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(71) Applicant(s):

Nichias Corporation (Incorporated in Japan) 1-26 Shiba Daimon 1-Chome, Minato-ku, Tokyo 105-8555, Japan

(72) Inventor(s):

Atsushi Omura Kenichirou Ishikawa Hideharu Aoyagi

(74) Agent and/or Address for Service:

Gill Jennings & Every LLP The Broadgate Tower, 20 Primrose Street, LONDON, EC2A 2ES, United Kingdom

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EP 1342751 A1 US 4173556 A

(58) Field of Search: INT CL CO8L, F16J Other: WPI; EPODOC

- (54) Title of the Invention: Gasket material Abstract Title: A gasket material including a steel plate and a foamed rubber layer
- (57) The present invention relates to a gasket material including a steel plate and a foamed rubber layer formed on one or both sides of the steel plate. The foamed rubber layer is obtained from a rubber composition containing :NBR, H-NBR or functional group-modified NBR, as a rubber compound; and a crosslinking agent, the crosslinking agent comprising at least one selected from the group consisting of p-quinone dioxime, p, p' -dibenzoylguinone dioxime and poly-p-dinitrobenzene. The foamed rubber layer has a quinoid crosslinking structure.

GASKET MATERIAL

5 TECHNICAL FIELD

[0001]

The present invention relates to a gasket material suitable for electric components and the like.

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BACKGROUND ART

[0002]

There have hitherto been used gasket materials having a steel plate and a foamed rubber layer comprising NBR (nitrile rubber), H-NBR (hydrogenated nitrile rubber) or functional group-modified NBR, which is formed on one or both sides of the steel plate. Such gasket materials have many air bubbles in the foamed layer, so that they have a 20 high compressibility compared with gasket materials in which a non-foamed rubber layer is formed. Accordingly, such gasket materials are excellent in sealing properties for flanges having a high surface roughness or in sealing under low surface pressure, and therefore become the mainstream at present.

[0003]

Further, NBR, H-NBR or functional group-modified NBR generally crosslinked with sulphur or However, it is difficult to perform a peroxide crosslinking in the atmosphere, unlike a sulphur that the peroxide crosslinking crosslinking, SO unsuitable for crosslinking in an oven, which is usually performed. The sulfur crosslinking is therefore generally performed also in the gasket materials (for example, see Patent Document 1). However, sulfur is liberated from the foamed rubber layer in some cases. In particular, when the gasket materials are used in electric components, there is a possibility of corroding metals which form the components or metal parts in the peripheries thereof.

[0004]

Furthermore, in recent years, there are being popularized

10 hybrid cars in which an engine and an electric motor are combined
as power sources and electric cars driven by only an electric
motor. However, when the gasket material with the
sulfur-crosslinked foamed rubber layer formed is used in the
electric components such as an alternator, it is presumed that

15 liberated sulfur corrodes the metal components of the electric
motor.

[0005]

Patent Document 1: JP-A-2003-268163

20 SUMMARY OF THE INVENTION

[0006]

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The invention has been made in view of such a situation, and an object thereof is to provide a gasket material capable of good sealing without the possibility of causing metal corrosion even when used in electric components, with respect

to a gasket material in which a foamed rubber layer composed of NBR, H-NBR or functional group-modified NBR is formed.

[0007]

In order to achieve the above object, the present invention provides the following gasket materials.

(1) A gasket material comprising a steel plate and a foamed rubber layer formed on one or both sides of the steel plate,

wherein the foamed rubber layer is obtained from a rubber composition containing: at least one of NBR, H-NBR or functional group-modified NBR, as a rubber compound; and a crosslinking agent, and

wherein the foamed rubber layer has a quinoid crosslinking structure.

- 15 (2) The gasket material according to the above (1), wherein the crosslinking agent comprises at least one selected from the group consisting of p-quinone dioxime, p,p'-dibenzoyl-quinone dioxime and poly-p-dinitrobenzene.
- (3) The gasket material according to the above (1) or 20 (2), wherein the rubber composition further contains at least one selected from the group consisting of lead oxide, copper oxide, manganese oxide, magnesium oxide, N,N'-m-phenylenedimaleimide and graphite, as a crosslinking accelerator.
- 25 (4) The gasket material according to any one of the

- above (1) to (3), wherein the rubber composition further contains at least one selected from the group consisting of: a DPT-based (dinitro(so)pentamethylenetetramine-based), ADCA-based (azodicarbonamide-based) and HDCA-based (hydrazodicarbonamide-based) thermal decomposition type foaming agent; and non-crosslinkable and thermally expansible microcapsules, as a foaming agent.
- (5) The gasket material according to any one of the above (1) to (4), wherein the foamed rubber layer has a thickness before foaming of 10 to 200 μ m and an expansion ratio of 1.5 to 5.0.
- (6) The gasket material according to any one of the above (1) to (5), which is a gasket material for an electric component.

15 [0008]

According to the invention, in a gasket material in which a foamed rubber layer comprising at least one of NBR, H-NBR or functional group-modified NBR is formed, liberation of sulphur from the gasket material can be eliminated by obtaining the foamed rubber layer by subjecting to quinoid crosslinking. Therefore, metal corrosion can be prevented even when used in electric components and good sealing properties can be maintained.

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MODE FOR CARRYING OUT THE INVENTION

[0009]

The invention will be described in detail below.

[0010]

The gasket material of the invention comprising a steel plate and a foamed rubber layer, in which the foamed rubber layer is obtained from a rubber composition containing at least on of NBR, H-NBR or functional group-modified NBR, is formed on one or both sides of the steel plate, and the foamed rubber layer has a quinoid crosslinking structure in place of a sulfur crosslinking structure.

[0011]

The quinoid crosslinking can be performed to NBR, H-NBR and every kind of functional group-modified NBR (these rubbers are hereinafter generically named "NBR"). As crosslinking agents used is preferably at least one crosslinking agent selected from the group consisting of p-quinone dioxime,

15 p,p'-dibenzoylquinone dioxime and poly-p-dinitrobenzene. The added amount of these crosslinking agents is preferably from 0.5 to 50.0 parts by mass, and more preferably from 2.0 to 15.0 parts by mass, based on 100 parts by mass of NBR. Further, these quinoid-based crosslinking agents may be used either alone or in combination of two or more thereof.

[0012]

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Further, in order to efficiently perform the crosslinking, a crosslinking accelerator is preferably used together. For example, at least one selected from the group consisting of lead oxide, copper oxide, manganese oxide, magnesium oxide,

N, N'-m-phenylenedimaleimide or graphite is used together. the added amount of these crosslinking accelerators, the lower limit thereof is an amount for achieving an effect of accelerating the crosslinking, and the upper limit thereof is an amount not causing excessive crosslinking. Specifically, in the case of lead oxide, copper oxide or manganese oxide, the added amount thereof is preferably from 0.1 to 20 parts by mass, and more preferably from 0.5 to 5 parts by mass, based on 100 parts by mass of NBR. In the case of magnesium oxide, it is preferably from 0.5 to 25 parts by mass, and more preferably. from 1 to 5 parts by mass, based on 100 parts by mass of NBR. In the case of N, N'-m-phenylenedimaleimide, it is preferably from 0.1 to 30 parts by mass, and more preferably from 0.3 to 5 parts by mass, based on 100 parts by mass of NBR. Incidentally, lead oxide, copper oxide, manganese oxide, magnesium oxide and N, N'-m-phenylenedimaleimide may be used either alone or as in combination of two or more thereof. In the case of graphite, the added amount thereof is preferably from 3 to 25 parts by mass, and more preferably from 5 to 10 parts by mass, based on 100 parts by mass of NBR.

[0013]

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At least one selected from the group consisting of a thermal decomposition type foaming agent and non-crosslinkable, thermally expansible microcapsules is preferably incorporated in NBR for foaming. Preferred examples of the thermal

decomposition type foaming agents include DPT-based, ADCA-based and HDCA-based foaming agents. Further, one having a decomposition temperature of 120°C or more is preferred, and one having a decomposition temperature of 150 to 210°C is most preferred. The amount thereof incorporated is preferably from 1 to 100 parts by mass, and more preferably from 3 to 30 parts bymass, based on 100 parts by mass of NBR. The non-crosslinkable, thermally expansible microcapsules are preferably ones including a hydrocarbon in the resin. The amount thereof incorporated is preferably from 0.5 to 30 parts by mass, and more preferably from 1 to 20 parts by mass, based on 100 parts by mass of NBR.

[0014]

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Incidentally, crosslinkable microcapsules are unfavorable because crosslinking proceeds at the time of heating, resulting in a failure to obtain a high expansion ratio.

[0015]

In view of sealing properties, the expansion ratio of the foamed rubber layer is preferably from 1.4 to 10, and more preferably from 1.5 to 5.0. Incidentally, the expansion ratio is the ratio of the thickness of the rubber layer after foaming to the thickness thereof before foaming, and determined from the following equation:

[0016]

25 Expansion ratio = [(thickness of rubber layer after

foaming)/(thickness of rubber layer before foaming)]
 [0017]

Further, a filler can also be incorporated in NBR for reinforcement. As the filler, carbon black is preferred. The filler is incorporated in an amount of preferably 5 to 100 parts by mass, more preferably 10 to 70 parts by mass, based on 100 parts by mass of NBR.

[0018]

Besides, additives usually incorporated, such as an antioxidant, may be incorporated in NBR in an appropriate amount.

[0019]

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For producing the gasket material of the invention, above-mentioned crosslinking agent, crosslinking accelerator, foaming agent, filler and other additives may add to NBR in specific amounts to prepare a rubber compound, the rubber compound is dissolved or dispersed in an organic solvent to prepare a coating solution, and the coating solution is applied to the steel plate, followed by foaming. The crosslinking may occur at any stage in The crosslinking may occur the process. when the crosslinking agent is added to NBR, when the rubber compound is dissolved or dispersed in the organic solvent and/or during the foaming step. Although the organic solvent is not limited, example thereof includes a mixture of an aromatic hydrocarbon-based solvent such as toluene or a ketone-based solvent and an ester-based solvent in an arbitrary mixing ratio of from 10:90 to 90:10. rubber compound is dissolved or dispersed in this organic solvent to a solid concentration of 10 to 60% by mass to prepare the coating solution.

[0020]

Although there is also no limitation on the method for applying the coating solution, a method of being able to control the coating thickness is preferred. It is suitable to use a clearance coater, a roll coater or the like. The coating thickness is preferably from 10 to 200 μ m, and more preferably from 20 to 150 μ m.

[0021]

Thereafter, the foaming agent is foamed by performing 10 heat treatment under foaming conditions of the foaming agent, for example, at about 150°C to 240°C for about 5 to 15 minutes, to thereby form the foamed rubber layer. In that case, the foaming conditions such as the foaming agent to be used, the heating temperature and the heating time are adjusted so that 15 the above-mentioned expansion ratio is achieved.

[0022]

Kinds of the steel plate are not limited, and there can be used a steel plate which has been conventionally used in the gasket material, such as a stainless steel plate (ferrite—20 based/martensite—based/austenite—based stainless steel), a SPCC steel plate or an aluminum steel plate. These steel plates may be subjected to a chemical conversion treatment for forming a rust preventive film with a chromate treating agent, a non-chromate treating agent or the like, after alkali degreasing.

25 Further, a zinc phosphate or iron phosphate film is formed on

the SPCC steel plate in some cases. A similar film may be formed also in the invention. Furthermore, a steel plate whose surface is roughened by shot blast, Scotch blast or the like can also be used.

5 [0023]

The gasket material thus obtained has a high network chain density, as shown in Examples described later.

EXAMPLES

10 [0024]

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The invention will be described with reference to Examples and Comparative Examples, but should not be construed as being limited thereto in any way.

[0025]

15 (Preparation of Samples)

Rubber compounds each obtained by blending a rubber material, a filler, a crosslinking agent, a cross linking accelerator and a foaming agent were each dissolved or dispersed in a mixed organic solvent, obtained by mixing toluene with ethyl acetate in a mixing ratio of 7:3, to a solid concentration of 28.5% by mass to prepare coating solutions. A primer treatment was performed on stainless steel plates subjected to a non-chromium treatment, and the coating solutions were each applied to the steel plates by roll coater, respectively, followed by heat treatment at 210°C for 10 minutes, thereby

obtaining samples. In the Table 1, added amount is shown in parts by mass based on 100 parts by mass of DN3350.

[0026]

Table 1: Rubber Formulation

Component	and the state of t	Composition (main component)	Manufactured By
DN3350	Nitrile Rubber		
Stearic Acid			
Zinc Oxide			*************************************
MT Carbon	Carbon Black		
Nocrac PA	Antioxidant	Phenyl-1-naphthylamine	A
Sulfur			
Balnoc DGM	Crosslinking	p-p'-Benzoyl quinone dioxime	A
Balnoc GM-P	Agent	p-Quinone dioxime	A
Balnoc DNB		25% Poly-p-dinitrobenzene	A
Balnoc R		4-4'-Dithiodimorphrine	A
Nocceler CZ		N-Cyclohexyl-2-benzothiazoly	A
		l sulfenamide	
Nocceler TBT		Tetrabutylthiuram disulfide	A
Balnoc PM	Crosslinking	N-N'-m-phenylene dimaleimide	A
Sanceler MG	Accelerator	2-Mercaptobenzothiazole	В
Lead Oxide			
Manganese Oxide			
Copper Oxide			
Graphite			
Celmike A (Thermal		Azodicarbonamide	C
Decomposition Type)			
CAP 250 (Thermal Decomposition	Foaming Agent	Azodicarbonamide	C
Type)		+ urea	
DU-40(Non-Crosslinkable		Undisclosed	D
Microcapsules)		by manufacturer	

A: OUCHI SHINKO CHEMICAL INDUSTRIAL CO., LTD

C: Sankyo kasei Co., Ltd.

B: SANSHIN CHEMICAL INDUSTRY CO., LTD.

D: Japan Fillite Co., Ltd.

Table 1: Rubber Formulation (continued)

		Quinoid Crosslinking						
Component		Compo-	Compo-	Compo-	Compo-	Compo-	Сотро-	Compo-
		sition	sition	sition	sition	sition	sition	sition
		(1)	(2)	(3)	(4)	(5)	(6)	(7)
DN3350	Nitrile Rubber	100	100	100	100	100	100	100
Stearic Acid		1	1	1	1	1	1	1
Zinc Oxide		5	5	5	5	5	5	5
MT Carbon	Carbon Black	50	50	50	50	50	50	50
Nocrac PA	Antioxidant	5	5	5	5	5	5	5
Sulfur								
Balnoc DGM	Crosslinking	5	10					10
Balnoc GM-P	Agent				8	10	10	
Balnoc DNB				10				
Balnoc R								
Nocceler CZ								
Nocceler TBT								
Balnoc PM								
Sanceler MG	Crosslinking							
Lead Oxide	Accelerator							
Manganese Oxide								
Copper Oxide							2	
Graphite								2
Celmike A (Thermal		30	30		10			
Decomposition Type)								
CAP 250 (Thermal	Foaming Agent			30				15
Decomposition Type)							alan jakinga, malika jakin kalikan jakan jakan jakin kalikan jakin jakin jakin jakin jakin jakin jakin jakin j	
DU-40 (Non-Crosslink-						10	5	
able Microcapsules)								

Table 1: Rubber Formulation (continued)

		Quinoid Cr	osslinking	Quinoid Cross-	Bismaleimide
Component				linking	Crosslinking
		Compo-	Compo-	Composition	Composition
		sition (8)	sition (9)	(10)	(11)
DN3350	Nitrile Rubber	100	100	100	100
Stearic Acid		1	1	1	1
Zinc Oxide		5	5	5	5
MT Carbon	Carbon Black	50	50	50	50
Nocrac PA	Antioxidant	5	5	5	5
Sulfur					
Balnoc DGM	Crosslinking	5		10	
Balnoc GM-P	Agent		8		
Balnoc DNB					
Balnoc R					
Nocceler CZ					
Nocceler TBT					
Balnoc PM			3		10
Sanceler MG	Crosslinking				
Lead Oxide	Accelerator				
Manganese Oxide	,	3			
Copper Oxide					
Graphite					
Celmike A (Thermal		12	10		30
Decomposition Type)					
CAP 250 (Thermal	Foaming Agent				
Decomposition Type)	,				
DU-40(Non-Crosslink-		:			
able Microcapsules)					

Table 1: Rubber Formulation (continued)

		Dithiodimorpholine-Based	Sulfur Crosslinking
Component		Crosslinking Agent	
		Composition (12)	Composition (13)
DN3350	Nitrile Rubber	100	100
Stearic Acid		1	1
Zinc Oxide		5	5
MT Carbon	Carbon Black	50	50
Nocrac PA	Antioxidant	5	5
Sulfur			2
Balnoc DGM	Crosslinking		
Balnoc GM-P	Agent		
Balnoc DNB			
Balnoc R		1	
Nocceler C2		1	2
Nocceler TBT		4	
Balnoc PM			
Sanceler MG	Crosslinking		
Lead Oxide	Accelerator		
Manganese Oxide			
Copper Oxide			
Graphite			
Celmike A (Thermal		30	
Decomposition Type)			
CAP 250 (Thermal	Foaming Agent		24
Decomposition Type)			
DU-40 (Non-Crosslink-			
able Microcapsules)			

[0027]

Then, the following evaluations were performed on the respective samples. The results thereof are shown in Table 2.

5 (Evaluation Methods)

(1) Expansion Ratio

The thickness of the rubber layer before foaming and the thickness of the rubber layer after foaming were measured with a micrometer, and the expansion ratio was calculated from the following equation:

[0028]

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Expansion ratio = [(thickness of rubber layer after
foaming) / (thickness of rubber layer before foaming)]
[0029]

15 (2) Measurement of Network Chain Density

The foamed rubber layer was cut out to a size of 10 mm square, and the area thereof was measured (the area before swelling in dioxane solution). Thereafter, the foamed rubber layer cut was immersed in dioxane solution for 5 minutes and taken out. Then, the area swollen was calculated (the area after swelling in dioxane solution (the area after swelling in dioxane solution). The area before swelling in dioxane solution and the area after swelling in dioxane solution were substituted in the Flory-Rehner equation to determine the network chain density of the rubber.

[0030]

(3) Amount of Sulfur Contained

The amount of residual sulfur was measured by using combustion ion chromatography.

5 [0031]

Table 2: Evaluation Results

	Formulation (Table 1)	Thickness of Rubber before Foaming (µm)	Thickness of Rubber after Foaming (µm)	Expansion Ratio	Network Chain Density (mol/cm³)	Amount of Sulfur Contained (wt%)
Example 1	Composition (1)	80	224	2.8	0.032	0.08
Example 2	Composition (2)	100	290	2.9	0.061	0.07
Example 3	Composition (3)	100	250	2.5	0.057	0.02
Example 4	Composition (4)	70	217	3.1	0.048	0.04
Example 5	Composition (5)	90	468	5.2	0.063	0.06
Example 6	Composition (6)	50	180	3.6	0.088	0.05
Example 7	Composition (7)	120	372	3.1	0.082	0.06
Example 8	Composition (8)	50	165	3.3	0.055	0.07
Example 9	Composition (9)	70	395	5.6	0.091	0.07
Comparative Example 1	Composition (10)	65	65	1.0	0.059	0.08
Comparative	Composition	60	210	3.5	0.008	0.05
Example 2	(11)					
Comparative	Composition	70	161	2.3	0.052	1.01
Example 3	(12)					
Comparative Example 4*	Composition (13)	100	230	2.3	0.062	2.10

^{*} Note 1): A conventional foamed gasket material using sulfur

[0032]

From Table 2, it is known that the foamed rubber layers obtained by the quinoid crosslinking according to the invention are excellent in sealing properties required for the gasket material, and do not substantially produce free sulfur. Incidentally, sulfur detected in Examples was caused by contamination in the course of preparation of the rubber.

[0033]

While the present invention has been described in detail
and with reference to specific embodiments thereof, it will
be apparent to one skilled in the art that various changes and
modifications can be bade therein without departing from the
spirit and scope thereof.

[0034]

This application is based on Japanese patent application No. 2009-233565 filed on October 7, 2009, and the entire contents of which are incorporated hereinto by reference. All references cited herein are also incorporated in their entirety.

WHAT IS CLAIMED IS:

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1. A gasket material comprising a steel plate and a foamed rubber layer formed on one or both sides of the steel plate,

wherein the foamed rubber layer is obtained from a rubber composition containing: at least one of NBR, H-NBR or functional group-modified NBR, as a rubber compound; and a crosslinking agent, and

- wherein the foamed rubber layer has a quinoid crosslinking structure.
- The gasket material according to claim 1, wherein the crosslinking agent comprises at least one selected from the group consisting of p-quinone dioxime, p,p'-dibenzoyl-quinone dioxime and poly-p-dinitrobenzene.
- 3. The gasket material according to claim 1 or 2, wherein the rubber composition further contains at least 20 one selected from the group consisting of lead oxide, copper oxide, manganese oxide, magnesium oxide, N,N'-m-phenylenedimaleimide and graphite, as a crosslinking accelerator.
- 4. The gasket material according to any one of claims 1 to 3, wherein the rubber composition further contains at least one selected from the group consisting of: a DPT-based, ADCA-based and HDCA-based thermal decomposition type foaming agent; and non-crosslinkable and thermally expansible microcapsules, as a foaming agent.
 - 5. The gasket material according to any one of claims 1 to 4, wherein the foamed rubber layer has a

thickness before foaming of 10 to 200 μm and an expansion ratio of 1.5 to 5.0.

- 6. The gasket material according to any one of claims 1 to 5, which is a gasket material for an electric component.
 - 7. A process for making a gasket material comprising
- (i) adding a crosslinking agent to a rubber selectedfrom NBR, H-NBR or functional group modified NBR to produce a rubber composition,
 - (ii) dissolving or dispersing the rubber composition in an organic solvent to produce a coating composition,
- (iii) applying the coating composition to a steel
 15 plate to form a rubber layer,
 - (iv) crosslinking the rubber to form a quinoid crosslinking structure, and
 - (v) foaming the rubber layer.
- 8. A process according to claim 7, wherein the crosslinking agent comprises at least one selected from the group consisting of p-quinone dioxime, p,p'-dibenzoyl-quinone dioxime and poly-p-dinitrobenzene.
- 9. A process according to claim 8 wherein the crosslinking agent is added in an amount of 0.5 to 50.0 parts per mass based on 100 parts by mass of rubber.
- 10. A process according to any of claims 7-9 wherein 30 step (i) further comprises adding at least one selected from the group consisting of: a DPT-based, ADCA-based and HDCA-based thermal decomposition type foaming agent; and non-crosslinkable and thermally expansible microcapsules, as a foaming agent.

- 11. A process according to claim 10 wherein the non-crosslinkable thermally expansible microcapsules are added in an amount of 0.5 to 30 parts by mass based on 100 parts by mass of rubber.
 - 12. A process according to claim 10 wherein the decomposition type foaming agent is added in an amount of 1 to 100 parts by mass, based on 100 parts by mass of rubber.

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- 13. A process according to any of claims 7-12 wherein step (i) further comprises adding at least one selected from the group consisting of lead oxide, copper oxide, manganese oxide, magnesium oxide, N,N'-m-phenylenedimaleimide and graphite, as a crosslinking accelerator.
- 14. An apparatus comprising the gasket material of any one of claims 1-6, or the gasket material produced by20 the process of any one of claims 7-13, and an electrical component.



Application No: GB1016920.9 **Examiner:** Mr Kevin Hewitt

Claims searched: 1 to 14 Date of search: 26 January 2011

Patents Act 1977: Search Report under Section 17

Documents considered to be relevant:

Category	Relevant to claims	Identity of document and passage or figure of particular relevance
A	-	JP 05117479 A (JAPAN SYNTHETIC RUBBER) See especially the Abstract.
A	-	EP 1342751 A1 (TICONA) See whole document.
A	-	US 4173556 A (CORAN ET AL) See whole document.
A	-	GB 1533227 A (MONSANTO) See whole document.

Categories:

_			
X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	Р	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

Field of Search:

Search of GB, EP, WO & US patent documents classified in the following areas of the UKC^X :

Worldwide search of patent documents classified in the following areas of the IPC

C08L; F16J

The following online and other databases have been used in the preparation of this search report

WPI; EPODOC

International Classification:

Subclass	Subgroup	Valid From
F16J	0015/10	01/01/2006
C08L	0021/00	01/01/2006
F16J	0015/12	01/01/2006