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Datasheet for the decision of 21 March 2007

Case Number: T 1750/06 - 3.3.03

Application Number: 01310286.8

Publication Number: 1217042

IPC: C08L 83/04

Language of the proceedings: EN

Title of invention:
Continuous preparation of a liquid silicone rubber composition

Applicant:
GENERAL ELECTRIC COMPANY

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step - no (all requests on file)"

Decisions cited:
-

Catchword:
-

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Case Number: T 1750/06 - 3.3.03

D E C I S I O N
of the Technical Board of Appeal 3.3.03
of 21 March 2007

Appellant: GENERAL ELECTRIC COMPANY
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 4 May 2006 refusing European application No. 01310286.8 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: R. Young
Members: W. Sieber
C. Heath

Summary of Facts and Submissions

I. European patent application No. 01 310 286.8 in the name of GENERAL ELECTRIC COMPANY, filed on 10 December 2001, claiming a US priority of 20 December 2000 (US 742911), and published under No. 1 217 042, was refused by a decision of the Examining Division issued on 4 May 2006. The decision was based on a set of 15 claims filed with letter dated 23 February 2005, whereby Claim 1 read as follows:

"A process of preparing a liquid silicone rubber composition, comprising:

introducing a filler, filler treating agent (16) and vinyl-terminated silicone polymer into an extruder (22) having a length to diameter ratio of at least greater than 70; and

continuously compounding and devolatilizing said filler, treating agent (16) and silicone polymer into said liquid silicone rubber composition in said extruder (22)."

Claims 2-15 were directed to preferred embodiments of the process of Claim 1.

II. According to the decision, the subject-matter of Claims 1 and 2 was obvious in view of D1.

D1: US-A-4 649 005.

III. On 7 July 2006, the appellant (applicant) filed a notice of appeal against the above decision with simultaneous payment of the prescribed fee.

A statement setting out the grounds of appeal and including new Claims 1-14 was filed on 13 September 2006.

IV. In a communication accompanying a summons to oral proceedings to be held on 21 March 2007 the board gave its preliminary opinion that Claim 1 contained added subject-matter, and that the claimed subject-matter appeared to lack an inventive step, starting out either from the disclosure of D1 or the disclosure of:

D2: US-A-5 198 171,

which was mentioned in the European Search Report.

V. In preparation for the oral proceedings, the appellant submitted with a letter dated 21 February 2007 Claims 1 according to a main request as well as to 1st to 3rd auxiliary requests.

VI. Oral proceedings were held before the board on 21 March 2007. Following a discussion as to whether or not Claims 1 filed with letter dated 21 February 2007 met the requirements of Article 84 EPC, the appellant filed a set of Claims 1 according to a new main request as well as to new 1st to 3rd auxiliary requests.

(a) Claim 1 according to the main request read as follows:

"A process of preparing a liquid silicone rubber composition, comprising:

introducing a filler, treating agent (16) and silicone polymer into an extruder (22) having a length to diameter ratio of at least greater than 70, wherein the silicon polymer is a vinyl-terminated polydimethylsiloxane having a viscosity varying from 100 to 2,000,000 mPas (100 to 2,000,000 cps) at 25°C and wherein the treating agent comprises silanol-stopped polydimethylsiloxane, octamethylcyclotetrasiloxane (D4) or hexamethyldisilazane (HMDZ) and wherein said filler contains silanol groups;

continuously compounding and devolatilizing said filler, treating agent (16) and silicone polymer into said liquid silicone rubber composition in said extruder (22);

controlling temperature in said extruder (22) in a feed section (L/D < 9) to less than 150°C and then from said feed section to discharge between 120 and 240°C".

The board raised no objection under Article 84, 123(2) or 54 EPC against Claim 1 according to the main request. With respect to inventive step, the appellant relied on its written submissions.

(b) Following a discussion as to whether or not Claims 1 according to the 1st and 2nd auxiliary requests met the requirements of Articles 84 and

123(2) EPC, the appellant withdrew the 1st and 2nd auxiliary requests.

(c) Claim 1 according to the 3rd auxiliary request read as follows:

"A process of preparing a liquid silicone rubber composition, comprising:

introducing a filler, treating agent (16) and silicone polymer into an extruder (22) having a length to diameter ratio of at least greater than 50, wherein the silicon polymer is a vinyl-terminated polydimethylsiloxane having a viscosity varying from 100 to 2,000,000 mPas (100 to 2,000,000 cps) at 25°C and wherein the treating agent comprises silanol-stopped polydimethylsiloxane, octamethylcyclotetrasiloxane (D4) or hexamethyldisilazane (HMDZ) and wherein said filler contains silanol groups;

continuously compounding and devolatilizing said filler, treating agent (16) and silicone polymer into said liquid silicone rubber composition in said extruder (22);

further comprising discharging said liquid rubber composition from said extruder (22) to a cooler, wherein said liquid rubber composition is cooled, homogenized and further devolatilized, wherein said liquid silicone rubber is in residence in said cooler for a period greater than residence in said extruder (22) ".

The board raised no objection under Article 84, 123(2) or 54 EPC against Claim 1 according to the 3rd auxiliary request.

With respect to inventive step, the appellant argued that the separate cooling step allowed more flexibility in the process. Not only the residence time but also further process conditions in the cooler might be chosen according to the particular requirements of the product and irrespective of the operating conditions of the extruder. Furthermore, neither D1 nor D2 suggested a separate cooler.

VII. The appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of

main request:
Claim 1 as filed during the oral proceedings (headed "Claim 1 according to the main request");
Claims 2-10 and 12 as originally filed,

or, in the alternative, on the basis of

3rd auxiliary request:
Claim 1 as filed during the oral proceedings (headed "Claim 1 according to the 3rd auxiliary request");
Claims 2, 3, 5, 8-10 and 12-15 as originally filed.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 EPC and Rule 64 EPC and is therefore admissible.

2. *Main request*

2.1 Amendments

Claim 1 of the main request is a combination of originally filed Claims 1, 11 (definition of filler and treating agent), 13 (temperature profile), 15 (L/D ratio) and the passage on page 5, lines 1-3 of the application as originally filed (definition of silicon polymer). Thus, no objection under Article 123(2) EPC arises.

Since, furthermore, the amended claim now clearly defines the three different components to be compounded in the extruder (22), no objection under Article 84 EPC arises.

2.2 The prior art

2.2.1 Document D1 describes a method for producing a filled liquid silicone rubber (LSR) base without plasticizer by passing all of the filler together with from 30 to 100 percent of the total weight of the polyorganosiloxane ingredient of the base through the first kneading section of a compounding extruder and adding any remaining polyorganosiloxane before passing the composition through a second kneading section of the compounding extruder, whereby the kneading sections are maintained at from 200-300°C (Claim 1). The

polyorganosiloxane used must contain at least two silicon-bonded alkenyl groups in each molecule. Examples of alkenyl groups are vinyl, allyl and 1-propenyl (column 2, lines 11-15). There is no general disclosure with respect to the L/D ratio of the extruder.

In Examples 1-5, wet-process silica and vinyl-terminated polydimethylsiloxane exhibiting a viscosity of 4 Pas were compounded and devolatilized in a twin-screw compounding extruder equipped with co-rotating triple flight screws measuring 3 cm in diameter and 129 cm in length which results in an L/D ratio of 43. The first 24 cm of the upstream end of the extruder was cooled to maintain the temperature at 70°C or below. The sections located from 24 to 39 cm and from 114 to 129 cm from the upstream end were heated to a temperature of 150°C. The remaining sections of the barrel were heated to a temperature specified as "kneading temperature", namely to 280°C (Examples 1, 3 and 4), 250°C (Example 2) and 200°C (Example 5).

2.2.2 Document D2 discloses a process for continuously producing a homogeneous silicone rubber compound, which includes the steps of (i) mixing a diorganopolysiloxane, an inorganic filler and a processing aid, as basic ingredients, by means of a high-speed mechanical shearing machine to obtain a flowable particulate mixture, and (ii) introducing the flowable particulate mixture into a twin-screw kneading and extruding machine, wherein that flowable particulate mixture is kneaded in the continuously kneading and extruding machine while the mixture is heated at 100 to 300°C,

and wherein cooling is conducted at the rear stage of the kneading step (Claim 1).

The diorganosiloxane which can be used in the process of D2 has a viscosity as measured at 25°C of $1x10^5$ mPas or higher (column 3, lines 41-44), and is a substantially linear polymer represented by the formula $R^2(R^1_2SiO)_nSiR^1_2R^2$ wherein R^1 represents a substituted or unsubstituted monovalent hydrocarbon group, provided that 0 to 1.0% of the hydrocarbon groups of R^1 are vinyl, and R^2 is a monovalent group selected from the group consisting of a methyl group a vinyl group, a phenyl group and a hydroxyl group, and n is a number of from 1,000 to 10,000 (column 3, lines 55-62). It is preferred that 50% or more of all the hydrocarbon groups of R^1 are methyl, from the standpoint of the heat resistance and other properties of silicone rubbers to be produced. Further, in the case where all the hydrocarbon groups of R^1 are not vinyl, R^2 should be vinyl (column 4, lines 4-8).

The inorganic filler which can be used in the process of D2 can be any of the inorganic fillers for use in blends with silicone rubbers. An example of such a filler is reinforcing silica such as fumed silica or precipitated silica having a specific surface area or a surface-treated silica which has been treated with an organosilicon compound such as an organopolysiloxane, an organoalkoxysilane, an organochlorosilane or a hexaorganodisilazane (column 4, lines 33-42).

The processing aid is used for the purpose of improving the dispersibility of the inorganic filler during the kneading step, reducing the period of time required for

the aging of a silicone rubber compound to be obtained, preventing crepe hardening, and regulating the plasticity of the compound, and for other purposes. The processing aid (C) is selected from organosilanes, low-viscosity organopolysiloxanes and silicone resins, which have in their molecules a silanol group and/or an alkoxy group having 1 to 6 carbon atoms (column 4, line 67 to column 5, line 9).

In Example 1, trimethylsilyl-terminated methylvinylpolysiloxane (a methylvinylpolysiloxane), fumed silica and two processing aids (α,ω-dimethoxypolydimethylsiloxane and α,ω-dimethoxypolymethylphenylsiloxane) were mixed in a Henschel mixer. This particulate mixture was fed at a constant feed rate to a same-direction twin-screw extruder having a screw diameter of 50 mm and a screw shaft length of 2,400 mm (L/D of the screw = 48). The heating and cooling system in the extruder was such that the L/D range of from 0 to 10 was able to be cooled with water, the L/D range of from 10 to 38 was able to be heated up to 300°C with an electric heater, and the L/D range of from 38 to 48 was able to be rapidly cooled with a refrigerant carrier of -10°C. During the operation of the extruder, the barrel was heated such that the temperature of the compound in the part of L/D = 36 to L/D = 38 was maintained at 270°C, thereby removing low-boiling components and degassing under reduced pressure through the second vent hole, and the compound in the part of L/D = 38 to L/D = 48 was cooled with a refrigerant carrier of -10°C. The temperature of the silicone rubber compound when it was discharged from the extruder was about 90°C. The thus-

obtained heat-vulcanizable silicone rubber compound was masticated with rolls.

2.3 Novelty

As can be seen from the above analysis of the prior art, both D1 and D2 are concerned with the preparation of an LSR composition in an extruder. However, neither D1 nor D2 discloses a process having all the features of Claim 1 of the main request: D1 does not disclose the presence of a treating agent or an L/D ratio of at least greater than 70 for the extruder. D2 does not disclose an L/D ratio of at least greater than 70 for the extruder, the now required combination of specified filler, filler treating agent and silicone polymer and the specified temperature profile. The subject-matter of the main request is therefore novel with respect to these documents.

2.4 Inventive step

2.4.1 Claim 1 of the main request is directed to a continuous process that consistently produces a devolatilized LSR composition in an extruder from three different components, namely filler, treating agent and silicone polymer. As shown in point 2.2.2, above, D2 likewise relates to a method for preparing an LSR composition in an extruder from three different components, namely filler, processing aid and silicone polymer. Although D2 refers to a processing aid, it is conspicuous to the board that the terms "processing aid" (used in D2) and "treating agents" (used in the present application) are merely different names for the same type of compounds. Thus, according to D2 (column 5, lines 5 to 9), a

processing aid is selected from organosilanes, low-viscosity organosiloxanes and silicone resins which have in their molecules a silanol group and/or an alkoxy group having 1 to 6 carbon atoms. An almost identical definition for treating agent is found in the application as originally filed on page 6, lines 18-21: "The treating agent can be an organosilane, organosilazane, a low-viscosity organosiloxane or a silicone resin, which has silanol group and/or an alkoxy group having 1 to 6 carbon atoms." This means that every processing aid as defined in D2 is also a treating agent within the meaning of the present application.

Thus, D2 is not only in the same field as the present application, it uses also the same three types of components, and is therefore considered to represent the closest prior art.

2.4.2 The subject-matter of Claim 1 of the main request differs from D2 in the following aspects:

(i) specific L/D ratio of greater than 70,
(ii) specific filler, treating agent and silicone polymer, and
(iii) a specific temperature profile.

It is not apparent from the application as originally filed that the claimed process achieves any technical effect over the process disclosed in D2. Hence, the objective technical problem can only be seen in the provision of a further process for providing a LSR composition.

2.4.3 In the board's view, the solution to this problem is obvious because the subject-matter of Claim 1 of the main request amounts to nothing else but an arbitrary selection from the general disclosure of D2 for the following reasons.

Although an L/D ratio of 25-50 is preferred in D2 (column 7, lines 51-52) and in Example 1 of D2 an extruder having an L/D ratio of 48 is used, it is also stated in column 7, lines 47-50 of D2 that the screws of the extruder are not particularly limited in L/D ratio. As regards the specific treating agent now required in Claim 1 of the main request, it is conspicuous to the board that one of these, namely the silanol-stopped polydimethylsiloxane, is still covered by the general disclosure of D2 (a low-viscosity organosiloxane or a silicone resin, which has silanol group and/or an alkoxy group having 1 to 6 carbon atoms). The same applies to the vinyl-terminated polydimethylsiloxane silicone polymer having viscosity varying from 100 to 2,000,000 mPas (D2: a diorganopolysiloxane having a viscosity as measured at 25°C of $1x10^5$ mPas, preferably containing vinyl groups) and the filler containing silanol groups (D2: surface treated silica which has been treated with an organosilicon compound). Finally, the temperature profile required in Claim 1 of the main request is completely within the range indicated in Claim 1 of D2 (100-300°C). Apart from that, the now required temperature profile appears to be common in the prior art as can be seen from Example 5 of D1 (point 2.2.1, above).

Consequently, the board can only come to the conclusion that the restrictions carried out in Claim 1 of the main request with respect to the L/D ratio, the components and the temperature profile amount to an arbitrary selection from the general disclosure of D2 lacking an inventive step (Article 56 EPC), in particular because the appellant has not demonstrated any particular effect associated with the further specified features, and, furthermore, the application as originally filed has put no particular emphasis on these specified features.

2.5 In view of the above, the main request must be refused.

3. It may be convenient to recall at this conjuncture that the 1st and the 2nd auxiliary requests were withdrawn during the oral proceedings (see point VI(b), above).

4. *3rd auxiliary request*

4.1 Amendments

Claim 1 of 3rd auxiliary request is a combination of originally filed Claims 1, 4 (discharging said liquid rubber composition to a cooler), 6 (cooled, homogenized and further devolatilized in said cooler), 7 (residence in said cooler) and 11 (definition of filler and treating agent) and the passage on page 5, lines 1-3 of the application as originally filed (definition of silicon polymer). Thus, no objection under Article 123(2) EPC arises.

Also no objection under Article 84 EPC arises.

4.2 Novelty

Neither D1 nor D2 discloses a process for producing a liquid silicone rubber composition comprising a separate cooler. The subject-matter of the 3rd auxiliary request is therefore novel with respect to these documents.

4.3 Inventive step

4.3.1 D2 remains the closest prior art for the subject-matter of the 3rd auxiliary request, in particular because D2 also discloses cooling of the LSR composition towards the rear part of the extruder (see point 2.2.2, above).

4.3.2 The process of Claim 1 of the 3rd auxiliary request differs from the disclosure of D2 in the following aspects:

(i) a specific L/D ratio of greater than 50,
(ii) specific filler, treating agent and silicone polymer; and
(iii) separate cooler with a specific residence time.

The appellant argued that the separate cooling step allowed more flexibility in the process. Not only the residence time but also further process conditions in the cooler might be chosen according to the particular requirements of the product and irrespective of the operating conditions of the extruder. It is not apparent from the application as originally filed that the claimed process achieves any other technical effect apart from those referred to by the appellant. Hence, the objective technical problem can be seen in the

provision of a process for providing a LSR composition which allows more flexibility with respect to the process conditions.

4.3.3 In D2, the cooling is done at rear part of the extruder. During the cooling in the extruder of D2, the LSR composition is automatically further homogenized and devolatilized. Faced with the problem of providing more flexibility to the process of D2, the most obvious thing for the skilled person to do would have been to carry out cooling (and the associated homogenization and further devolatilization) in a separate apparatus which allows to elect the process conditions independently of those of the extruder. This step is a straightforward possibility the person skilled in the art would select without the exercise of inventive skill in order to solve the posed problem. The gain in flexibility, relied upon by the appellant as an indication for inventive step, is foreseeable when a process uses two apparatuses instead of one apparatus for the same overall process scheme.

As regards the specified residence time, it has not been shown how this feature would contribute to inventive step. In Examples 1 and 2 of the application as originally filed, which are the only examples, don't even indicate the residence time in the cooler. Therefore, this feature cannot be taken into account to justify the presence of an inventive step.

4.3.4 As regards the differences (i) and (ii), the same arguments as for the main request apply (see point 2.4.3, above).

In the context of (i) it might be worth mentioning that the L/D ratio in Claim 1 of the 3rd auxiliary request has to be only greater than 50 whereas Claim 1 of the main request requires an L/D ratio of at least 70. The value of 50 is just above the preferred L/D ratio disclosed in D2, namely 25-50 (column 7, lines 51-53).

4.3.5 The appellant's argument that none of the documents suggests a separate cooler is not convincing. Firstly, the separating of the cooling step is a straightforward possibility the skilled person would consider anyway, and secondly, D2 itself provides a hint to a separate process step subsequent to the extruder. In Example 1 of D2, the LSR composition obtained from the extruder is masticated with rolls (point 2.2.2, above). Such a process implies further homogenization and devolatilizing. It appears obvious to combine this extra process step with a cooling function if more flexibility of the overall process is sought.

4.3.6 In summary, the process as claimed in Claim 1 of the 3rd auxiliary request is not based on an inventive step (Article 56 EPC).

4.4 Consequently, the 3rd auxiliary request must be refused.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

E. Görgmaier R. Young