consequence of the use of the said particles for removing oxygen from a nitrogen gas stream from a cryogenic air plant. Stating explicitly

the consequences of an obvious process does not make the process less obvious. The subject matter of claim 1, therefore, does not involve an inventive step.

4. *Auxiliary requests*

4.1 D12 does not explicitly disclose at which temperature the inert gas stream is passed through the nickel-containing bed. Because the document is silent about the adsorption temperature but indicates that the regeneration takes place at an elevated temperature, the skilled person will assume that the removal of the impurities can be performed at room temperature. The fact that, in the only example of D10, the inert gas was treated at a temperature of 90°C will not deter the skilled person from using temperatures in the range of 0 to 50°C as required by claim 1 of the second auxiliary request, since D10 recommends a temperature range of 0 to 150°C.

4.2 The additional features claimed in the independent claims of the other auxiliary requests such as the presence of nickel oxide and the use of an inert gas comprising oxygen, carbon monoxide and hydrogen are either known from D10 (nickel oxide) or are the inevitable consequence of the treatment of nitrogen gas from a cryogenic air plant with an adsorbent according to D10. Thus the arguments against inventive step with regard to the process of claim 1 of the main request equally apply to the subject matter of the claims of the auxiliary requests on file.

5. For these reasons the Board holds that the subject matter of all the independent claims of all requests on file lacks an inventive step within the meaning of

Article 56 EPC so that the patent in suit cannot be maintained on the basis of any of the Appellant's requests.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

S. Hue R. Spangenberg