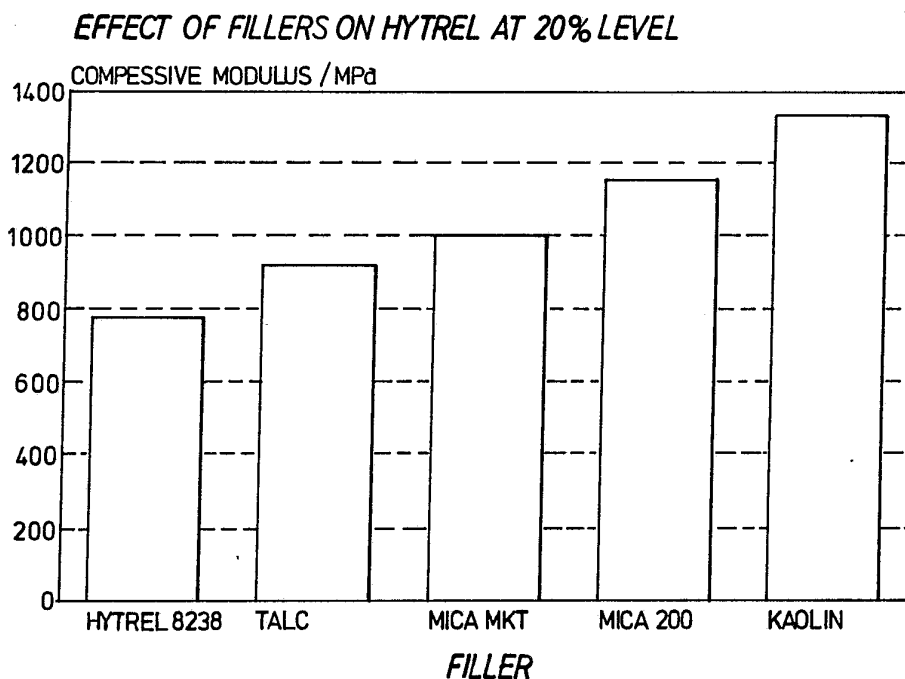




INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵ : F16J 15/12	A1	(11) International Publication Number: WO 92/08917 (43) International Publication Date: 29 May 1992 (29.05.92)
(21) International Application Number: PCT/GB91/01997 (22) International Filing Date: 13 November 1991 (13.11.91) (30) Priority data: 9024678.6 13 November 1990 (13.11.90) GB (71) Applicant (for all designated States except US): T&N TECHNOLOGY LIMITED [GB/GB]; Cawston House, Cawston, Rugby, Warwickshire CV22 7SB (GB). (72) Inventor; and (75) Inventor/Applicant (for US only) : FERDANI, Philip [GB/GB]; 36 Western Close, Dunchurch, Rugby, Warwickshire CV33 6QD (GB). (74) Agents: CRUX, John, Anthony et al.; T&N plc, Bowdon House, Ashburton Road West, Trafford Park, Manchester M17 1RA (GB).		(81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, LU (European patent), NL (European patent), SE (European patent), US. Published With international search report.

(54) Title: IMPROVED GASKETS



(57) Abstract

Injection moulded gaskets comprise a thermoplastic elastomer material containing a filler predominantly comprised of plate-like particles, the amount of filler being up to 30 % by weight of the material. The filler is preferably selected from the group comprising kaolin, graphite, mica and talc and the elastomer is preferably a polyester block copolymer predominantly comprised of polybutylene terephthalate.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	ES	Spain	MG	Madagascar
AU	Australia	FI	Finland	ML	Mali
BB	Barbados	FR	France	MN	Mongolia
BE	Belgium	GA	Gabon	MR	Mauritania
BF	Burkina Faso	GB	United Kingdom	MW	Malawi
BG	Bulgaria	GN	Guinea	NL	Netherlands
BJ	Benin	GR	Greece	NO	Norway
BR	Brazil	HU	Hungary	PL	Poland
CA	Canada	IT	Italy	RO	Romania
CF	Central African Republic	JP	Japan	SD	Sudan
CG	Congo	KP	Democratic People's Republic of Korea	SE	Sweden
CH	Switzerland	KR	Republic of Korea	SN	Senegal
CI	Côte d'Ivoire	LI	Liechtenstein	SU ⁺	Soviet Union
CM	Cameroon	LK	Sri Lanka	TD	Chad
CS	Czechoslovakia	LU	Luxembourg	TG	Togo
DE*	Germany	MC	Monaco	US	United States of America
DK	Denmark				

⁺ Any designation of "SU" has effect in the Russian Federation. It is not yet known whether any such designation has effect in other States of the former Soviet Union.

- 1 -

Improved gaskets

This invention relates to gaskets at least partly comprised of non-metallic material.

Known non-metallic gasket materials include fibre reinforced thermosetting materials, typically fibrous compositions which employ rubber as a binder. More recently, it has been proposed to use thermoplastic elastomers as gasket materials, but although these materials have excellent properties, resistance to progressive permanent deformation under load (herein referred to as "creep") is not one of them. Creep resistance is an important characteristic for a gasket material.

Whilst the introduction of some fibrous material as a reinforcement does improve the resistance to creep, this

- 2 -

improvement is only achieved at the expense of mouldability. The mouldability of fibre-free thermoplastic elastomer compositions is important, because it enables gasket manufacture by injection moulding, a process which makes optimum use of expensive materials by largely eliminating waste. But fibre reinforced materials have intrinsically higher melt viscosities which adversely affect their flow behaviour, for example in the passageways in moulding tools.

According to this invention, a gasket is injection moulded from a fibre-free thermoplastic elastomer material including a filler in the form of plate-like particles the amount of said filler being up to 30% by weight of the material. Particularly preferred fillers include kaolin, graphite, mica and talc, although other minerals such as vermiculite may also be useful. Kaolin has proved especially useful in amounts of up to 30 percent by weight. Wherein kaolin is used, it is preferably calcined and surface modified by treatment with an aminosilane. The use of plate-like fillers does not significantly detract from the elastomer flow properties, contrary to experience gained with fibre reinforced elastomers, so that the compositions can be injection moulded without a special mould design. A kaolin content of 20 percent by weight has proved very satisfactory for injection moulding in circumstances where the same thermoplastic elastomer

- 3 -

material was rendered virtually unmouldable by the addition of only 10 percent by weight of glass fibres.

The thermoplastic elastomer is preferably a polyester, polyester block copolymers being preferred. For example, a copolymer of polybutylene (polytetramethylene) terephthalate (PBT) with a polyester ether or a polyester glycol may be used, the "hard" segments of the PBT being complemented by the "soft" segments of the other component. It will be appreciated that PBT itself is not an elastomer; the desired blend of hard and soft segments may be achieved by copolymerisation and/or by blending with a polyester elastomer which is itself a block copolymer of PBT. However, for the present purposes a PBT content of at least about 70% by weight is preferred. It should be noted that blends of homopolymers are not generally desirable due to their tendency to undergo phase separation on injection moulding.

It has been found that whilst gaskets made according to the invention by injection moulding are in some ways inferior to fibre reinforced gaskets made from the same thermoplastic elastomer, they are at least equal when used as gaskets. Thus, although their tensile strength properties may well be inferior, their behaviour under compression is very similar to that of the fibre reinforced material. Behaviour under compressive loads is

SUBSTITUTE SHEET

- 4 -

much more significant than tensile strength for a gasket. However, the performance under injection moulding conditions is vastly superior to that of an equivalent fibre reinforced material for a given gasket configuration and moulding tool. A further advantage of the invention resides in the discovery that the products tend to be very significantly less prone to warpage and distortion than fibre reinforced gaskets of the same material.

In order that the invention be better understood, a preferred embodiment of it will now be described with the aid of the following Examples.

Comparative Example 1

An automotive engine sump gasket was injection moulded from a thermoplastic elastomer constituted by a polyester block copolymer sold under the registered trade mark HYTREL. The product was a flat gasket with good sealing properties, including heat resistance. However, it exhibited a tendency to creep, on prolonged exposure to normal clamping pressures in a test rig.

Comparative Example 2

An attempt was made to produce an identical gasket from the same elastomer material as that of comparative Example

- 5 -

1, but this time compounded to contain about 10 percent by weight of short glass fibres. In order to satisfactorily injection mould this new composition, it was necessary to modify the tooling, to provide wider flow channels. Even so, it was not easy to produce a satisfactory moulding. The product had significantly better tensile strength than the unreinforced gasket and its resistance to creep was greatly improved. However, its processing by injection moulding was at best difficult and the finished product exhibited unacceptable warpage for use in practical gasket applications.

Example 3

Next an identical gasket was produced in accordance with the present invention by injection moulding the same fibre-free elastomer material as was used in comparative Example 1 and to which had been added about 20 percent by weight of finely divided kaolin. The kaolin used was of a calcined and surface modified grade, the latter taking the form of an aminosilane surface treatment. Moulding performance was similar to that of unreinforced material, but the tensile strength was not as good as that of the glass fibre reinforced material. However, the behaviour under compression, including resistance to creep, was excellent, even after sustained exposure to clamping loads in a test rig.

- 6 -

To further illustrate this, the following table compares two grades of glass and kaolin filled HYTREL thermoplastic polyester elastomer when subjected to different tests, including the ASTM test designated F38. The latter test determines the percentage of clamping load lost after 22 hours at 100°C, starting from an initial load of 60 kN. It will be appreciated that ASTM test F38 is in this instance really a measure of creep.

<u>Grade</u>	<u>Filler</u>	<u>% wt</u>	<u>Tensile Modulus (GPA)</u>	<u>Compressive Modulus (GPA)</u>	<u>Tensile</u>	
					<u>Elongation at break %</u>	<u>F38 load loss %</u>
8238	none	-	0.5	0.97	400	52
8238	glass fibre	20%	1.41	2.24	6	27
8238	kaolin	20%	0.63	2.20	31	39
7246	none	-	0.29	0.27	28	59
7246	glass fibre	20%	1.05	1.02	8	41
7246	kaolin	20%	0.46	0.83	33	40

SUBSTITUTE SHEET

- 7 -

It can be seen that whilst the tensile modulus of kaolin filled material was inferior to that of the glass fibre filled material, the compressive moduli were comparable. The F38 load loss test confirmed this. The behaviour under injection moulding was however very much better, clearly demonstrating the benefit of the invention. Elastomer containing as much as 20 percent by weight of glass fibres was in fact useless for moulding into the form of the test sump gasket mentioned earlier. The F38 test results reflect the fact that in real gaskets, some load loss is to be expected. However, the 52-59 percent determined for unreinforced elastomer was too great for commercial utility in the particular application under investigation.

The invention is further illustrated by the accompanying bar chart Figure 1 in which the effect on compressive modulus of various fillers is shown by comparison with that of an unreinforced thermoplastic elastomer sold under the trade mark HYTREL, grade 8238. It will be seen that kaolin was superior to other materials tested, although finely divided mica ("MICA 200") also performed well.

To further illustrate the behaviour of gaskets made in accordance with the invention, a series of tests were carried out on a range of engines from a given manufacturer. Each test took the form of running the engine with a sample sump gasket through a series of

- 8 -

hot/cold cycles. The gasket was monitored for leakage and examined after testing to determine the loss of bolt loading developed.

Engine No 1 was tested with three different sump gaskets.

Gasket A was injection moulded from thermoplastic polyester block copolymer HYTREL grade 8238, with no filler.

Its leakage behaviour after only 100 cycles was rated as good, but a bolt load loss of 47% was observed. A test on a different engine showed oil leakage at 400 cycles, with a load loss of 33%.

Gasket B was injection moulded from HYTREL thermoplastic polyester block copolymer grade 8238, as before, but this time containing 20% by weight of a finely divided calcined kaolin which had been treated with an aminosilane. Its leakage behaviour after 400 cycles was rated as good and the bolt load loss was 28%.

Gasket C was injection moulded from a pure polybutylene terephthalate (PBT) containing 10% by weight of the same kaolin as was used for gasket B. The leakage performance at 400 cycles was again rated as good, but on inspection the gasket had cracked. Its bolt load loss was 36%.

- 9 -

The composition used for gasket B was also tested in several other engines, with similar results as regard leakage, no leakage being recorded in any case at 300 cycles and more. It is important to note with a gasket according to the present invention, satisfactory sealing was still achieved after 300-400 cycles, even in cases where a relatively high load loss was observed.

- 10 -

CLAIMS

1. An injection moulded gasket comprising an essentially fibre-free thermoplastic elastomer material including a filler in the form of plate-like particles, the amount of said filler being up to 30 percent by weight.
2. A gasket according to claim 1 wherein said filler is selected from kaolin, graphite, mica and talc.
3. A gasket according to any preceding claim wherein the filler is finely divided kaolin present in an amount from 10 to 20 percent by weight.
4. A gasket according to claim 2 or claim 3 wherein the kaolin is calcined and surface modified with a aminosilane prior to incorporation into the fibre-free thermoplastic elastomer material.
5. A gasket according to any preceding claim wherein said thermoplastic elastomer comprises a thermoplastic polyester.
6. A gasket according to any preceding claim wherein the thermoplastic elastomer is a polyester block copolymer.

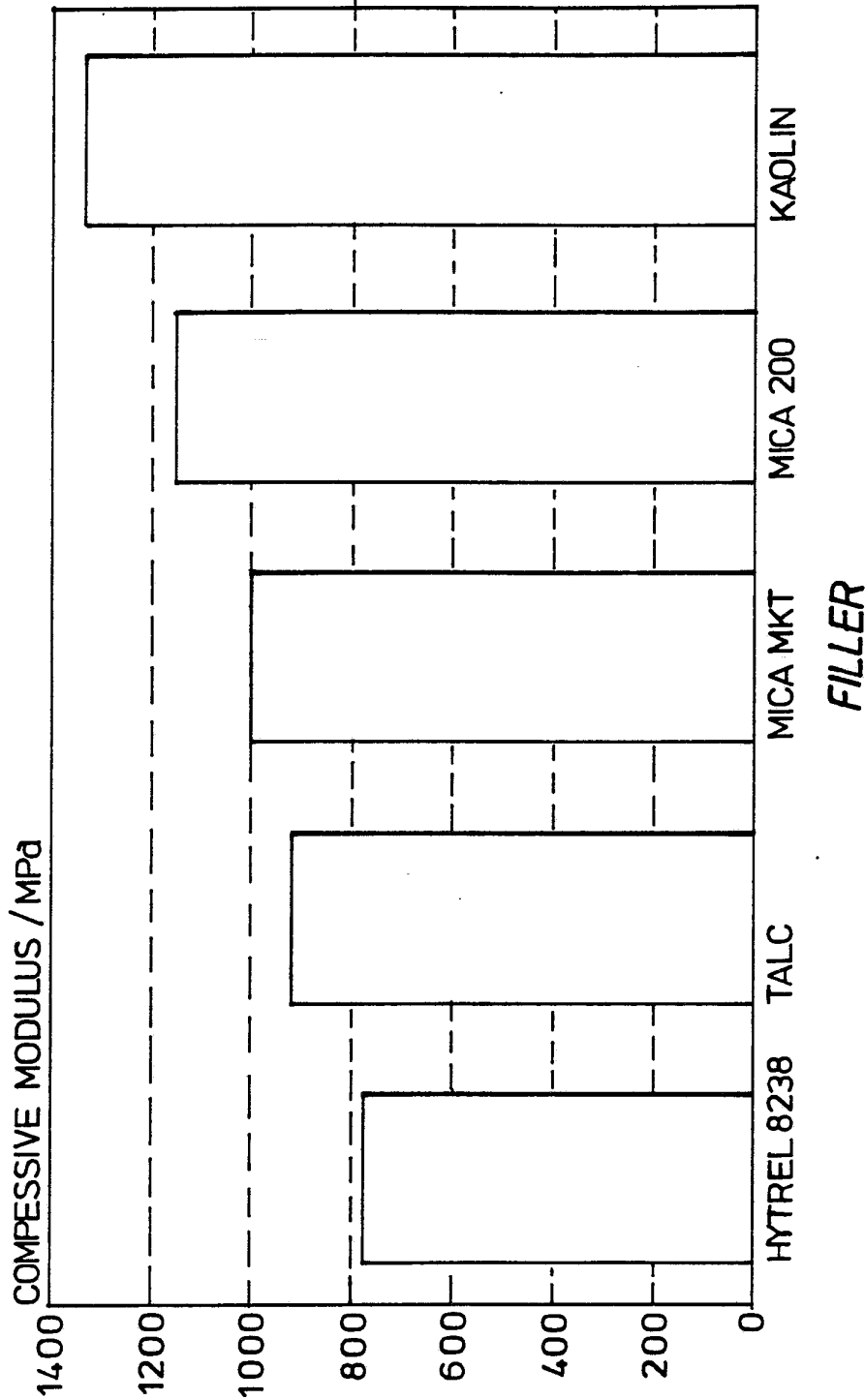
- 11 -

7. A gasket according to claim 6 wherein the polyester block copolymer comprises polybutylene terephthalate and a polyether ester.
8. A gasket according to claim 6 wherein the polyester block copolymer comprises polybutylene terephthalate and a polyether glycol.
9. A gasket according to claim 7 or claim 8 wherein the polybutylene terephthalate constitutes at least about 70% of the copolymer.
10. An injection moulded gasket comprising a thermoplastic elastomer including finely divided kaolin as a filler, substantially as described and illustrated with reference to Example 3.

1/1

FIG.1

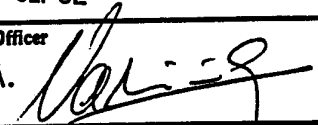
EFFECT OF FILLERS ON HYTREL AT 20% LEVEL



INTERNATIONAL SEARCH REPORT

PCT/GB 91/01997

International Application No

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 F16J15/12		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	F16J	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	EP,A,0 205 914 (W.R GRACE) 30 December 1986 see page 4, line 1 - line 12 ---	1-3,10
A	DE,U,8 510 511 (BURGMANN) 7 August 1986 see claim 1; figure 4 ---	1
X	PATENT ABSTRACTS OF JAPAN vol. 9, no. 324 (C-320)19 December 1985 & JP,A,60 155 284 (BRIGESTONE) 15 August 1985 see abstract ---	1
A	PATENT ABSTRACTS OF JAPAN vol. 11, no. 038 (C-401)4 February 1987 & JP,A,61 203 165 (TORAY) 9 September 1986 see abstract ---	5-9
A	EP,A,0 320 287 (EAGLE-PICHER) 14 June 1989 see abstract ---	1-2
¹⁰ Special categories of cited documents : ¹⁰ "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier document but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. "&" document member of the same patent family		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
11 FEBRUARY 1992	24. 02. 92	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	NARMINIO A. 	

ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO. GB 9101997
SA 53173

This annex lists the patent family members relating to the patent documents cited in the above-mentioned international search report.
The members are as contained in the European Patent Office EDP file on
The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information. 11/02/92

Patent document cited in search report	Publication date	Patent family member(s)	Publication date	
EP-A-0205914	30-12-86	DE-A-	3519053	18-12-86
		AU-B-	578155	13-10-88
		AU-A-	5742086	04-12-86
		CA-A-	1266734	13-03-90
		JP-A-	61276839	06-12-86
		US-A-	4801418	31-01-89
		US-A-	4743636	10-05-88

DE-U-8510511	07-08-86	None		

EP-A-0320287	14-06-89	US-A-	4849295	18-07-89
		AU-A-	2657788	15-06-89
		JP-A-	1266181	24-10-89
