BESCHWERDEKAMMERN DES EUROPÄISCHEN PATENTAMTS

BOARDS OF APPEAL OF THE EUROPEAN PATENT OFFICE

CHAMBRES DE RECOURS DE L'OFFICE EUROPEEN DES BREVETS

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File Number: T 688/89 - 3.3.3

Application No.: 84 306 447.8

Publication No.: 0 164 470

Title of invention: Coating process and moisture-curable organopolysiloxane compositions therefor

Classification: C08L 83/14

D E C I S I O N
of 26 January 1993

Applicant: Dow Corning Corporation

Headword:

EPC Article 56

Keyword: "Inventive step, yes, after amendment"

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Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number : T 688/89 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 26 January 1993

Appellant : Dow Corning Corporation
3901 S: Saginaw Road
Midland
US - Michigan 48640 (US)

Representative : Laredo, Jack Joseph
Elkington and Fife
Prospect House
8 Pembroke Road
GB - Sevenoaks, Kent TN13 1XR (GB)

Decision under appeal : Decision of the Examining Division 014 of the European Patent Office dated 19 June 1989 refusing European patent application No. 84 306 447.8 pursuant to Article 97(1) EPC.

Composition of the Board :

Chairman : F. Antony
Members : R.A. Lunzer
M.K.S. Aúz Castro

Summary of Facts and Submissions

I. European patent application No. 84 306 447.8, published as No. 164 470, was filed on 20 September 1984, having a priority date of 30 April 1984 derived from US application No. 605 315.

II. By its decision given on 1 June 1989, the Examining Division refused the application. The decision made reference to the following prior art:

(1) US-A-3 175 993
(2) EP-A-0 158 427 (published 16.10.85)
(3) US-A-4 269 963,

and found that for the Contracting States DE, FR, GB, SE, Claim 1 lacked novelty having regard to document (2). In relation to the remaining Contracting States (BE, IT, NL) it found that the composition defined by Claim 1 and the method of Claim 5 were lacking in any inventive step having regard to the disclosures of documents (1) and (3).

III. Claims 1, 2 and 5 as filed by the Appellant on 23 December 1988, and as considered in the decision of the Examining Division, were in the following form:

"1. An organopolysiloxane fluid, stable in the absence of moisture, which has the formula

$$Z^1R^1_2SiO(Me_2SiO)_x(DR^1SiO)_y(R^1_2SiO)_zSiR^1_2Z^1$$

wherein

Me denotes a methyl radical,

D denotes an alkoxysilylorganic radical having the formula $-Q^1SiR^1_a(OR^2)_{3-a}$,

R^1 denotes a monovalent hydrocarbon or substituted hydrocarbon radical having from 1 to 6 carbon atoms,

R^2 denotes a methyl, ethyl, propyl or methoxyethyl radical,

Q^1 denotes a divalent organic radical free of sulfur atoms,

Z^1 denotes a D radical or an R^1 radical,

$\underline{a}$ has a value of 0 or 1,

$\underline{x}$ has an average value of at least 50,

$\underline{y}$ has an average value of from 1 to $(\underline{x} + \underline{y} + \underline{z})/4$,

$\underline{z}$ has an average value not exceeding the value of $\underline{x}$ and the organopolysiloxane fluid contains an average of at least 3 D radicals per molecule.

2. A liquid composition which cures to a non liquid composition when exposed to moisture said liquid composition being prepared by mixing under substantially anhydrous conditions, components comprising:

(A) an organopolysiloxane fluid as defined in claim 1; and

(B) a metal ester, soluble in said liquid composition, selected from the group consisting of titanium esters, zirconium esters, hafnium esters and vanadium oxide esters; the amounts of components (A) and (B) being sufficient to provide a tack-free cure time of less than 120 seconds for the liquid composition.

5. A method comprising

(i) applying to a substrate a liquid composition which cures to a non-liquid composition when exposed to moisture, said

liquid composition being prepared by mixing, under substantially anhydrous conditions, components comprising

(A) an organopolysiloxane fluid as claimed in Claim 1, and

(B) a metal ester, soluble in said liquid composition, selected from the group consisting of titanium esters, zirconium esters, hafnium esters and vanadium oxide esters; the amounts of components (A) and (B) being sufficient to provide a tack-free cure time of less than 120 seconds for the liquid composition and

(ii) exposing the applied liquid composition to a moisture-containing environment until the applied composition has attained at least the desired amount of cure."

IV. An appeal against that decision was lodged on 24 July 1989, the appeal fee was paid on 25 July 1989, and the Grounds of Appeal were filed on 5 October 1989. In the Statement of Grounds of Appeal, the Appellant argued that the disclosure of document (2) was confined to compositions which necessarily included an aminoorgano-siloxane, and thus could not affect the novelty of compositions of the alleged invention, which did not include that constituent. It also attacked the finding of lack of inventiveness based on documents (1) and (3), contending that the alleged invention had brought about a significant advance in the art which was by no means foreshadowed by the teachings of either of these documents. Together with its Statement of Grounds of Appeal, the Appellant filed two auxiliary requests.

V. In response to a telephone communication from the Board indicating that it regarded the existing requests as remaining open to objection, on 20 October 1992 the Appellant filed a new set of Claims 1 to 7. In response to a further written communication from the Board of 27 November 1992, a further modification was made by a letter of 8 January 1993 as the Appellant's sole request. Claims 1 and 5 as finally considered by the Board were in the following form:

"1. A liquid composition which cures to a non liquid composition when exposed to moisture, said liquid composition being prepared by mixing, under substanially anhydrous conditions, an organopolysiloxane component (A) and a metal ester component (B) soluble in said liquid composition, characterised in that:

(i) said organopolysiloxane component (A) consists of an organopolysiloxane fluid, stable in the absence of moisture, which has the formula (IIa):

$$Z^1R^1_2SiO(Me_2SiO)_x(DR^1SiO)_y(R^1_2SiO)_zSiR^1_2Z^1 \qquad (IIa)$$

wherein

Me denotes a methyl radical,
D denotes an alkoxysilylorganic radical having the formula $-Q^1SiR^1_a(OR^2)_{3-a}$,
R^1 denotes a monovalent hydrocarbon or substituted hydrocarbon radical having from 1 to 6 carbon atoms,
R^2 denotes a methyl, ethyl, propyl or methoxyethyl radical,
Q^1 denotes a divalent organic radical free of sulfur atoms,
Z^1 denotes a D radical or an R^1 radical,

a has a value of 0 or 1,
x has an average value of at least 50,
y has an average value of from 1 to (x + y + z)/4,
z has an average value of from 0 to not more than x, the organopolysiloxane fluid having an average of at least 3 D radicals per molecule, said organopolysiloxane component (A) excluding any aminoorganosiloxane, and

(ii) said metal ester component (B) consists of a titanium ester in an amount that will provide from 0.5 to 4.0 parts by weight of titanium per 100 parts by weight of said organopolysiloxane component (A), provided that said liquid composition does not contain an organopolysiloxane having the formula

$RMe_2/SiO(Me_2SiO)_{142}(MeRSiO)_3SiMe_2R$,

wherein R denotes $-CH_2CH_2Si(OMe)_3$ or
$Me_3SiO(Me_2SiO)_{210}(MeRSiO)_{12}-SiMe_3$,
wherein R denotes $-CH_2CH_2Si(OMe)_3$.

5. A method comprising

(i) applying to a substrate a liquid composition as defined in Claim 1; and

(ii) exposing the applied liquid composition to a moisture-containing environment until the applied composition has attained at least the desired amount of cure."

VI. The Appellant requested that the decision under appeal be set aside, and a patent granted on the basis of Claims 1 to 7 filed on 20 October 1992, as amended on 8 January 1993 and a description yet to be adapted.

Reasons for the Decision

1. The appeal is admissible.

2. Admissibility of amendments

Claim 1 as it now stands combines the subject-matter of Claims 1, 2 and 5 of the application as filed, subject to two further limitations. These are an express disclaimer of an additional component being an aminoorganosiloxane, and also a disclaimer of the particular polysiloxanes which are disclosed in comparative Examples 1 and 2 of document (2), so as to avoid an objection of lack of novelty based on them. Claim 2 corresponds to original Claim 3. Present Claim 3 is directed to a feature of original Claim 4, the resulting generalisation being acceptable in view of the disclosure at page 19, lines 1 to 7, and page 10, line 1, of the original documents. The formula of present Claim 4 is also taken from original Claim 4, the letters of some of the symbols used having been changed without any change of substance. Claim 5 corresponds essentially to original Claim 6, and Claims 6 and 7 to original Claims 7 and 8.

3. Novelty

3.1 The Board is satisfied that there being no longer any claim to component (A) as such, and appropriate disclaimers having been introduced, Claims 1 and 5 are now novel over document (2) in terms of Article 54(3) EPC.

3.2 Now that Claim 1 has been amended so as to specify the proportions of catalyst in a range well outside the disclosure of document (1) (see the figures tabulated in point 5.4 below), and as document (3) is not relevant to

novelty because it relates to sulphur containing organosiloxanes, the Board is satisfied that the alleged invention is novel over the cited prior art for the purposes of Article 54(2) EPC.

4. The closest prior art

As document (2) is prior art solely by virtue or Article 54(3) EPC (and is therefore excluded from consideration when dealing with obviousness having regard to the provisions of Article 56 EPC), the Board agrees with the Examining Division that document (1) is the closest prior art to be used as a starting point for such considerations. This document refers to the well known and widely used polysiloxane room temperature curing systems which had been used in the past, in which reaction with atmospheric moisture leads to the evolution of a carboxylic acid, usually acetic acid, and to the curing of the composition (col. 1, lines 28 to 43). It then refers to the use of a new class of organopolysiloxanes, which are endblocked with - at least two - alkoxylated siloxane groups, and which, when used in the presence of a suitable catalyst, is said to produce useful compositions (col. 1, lines 44 to 51). The catalysts proposed overlap those in accordance with the present alleged invention (col. 4, lines 26 to 54), but are used at the rate of 0.1 to 2% by weight based on the weight of the siloxane (col. 4, line 57). (The relationship between proportions of catalyst measured in terms of its titanium content, as contrasted with the quantities of the catalyst compound itself, is dealt with in paragraph 5.6 below.) The compositions of document (1) are also said to be useful for coating (col. 4, line 11).

5. Problem and its solution

5.1 The application in suit includes in its introduction at page 2, line 12 to page 3, line 3 a brief discussion of document (1), and points to three matters which are said to distinguish that disclosure from the alleged invention. First, reference is made to the different class of siloxanes embraced by document (1); secondly emphasis is placed on their slow curing rates; and thirdly attention is drawn to the fact that the amount of catalyst there used is in the range of 0.1 to 2%.

5.2 Regarding the choice of the polysiloxanes to be used, it is explained in the application in suit at page 13, lines 6 to 11, that when the number of alkylsiloxyorganic radicals has a value of at least three, as is now required by the amended Claim 1 in suit, there must be at least one alkylsiloxyorganic radical which is bonded to a non-terminal siloxane unit of the organopolysiloxane. The existence of this distinction over the disclosure of document (1) was accepted by the Examining Division at page 6 of the decision under appeal. It was held there that in accordance with the disclosure of document (1), the units $ZSi(R)_{3-a}(OR')_a$ were linked to a single Si atom via an $(SiOR_2)_x$ group. That was not the case for the D moiety $(DR^1SiO)_y$ of the alleged invention. The slower reaction rate which results when the organopolysiloxane contains only two alkoxysilylorganic radicals is also illustrated in Example 4 of the application in suit.

5.3 Thus, in comparison with the teaching of document (1), the two changes made in accordance with the alleged invention are that a different siloxane is used, and in addition the proportion of catalyst is increased.

5.4 The curing times given in the application in suit are expressed in seconds, while those expressed in document (1) are expressed in minutes and hours. Whereas in document (1), it is only in Example 2 that a figure is given for the time taken to achieve a complete cure, which there required 72 hours, the numerous Examples of the application in suit indicate that compositions in accordance with the alleged invention readily give a complete curing time in the range of 5 seconds to 2 minutes, with many of the Examples showing times towards the lower end of that range. The actual curing times are influenced by the choice of the reactive polysiloxane, and the quantity of catalyst used, the shortest curing times being shown when 10% or more of catalyst is used.

5.5 Accordingly, the objective problem of the application may be regarded as being the attainment of very rapid curing, without the use of added energy, such as to make the compositions suitable for use in high-speed coating operations, e.g. paper and fibre coating, where the application and curing of the composition is more or less immediately followed by further operations requiring a cured, non-tacky coating. The solution to that problem lies in the use of the specific polysiloxanes, and the proportions of catalyst, specified in Claim 1.

5.6 The choice of the polysiloxanes has been dealt with in paragraph 5.2 above. Regarding the proportions of catalyst, as is indicated at page 19, lines 1 to 10 of the application in suit, the claimed range of 0.5 to 4.0 parts by weight of titanium per 100 parts by weight of organic polysiloxane corresponds to 3 to 24 parts of the preferred titanium ester, tetraisopropyl titanate, or 3.7 to 29.7 parts of the preferred partially methanolated tetraisopropyl titanate. Those amounts are to be contrasted with the proportion of catalyst in accordance

with document (1), stated at col. 4, lines 56 to 57 not to be critical, and to be in the range of 0.1 to 2.0% by weight, while its examples involve the use of about 1% of catalyst. The curing times achieved in accordance with the alleged invention mentioned in paragraph 5.4 above show that the alleged invention affords a credible solution to the problem of attaining very much shorter curing times is provided.

6. Inventive step

6.1 The issue of inventiveness turns on whether a skilled person, having as his starting point the disclosure of document (1), and confronted with the problem of finding a polysiloxane composition capable of the above mentioned very rapid curing, without the use of added energy, would have found in it, or in document (3), any pointer in the direction of solving that problem by using the specific polysiloxanes, together with the larger proportions of catalyst, both of which now characterise Claim 1 of the application.

6.2 The Board would accept that a skilled worker, starting from the teaching of document (1), and seeking some increase in the rate of curing, might have tried as a first step increasing the proportion of catalyst. Any finding of such an improvement resulting alone from increasing the amount of catalyst might not suffice to support the existence of an inventive step.

6.3 However, in the light of the problem as defined above, the skilled person would not reasonably have expected that by modifying the structure of the polysiloxanes of document (1) to those now covered by Claim 1, coupled with increasing the proportion of catalyst, he could reduce the curing times of these compositions from the order of

72 hours disclosed in document (1), to a time within the range of 5 seconds to a few minutes as disclosed in the application in suit. The structural difference cannot in the view of the Board be designated as only "minimal" (decision under appeal page 6, para. 3). This is particularly so because document (1) emphasises the structural element of the siloxanes as being "endblocked" with alkoxylated silcarbane groups, this contributing generally, as is shown by Example 5 of the application in suit, to a more rapid cure rate; the claimed subject-matter, however, does not require such groups to be in a terminal position. Accordingly, the Board is not satisfied that document (1) would have directed the skilled worker in the direction of the invention.

6.4 This result is unaffected by taking into account the teaching of document (3). Apart from the fact that that document relates to different compositions, i.e. sulphur containing organosiloxanes, it is noted that each of its Examples 1 to 8 refers to curing by standing overnight, while Examples 9 and 10 refer to curing for five days at room temperature. It is therefore not a pointer in the direction of finding a system capable of affording very rapid curing.

7. Conclusion

The Board concludes that the subject-matter of Claim 1 of the application involves an inventive step as required by Article 56 EPC, and is therefore patentable. The same applies to the dependent Claims 2 to 7, Claims 2 to 4 deriving their validity through dependency on Claim 1, and Claims 5 to 7 relating to a method of using an organopolysiloxane fluid as claimed in Claim 1.

Order

For these reasons, is is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the Examining Division with the order that a patent be granted on the basis of Claims 1 to 7 filed on 20 October 1992, as amended on 8 January 1993, the description to be adapted.

The Registrar: The Chairman:

E. Görgmaier F. Antony