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(54) Title: FOAM STRUCTURE WITH AN INORGANIC BLOWING AGENT

(57) Abstract: A melt polymer material for afoam structure, e.g. insulation board, comprises 80% to 98% by weight of a styrenic polymer, preferably polystyrene; 1% to 10% by weight of a low molecular weight random styrene butadiene copolymer resin; and 1% to 10% by weight of an inorganic blowing agent, preferably carbon dioxide. The low molecular weight random styrene butadiene copolymer resin comprises 80% to 95% by weight styrene and 5% to 20% by weight butadiene; has a weight average molecular weight of 100,000 to 140,000; and a molecular weight distribution (MWD) of 2.0 to 8.0. A process for making the foam structure involves heating the styrenic polymer and mixing the random styrene butadiene copolymer resin and the blowing agent into the styrenic polymer, and then molding the resultant mixture. The foam structure generally is closed-cell; has an average cell size of 300 microns (0.3 millimeters); a density between 20 g/l and 45 g/ml; and if insulation board, measures 1 meter wide up to 100 millimeters thick.

FOAM STRUCTURE WITH AN INORGANIC BLOWING AGENT

The present invention generally relates to foam structures, and more particularly, to foam structures made from a melt polymer material comprised of a styrenic polymer, a random styrene butadiene copolymer resin, and an inorganic blowing agent, and to a process for forming the same.

Extruded synthetic resinous foams, such as extruded polystyrene foams, are useful materials for many applications including thermal insulation, decorative purposes, packaging articles, such as food products, and as shock absorbing material for shipping purposes, and the like. Rigid foams for insulation and food packaging are made by the extrusion of molten polystyrene to which a blowing agent has been added. Typical blowing agents include hydrocarbons, halofluorocarbons, and mixtures of these blowing agents with carbon dioxide. Foam structures can also be made using alternative methods, such as injection molding.

Thermal insulation is a particularly important application for styrene polymer foams. In this application, it is desirable to maintain the insulating value of the foam for as long as possible. It is also desirable for the foam to have dimensional stability. These desirable characteristics can be achieved, in part, by providing foams having uniform cell size.

Due to present environmental concerns over the use of potentially ozone-depleting or flammable blowing agents, such as hydrocarbons and halofluorocarbons, it is more desirable to make styrenic polymer foam structures with an inorganic blowing agent, such as carbon dioxide, nitrogen, argon, air and helium. However, it has been recognized that there are a number of problems associated with the use of these types of blowing agents in extruded foam structures. These problems include low solubility of the inorganic blowing agents and a higher diffusivity in styrene polymers, low quality foam production, dimensional instability, non-uniform cell size, etc.

Prior art patents that have attempted to overcome the disadvantages of using inorganic blowing agents for extruded polystyrene foam structures include U.S. Patent Nos. 5,286,429; 5,356,944; 5,688,832; 6,268,046, and 6,572,800.

The aforesaid U.S. Patent No. 5,286,429 issued to James E. Blythe, et al on February 15, 1994 and assigned to Mobil Oil Corporation and U. S. Patent No. 5,356,944 issued on

October 18, 1994 to James E. Blythe, et al and assigned to Mobil Oil Corporation use high-melt index polystyrene where the melt index is about 8 to 25. Polystyrene is combined in molten form under pressure with a 100% carbon dioxide blowing agent and the mixture is extruded through a die into an atmosphere of reduced pressure to form the foam structure. The foam structure has a basis weight of less than 20 gms/100 sq. inch and a density of about 6 lbs/cubic feet or less. The resins may be styrene homopolymers or copolymers containing a predominant portion of styrene, i.e. greater than about 50% wt styrene. For a styrene copolymer, the comonomer may be any other ethylenically unsaturated material such as the conjugated 1,3-dienes, e.g., butadiene, isoprene, etc.

The aforesaid U.S. Patent No. 5,688,832 issued to Andrew N. Paquet, et al on November 18, 1997 and assigned to The Dow Chemical Company is addressed to the problem associated with low solubility in melts of an alkenyl aromatic polymer when carbon dioxide is used as the blowing agent. It is stated that low solubility results in high system pressure, which results in high pressure drop and high die pressure in extrusion apparatuses. It is recognized that lowering pressure drop and die pressure would save energy costs and reduce the pressure duty requirements for process equipment. This invention reduces the operating pressures in making foam structures by using low molecular weight polystyrene, i.e. weight average molecular weight between 100,000 and 165,000, and carbon dioxide as the blowing agent. The foam structure has a density of 10 to 150 kilograms per cubic meter (kg/m^3) according to ASTM D-166; an average cell size of 0.05 to 5.0 millimeters according to ASTM D3576-77; and preferably is greater than 90 percent closed-cell according to ASTM D2856-A.

The aforesaid U.S. Patent No. 6,268,046 B1 issued to Larry M. Miller, et al on July 31, 2001 and assigned to Owens Corning Fiberglas Technology Inc. discloses foamable mixtures containing two different styrenic polymers and carbon dioxide as a blowing agent. The foamable mixture comprises a minor amount of styrenic polymer having a high melt index of 10 to 35, a major amount of styrenic polymer having a low melt index; and a blowing agent having a major amount of carbon dioxide. It is disclosed in Column 7, lines 18 through 38 of the '046 patent that elastomeric rubbers may be added to the foamable mixture to facilitate processing of the foamable mixture in the extruder and to enhance relaxation of the resultant foam bodies. The elastomers may be copolymers of styrene and a diene, such as butadiene or isoprene. The copolymers typically are block copolymers such as diblock, triblock or radial block copolymers.

The aforesaid U.S. Patent No. 6,572,800, issued to Jesus Ladera Sainz, et al on June 3, 2003 and assigned to Poliglas, S.A. discloses polystyrene foam blocks and boards that have dimensional stability and good self-extinction features when carbon dioxide is used by injecting control agents into the melt. A first control agent consists of ethanol that dissolves and cools the melt, and the second control agent consists of H_2O_2 that cools the melt. The amount of ethanol is sufficient to achieve the highest possible dissolution of CO_2 in the melt and the injected amount of H_2O_2 is such that the foaming produced by ethanol is minimized.

From a practical standpoint, it is important that the styrenic polymer foam emerging from a foam extrusion line attains a stable physical form as quickly as possible. A stable physical form is achieved when the dimensional and/or mechanical properties of the extruded structure do not change appreciably with time. Factors that influence the rate of achieving such stability include: 1) the rate at which the blowing agent gas diffuses from the nascent foam at the extruder die head, and 2) the solubility of the blowing agent in the polymer. A further disadvantage of using an inorganic blowing agent in extruded styrenic polymer foam structures is that the foam structures require a long storage time in order for the foam structures to reach mechanical stability, which adds to the overall production costs.

Therefore, it would be desirable to reduce the storage time for attaining at least the required mechanical stability of extruded styrenic polymer foam structures. This also applies to foam structures made by other methods, such as injection molding.

The present invention relates to styrenic polymer foams, which may be referred to as "extruded foams" if made by extrusion. The invention involves the addition of a low molecular weight random styrene butadiene copolymer resin (SBC) to a styrenic polymer, e.g. polystyrene of the type conventionally used in making polystyrene foam, e.g. extruded polystyrene foam, particularly insulation board, where in the invention the time for the foam board to achieve at least the required mechanical stability is substantially reduced.

In an embodiment of the present invention, a melt polymer material for a foam structure comprises from about 80% to about 98% by weight of a styrenic polymer; from about 1% to about 10% by weight of a low molecular weight random styrene butadiene copolymer

resin; and from about 1% to about 10% by weight of an inorganic blowing agent. Preferably the inorganic blowing agent is carbon dioxide.

The foam structure is preferably an extruded foam structure, but may be, for example, an injection molded structure.

In another embodiment, the present invention relates to a process for making a styrenic polymer foam structure, comprising: a) heating a styrenic polymer to form a melt polymer material; b) incorporating into the melt polymer material, a low molecular weight random styrene butadiene copolymer resin in an amount ranging from about 1% to about 10% by weight based on the total weight of the melt polymer material, and an inorganic blowing agent in an amount ranging from about 1% to about 10% by weight based on the total weight of the melt polymer material material; and c) subjecting the resultant mixture of b) to a molding process to form a foam structure.

If the foam structure is an extruded foam structure, then in step c) the resultant mixture of b) is extruded through a die to form the foam structure. If the foam structure is an injection molded structure, then in step c) the resultant mixture of b) is injection molded to form the foam structure,

Preferably, the random styrene butadiene copolymer resin has a low weight average molecular weight ranging from about 100,000 to about 140,000; a molecular weight distribution (MWD) ranging from about 2.0 to about 8.0; and a ratio of styrene monomer to butadiene monomer ranging between about 80:20 and about 95:5 by weight. Preferably, the weight percent of styrene is 86% and that of butadiene is 14% based on the weight of the copolymer resin.

The random styrene butadiene copolymer resin and the blowing agent generally are mixed or blended into the styrenic polymer in a heat plastifying and mixing apparatus, such as an extruder.

It is, therefore, an object of the present invention to provide a melt polymer material for a styrenic foam structure, e.g. an extruded styrenic foam structure, that uses an inorganic blowing agent whereby at least mechanical stability can be achieved faster than that of conventional extruded foam structures, thereby reducing storage time and production

costs while still maintaining the required mechanical properties and/or thermal properties for a foam structure.

These and other objects of the present invention will be better appreciated and understood by those skilled in the art from the following description and appended claims.

Detailed Description

A melt polymer material is extruded and foamed into foam products, such as foam board, foam sheet and other foam structures, or can be subject to other processes, such as injection molding, to produce foam structures. The melt polymer material is comprised of a styrenic polymer, a low molecular weight random styrene butadiene copolymer resin, and an inorganic blowing agent.

A suitable styrenic polymer includes styrene homopolymers or copolymers containing a predominant portion of styrene, i.e. greater than about 50% by weight styrene. For a styrene copolymer, the comonomer can be any other ethylenically unsaturated material such as the conjugated 1,3-dienes, e.g. butadiene, isoprene, etc. The styrenic polymer as used in the invention may also include polymers of alkyl and halogen substituted styrene such as alpha-methylstyrene, para-isobutylstyrene, para-chlorostyrene, and the like as well as copolymers of styrene and vinyl substituted monomers such as maleic anhydride, etc.

A preferred styrenic polymer has styrenic monomer units greater than 50% by weight, and preferably, about 90% to about 100% by weight. Preferred styrenic polymers are polystyrene, styrene-acrylonitrile, styrene methylmethacrylate, and styrene-maleic anhydride.

If the styrenic polymer is polystyrene, preferably, it is crystal polystyrene with a melt flow index of about 5 g/10 minutes. The styrenic polymer has a weight average molecular weight ranging from about 200,000 to about 500,000.

Suitable styrenic polymers are available commercially from a variety of sources and are available with different properties such as melt flow index, molecular weight, and so on. Such polystyrenes are available from NOVA Chemicals Inc. (U.S., Canada, and Europe).

The melt polymer material of the invention may include flame-retardant chemicals, stabilizers, pigments, extrusion aids, antioxidants, fillers, antistatic agents, UV absorbers, etc. As is known to those skilled in the art, these additives may be included in any amount to obtain the desired characteristics in the melt polymer material or in the resultant foamed bodies, and may be added to the styrenic polymer before, during, or after polymerization of the styrenic polymer, or may be added to the styrenic polymer in an extrusion, or other, process.

The random styrene butadiene copolymer resin has a low molecular weight ranging from about 100,000 to about 140,000 weight average molecular weight; a molecular weight distribution (MWD) ranging from about 2.0 to about 8.0; and a ratio of styrene monomer and butadiene monomer ranging between about 80:20 and about 95:5 by weight.

Preferably, the weight percent of styrene in the random styrene butadiene copolymer resin is about 86% and the weight percent of butadiene is about 14% based on the weight of the copolymer. Preferably, the weight average molecular weight of the random styrene butadiene copolymer is about 120,000, and the molecular weight distribution (MWD) is about 4, and more preferably, about 3. The MWD is obtained by dividing the weight average molecular weight by the number average molecular weight. Thus if the weight average molecular weight is 120,000 and the MWD is 4, the number average molecular weight is 30,000.

A suitable random styrene butadiene copolymer resin is one that is made through a suspension polymerization process via a free radical polymerization initiator. Such random styrene butadiene copolymer resin is that produced by the process taught in the aforesaid U.S. Patent No. 4,558,108, which is incorporated herein by reference in its entirety. This random styrene butadiene copolymer resin may be obtained from NOVA Chemicals (International) S.A., Fribourg, Switzerland under the trade name XP-808. As used herein, a "random styrene butadiene copolymer resin" is defined as a low molecular weight copolymer of styrene and butadiene monomers in which the monomers are incorporated randomly into the copolymer chains.

In the melt polymer material of the invention, the amount of random styrene butadiene copolymer resin (SBC) ranges from about 1% by weight to about 10% by weight, and preferably from about 2% to about 9% by weight, based on the weight of the melt polymer

material. The amount of styrenic polymer ranges from about 80% to about 98% by weight, and preferably from about 82% by weight to about 96% by weight based on the weight of the melt polymer material. The amount of inorganic blowing agent ranges from about 1% to about 10% by weight, and preferably from about 2% to about 9% by weight based on the weight of the melt polymer material.

The inorganic blowing agent is selected from the group consisting of carbon dioxide, nitrogen, air, water, helium, and argon, and/or blends thereof. A preferred inorganic blowing agent is carbon dioxide. If a blend of inorganic blowing agents is used, then preferably, the amount of carbon dioxide will be greater than about 50% by weight, and preferably 90% by weight, based on the total weight of the blowing agent.

The blowing agent may be a mixture of carbon dioxide and at least one lower alcohol. A lower alcohol is an alkyl alcohol containing from 1 to about 4 carbon atoms. Lower alcohols include methanol, ethanol, propanol, isopropanol and butanol. The carbon dioxide and blowing agent mixtures may also be used with additional, optional and supplemental blowing agents, such as air, nitrogen, helium, argon, and water. The amount of alkyl alcohol will range from about 0.1% to about 0.5% by weight based on the weight of the melt polymer material.

In the invention, the alkyl alcohol and the blowing agent may be combined as a mixture or may be added separately to the melt polymer material. Preferably, these additives are added in an extrusion, or other, process for forming the foam structure. Also, the random styrene butadiene copolymer resin preferably is added to the styrenic polymer during the extrusion, or other, process.

A general procedure for forming a foam structure in an extrusion process involves the following steps: The resin is heat plastified and the blowing agent is incorporated and thoroughly mixed into the plastified resin under conditions that permit thorough mixing of the blowing agent into the plastified resin and that prevent foaming of the mixture. The mixture of resin, blowing agent, and optional additives is cooled, and the pressure on the mixture is reduced resulting in foaming of the mixture and formation of the desired foam body. That is, foam bodies are obtained by extruding the cooled plastified mixture of resin, blowing agent and optional additives into a region of lower pressure.

In the invention, a similar procedure is followed in a tandem extruder foam line. The foam line consists of a first extruder in which the low molecular weight random styrene butadiene copolymer resin is blended with the styrenic polymer. This polymer mixture is then melted at a temperature ranging between about 200°C and about 220°C, preferably 210°C, and held at this temperature to form the melt polymer material. The inorganic blowing agent, and optionally the alkyl alcohol, is injected into the barrel of the extruder and is dissolved in the molten polymer. The gassed molten polymer is passed continuously to a second extruder where the polymer material is cooled at a temperature ranging from about 110°C to about 130°C, preferably 120°C, and then is passed to a slit die from which the polymer emerges to form a foam structure. The die width is about 0.8 meters and the die gap is about 6 millimeters. The resultant foam generally will have a density ranging from about 20 grams per litter (g/l) to about 45 grams per liter (g/l) and a thickness up to about 100 millimeters (mm), and preferably around 80 millimeters. This resultant foam generally is suitable for insulation purposes. The type of tandem extruder may be any of the several extruders known to those skilled in the art and commercially available.

It has been found by the inventors that the low molecular weight random styrene butadiene copolymer resin, as taught herein, added to a styrenic polymer, especially polystyrene and when an inorganic blowing agent, for example carbon dioxide, is used, the solubility of the inorganic blowing agent in the molten polymer is enhanced and the behavior of the nascent foam board at the extruder die head is stabilized.

The resultant foam structure generally is closed-cell; has an average cell size of about 300 microns (0.3 millimeters); and as stated herein above, has a density ranging from about 20 g/l to about 45 g/l; is about 1 meter wide, and has a thickness of up to about 100 millimeters thick, preferably around 80 millimeters thick.

Injection molding processes suitable for use in the invention are well known in the art.

The following example is intended to assist in understanding the present invention. However, in no way, should this example be interpreted as limiting the scope thereof.

Example

Control

Crystal polystyrene obtained from NOVA Chemicals, Breda, The Netherlands, under the designation "171N" with a melt flow index of 1.5 g/10 minutes was fed to a tandem extruder foam line similar to that discussed herein above. In the first extruder, the polystyrene was melted and held at a temperature of 210°C. Carbon dioxide at 4% by weight and ethanol at 0.4% by weight based on the weight of the polystyrene were injected into the barrel of the extruder and were dissolved in the molten polystyrene. This melt polymer material was passed continuously to the second extruder where it was cooled to 120°C. The cooled polymer material was then passed to the slit die where it was formed into a foam board. The resultant foam board was 80 millimeters thick and had a density of 45 g/l.

Periodic measurements of compressive and flexural strength conducted on the foam board showed that the board required about 60 days to reach mechanical stability. After 90 days, the thermal conductivity of the foam board was 33 mW/m/°C. The dimensional stability was measured according to ISO 2796; the flexural strength was measured in accordance with ISO 1290-2; and the compressive strength was measured according to ISO 7616.

Invention

In the extrusion system for the Control, 90 parts crystal polystyrene were blended with 10 parts low molecular weight random styrene butadiene copolymer resin. The low molecular weight random styrene butadiene copolymer (SBC) resin was the XP 808 product obtained from NOVA Chemicals (International) S.A., Fribourg, Switzerland, having a weight average molecular weight of about 120,000 and a MWD of about 4. The composition of the random styrene butadiene copolymer resin was 86% by weight styrene and 14% by weight butadiene. Using a load of 2.16 kilograms, the melt index of the melt polymer material was 28 g/10 minutes when measured at 120°C.

The extrusion conditions and the addition of the carbon dioxide and ethanol were the same as for the Control. Also, the resultant foam board had the same density and dimensions as that of the Control.

Periodic measurements of the compressive and flexural strengths of the foam board indicated that the mechanical stability was achieved in 40 days. The thermal conductivity of the foam board after 90 days was 34 mW/m/°C.

This Example illustrates that the random styrene butadiene copolymer resin in the styrenic polymer reduces the storage time necessary for the foam board to achieve mechanical stability without affecting the insulating properties of the foam board.

While the present invention has been particularly set forth in terms of specific embodiments thereof, it will be understood in view of the instant disclosure that numerous variations upon the invention are now enabled yet reside within the scope of the invention. Accordingly, the invention is to be broadly construed and limited only by the scope and spirit of the claims now appended hereto.

CLAIMS

1. A melt polymer material for a foam structure, comprising:
from about 80% to about 98% by weight of a styrenic polymer, based on the weight of the melt polymer material;
from about 1% to about 10% by weight of a low molecular weight random styrene butadiene copolymer resin, based on the weight of the melt polymer material; and
from about 1% to about 10% by weight of an inorganic blowing agent, based on the weight of the melt polymer material.

2. A melt polymer material of claim 1 wherein the foam structure is an extruded foam structure.

3. A melt polymer material of claim 2 wherein said low molecular weight random styrene butadiene copolymer resin is melt blended into said styrenic polymer in an extrusion process.

4. A melt polymer material of claim 1 wherein the foam structure is an injection molded foam structure.

5. . A melt polymer material of claim 4 wherein said low molecular weight random styrene butadiene copolymer resin is melt blended into said styrenic polymer in an injection molding process.

6. A melt polymer material of any one of claims 1 to 5 wherein said low molecular weight random styrene butadiene copolymer resin is prepared in a reactor under free radical suspension polymerization conditions.

7. A melt polymer material of any one of claims 1 to 6 wherein said low molecular weight random styrene butadiene copolymer resin is comprised of about 80% to about 95% by weight styrene and of about 20% to about 5% by weight butadiene.

8. A melt polymer material of claim 7 wherein said low molecular weight random styrene butadiene copolymer resin is comprised of about 86% by weight styrene and about 14% by weight butadiene.

9. A melt polymer material of any one of claims 1 to 8 wherein said low molecular weight random styrene butadiene copolymer resin has a weight average molecular weight ranging from about 100,000 to about 140,000, and a molecular weight distribution (MWD) ranging from about 2.0 to about 8.0.

10. A melt polymer material of any one of claims 1 to 9 wherein said styrenic polymer is selected from the group consisting of polystyrene, styrene-acrylonitrile, styrene methylmethacrylate, and styrene-maleic anhydride, and wherein said inorganic blowing agent is selected from the group consisting of carbon dioxide, nitrogen, air, water, helium, argon, and blends thereof.

11. A melt polymer material of claim 10 wherein said styrenic polymer is polystyrene and wherein said inorganic blowing agent is carbon dioxide.

12. A melt polymer material of any one of claims 1 to 11 wherein said foam structure is selected from the group consisting of foam board and foam sheet.

13. A melt polymer material of any one of claims 1 to 11 wherein said foam structure is insulation board.

14. A foam structure of a melt polymer material of any one of claims 1 to 11 which:
a) is greater than 95 per cent closed-cell;
b) has an average cell size of about 300 microns; and
c) has a density ranging from about 20 g/l to about 45 g/l.

15. A foam structure of claim 14 which is insulation board that is about 1 meter wide and up to about 100 millimeters thick.

16. A process for making a styrenic polymer foam structure, comprising:
a) heating a styrenic polymer resin to form a melt polymer material;
b) incorporating into the styrenic polymer a low molecular weight random styrene butadiene copolymer resin in an amount ranging from about 1% to about 10% by weight based on the total weight of the melt polymer material, and an inorganic blowing agent in

an amount ranging from about 1% to about 10% by weight based on the total weight of the melt polymer material; and

c) subjecting the resultant mixture of b) to a molding process to form said foam structure.

17. A process of claim 16 wherein in step c) the resultant mixture of b) is extruded through a die to form the foam structure.

18. A process of claim 16 wherein in step c) the resultant mixture of b) is injection molded to form the foam structure,

19. A process of any one of claims 16 to 18 wherein said styrenic polymer of a) is selected from the group consisting of polystyrene, styrene-acrylonitrile, styrene methylmethacrylate, and styrene-maleic anhydride, and wherein said inorganic blowing agent is selected from the group consisting of carbon dioxide, nitrogen, air, water, helium, argon and blends thereof.

20. A process of any one of claims 16 to 19 wherein said styrenic polymer is polystyrene and said inorganic blowing agent is carbon dioxide.

21. A process of claim 19 wherein said low molecular weight random styrene butadiene copolymer resin of b) is comprised of about 80% to about 95% by weight styrene and of about 20% to about 5% by weight butadiene.

22. A process of claim 21 wherein said low molecular weight random styrene butadiene copolymer resin of b) is comprised of about 86% by weight styrene and about 14% by weight butadiene.

23. A process of claim 22 wherein said low molecular weight random styrene butadiene copolymer resin has a weight average molecular weight ranging from about 100,000 to about 140,000, and a molecular weight distribution (MWD) ranging from about 2.0 to about 8.0.

24. A process of any one of claims 16 to 23 wherein the foam structure is greater than 95 per cent closed-cell.

25. A process of any one of claims 16 to 24 wherein the foam structure has an average cell size of about 300 microns.

26. A process of any one of claims 16 to 25 wherein the foam structure has a density ranging from about 20 g/l to about 45 g/l.

27. A process of any one of claims 16 to 26 wherein the foam structure is about 1 meter wide and up to about to about 100 millimeters thick.

28. A process for attaining mechanical stability of styrenic polymer foam structure faster than conventional foam structure by using the process of any one of claims 16 to 27.

INTERNATIONAL SEARCH REPORT

PCT/GB2005/001297

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 C08J9/00 C08J9/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 00/23511 A (OWENS CORNING; MILLER, LARRY, M; CISAR, THOMAS, E; BREINDEL, RAYMOND,) 27 April 2000 (2000-04-27) cited in the application page 1, lines 5-7 page 2, line 14 - page 5, line 27 page 7, line 10 - page 8, line 15 page 9, line 22 - page 10, line 2 page 11, line 19 - page 12, line 32; claims 13,19; example	1-28
X	DATABASE WPI Derwent Publications Ltd., London, GB; AN 1988-054214 XP002331473 & JP 63 011102 A (JAPAN SYNTHETIC RUBBER CO LTD) 18 January 1988 (1988-01-18) abstract	1-28

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[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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10 June 2005	21/06/2005

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Otegui Rebollo, J

Form PCT/ISA/210 (second sheet) (January 2004)

INTERNATIONAL SEARCH REPORT

PCT/GB2005/001297

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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