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(54) Title: THERMOPLASTIC ELASTOMERS HAVING ENHANCED FOAMING AND PHYSICAL PROPERTIES

(57) Abstract: Soft thermoplastic vulcanizate foams are described comprising polyolefin thermoplastic resin, an at least partially crosslinked olefinic elastomer, hydrogenated styrenic block copolymer, and optional additives. The soft foams have smooth surfaces, low water absorption, improved compression set and compression load deflection.



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THERMOPLASTIC ELASTOMERS HAVING ENHANCED FOAMING AND PHYSICAL PROPERTIES

BACKGROUND OF THE INVENTION

Field of the Invention

This invention relates generally to thermoplastic elastomer (TPE) materials. Thermoplastic elastomers are broadly defined as rubber-like materials that, unlike conventional vulcanized rubbers, can be processed and recycled like thermoplastic materials, yet have properties and performance similar to that of vulcanized rubber at service temperatures. The invention more specifically relates to thermoplastic vulcanizates (TPV), which are thermoplastic elastomers with a cross-linked rubbery phase produced by the process of dynamic vulcanization. The thermoplastic vulcanizates of the invention are foamed materials produced by physical or chemical blowing agents, wherein processing characteristics and foam properties are improved by the inclusion of a modifier derived from styrene block copolymers. The invention also relates to foamed articles obtainable by the process of the invention.

Description of the Prior Art

There has been considerable activity on the development of thermoplastic vulcanizate compositions, especially those based on polyolefin thermoplastic resins, which have good foaming properties, and on processes for producing foams having improved properties. U.S. Pat. No. 5,070,111, incorporated herein by reference, discloses a process of foaming thermoplastic vulcanizate compositions using water as the sole foaming agent. U.S. Pat. Nos. 5,607,629 and 5,788,889, both incorporated herein by reference, describe methods for the production of foamed thermoplastic vulcanizate profiles by extrusion with a water blowing agent. U.S. Pat. No. 5,824,400 discloses foamed thermoplastic elastomer compositions which incorporate styrenic elastomers. Published European Patent Application No. 0 860 465 teaches a method of foaming thermoplastic vulcanizates using a water containing chemical compound which releases water at temperatures above the

melting point of the thermoplastic vulcanizate. Published European Patent Application 0 872 516 discloses the use of polypropylene resins having specific rheological properties to enhance the foaming performance of olefinic thermoplastic vulcanizates.

5 However, the problems of providing thermoplastic elastomer foams which are soft, with good surface smoothness, low water absorption, improved compression set and compression load deflection, and having fine and uniform cell structure have not been overcome by the prior art.

10 SUMMARY OF THE INVENTION

The present invention is based on the discovery that superior thermoplastic vulcanizate foams can be produced by incorporating into the thermoplastic vulcanizate, prior to foaming, a hydrogenated styrenic block copolymer. The use of this additive copolymer provides a very soft foam product having a number of
15 desirable attributes, including improved processability, high melt strength, high cell density and uniformity, smooth surface, low water absorption, with improved compression set and compression load deflection.

The present invention relates to a process for foaming a thermoplastic vulcanizate using a physical or chemical blowing agent, wherein a hydrogenated
20 poly(styrene-*b*-isoprene-*b*-styrene) block copolymer (SEPS) is incorporated into the thermoplastic vulcanizate composition prior to foaming. Sufficient SEPS is incorporated to be effective in achieving the desired attributes. The invention also encompasses thermoplastic vulcanizate compositions containing the SEPS, foamed articles prepared therefrom, and to the use of SEPS to improve said foams.

25 In detail the invention relates to a process for preparing a foamed thermoplastic vulcanizate, comprising the steps of blending

- A) polyolefin thermoplastic resin,
- B) olefinic elastomer,
- C) hydrogenated styrenic block copolymer,
- 30 D) crosslinking agent for said elastomer; and

- E) optional additives;
processing the resulting blend under dynamic vulcanization conditions to at least partially crosslink said elastomer;
incorporating a blowing agent into the blend; and
5 activating said blowing agent to form a foam.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

THERMOPLASTIC VULCANIZATE

10 Thermoplastic Resin Component

Thermoplastic resins suitable for use in the compositions of the invention include thermoplastic, crystalline polyolefin homopolymers and copolymers. They are desirably prepared from monoolefin monomers having 2 to 7 carbon atoms, such as ethylene, propylene, 1-butene, isobutylene, 1-pentene, 1-hexene, 1-octene, 3-
15 methyl-1-pentene, 4-methyl-1-pentene, 5-methyl-1-hexene, mixtures thereof and copolymers thereof with (meth)acrylates and/or vinyl acetates. Preferred, however, are monomers having 3 to 6 carbon atoms, with propylene being most preferred. As used in the specification and claims the term polypropylene includes homopolymers of propylene as well as reactor and/or random copolymers of propylene which can
20 contain about 1 to about 30 weight percent of ethylene and/or an alpha-olefin comonomer of 4 to 16 carbon atoms, and mixtures thereof. The polypropylene can have different types of molecular structure such as isotactic or syndiotactic, and different degrees of crystallinity including materials with a high percentage of amorphous structure such as the "elastic" polypropylenes. Further polyolefins,
25 which can be used in the invention, are high, low, linear-low and very low-density polyethylenes, and copolymers of ethylene with (meth)acrylates and/or vinyl acetates. These polyolefins include both linear or branched polymers.

The polyolefins mentioned above can be made using coordination polymerization catalyst systems, such as conventional catalyst systems,
30 conventional Ziegler/Natta catalyst and single site catalyst systems, such as

metallocene catalysts, and as well by other known means of olefin polymerization, such as free-radical polymerization. Commercially available polyolefins may be used in the practice of the invention.

The amount of thermoplastic polyolefin resin found to provide useful thermoplastic elastomer compositions is generally from about 8 to about 90 weight percent. Preferably, the thermoplastic polyolefin content will range from about 9 to about 60 percent by weight.

Elastomer Component

Suitable rubbers include non-polar, rubbery copolymers of two or more alpha-monoolefins, preferably copolymerized with at least one polyene, usually a diene. Saturated monoolefin copolymer rubber, for example ethylene-propylene copolymer rubber (EPM) can be used. However, unsaturated monoolefin rubber such as EPDM rubber is more suitable. EPDM is a terpolymer of ethylene, propylene and a non-conjugated diene. Satisfactory non-conjugated dienes include 5-ethylidene-2-norbornene (ENB); 1,4-hexadiene; 5-methylene-2-norbornene (MNB); 1,6-octadiene; 5-methyl-1,4-hexadiene; 3,7-dimethyl-1,6-octadiene; 1,3-cyclopentadiene; 1,4-cyclohexadiene; dicyclopentadiene (DCPD); and vinyl norbornene (VNB).

Butyl rubbers are also useful in the thermoplastic vulcanizate compositions.

As used in the specification and claims, the term butyl rubber includes copolymers of an isoolefin and a conjugated monoolefin, terpolymers of an isoolefin with or without a conjugated monoolefin, divinyl aromatic monomers and the halogenated derivatives of such copolymers and terpolymers. Another suitable copolymer within the scope of the olefin rubber of the present invention is a copolymer of a C₄₋₇ isomonoolefin and a para-alkylstyrene, and preferably a halogenated derivative thereof. The amount of halogen in the copolymer, predominantly in the para-alkylstyrene, is from about 0.1 to about 10 weight percent. A preferred example is the brominated copolymer of isobutylene and para-methylstyrene. Natural rubbers are also olefin rubbers suitable for use in the thermoplastic elastomer composition.

The amount of rubber in the thermoplastic vulcanizate generally ranges from about 92 to about 10 weight percent. Preferably the rubber content will be in the range of from about 40 to about 91 weight percent.

Additives

5 The thermoplastic vulcanizate may optionally contain reinforcing and non-reinforcing fillers, plasticizers, antioxidants, stabilizers, rubber processing oils, extender oils, lubricants, antiblocking agents, antistatic agents, waxes, foaming agents, pigments, flame retardants and other processing aids known in the rubber compounding art. Such additives may comprise up to about 70 weight percent,
10 more preferably up to about 65 weight percent, of the total composition. Fillers and extenders which can be utilized include conventional inorganics such as calcium carbonate, clays, silica, talc, titanium dioxide, carbon black and the like. The rubber processing oils generally are paraffinic, naphthenic or aromatic oils derived from petroleum fractions. The oils are selected from those ordinarily used in conjunction
15 with the specific rubber or rubber component present in the composition.

Processing

The rubber component of the thermoplastic vulcanizate is generally present as small, i.e. micro size, particles within a continuous thermoplastic resin matrix, although a co-continuous morphology or a phase inversion is also possible
20 depending upon the amount of rubber relative to thermoplastic resin and the degree of vulcanization, if any, of the rubber. Preferably, the rubber is at least partially vulcanized, and most preferably it is fully vulcanized (crosslinked).

The partial or full crosslinking can be achieved by adding an appropriate rubber curative to the blend of thermoplastic olefin polymer, or resin, and olefin
25 rubber, or elastomer, and vulcanizing the rubber to the desired degree under vulcanizing conditions. It is preferred that the rubber be Crosslinked by the process of dynamic vulcanization. As used in the specification and claims, the term dynamic vulcanization means a vulcanization or corsslinking (curing) process wherein the rubber is vulcanized under conditions of shear at a temperature above the melting
30 point of the thermoplastic resin component.

Those of ordinary skill in the art will appreciate the appropriate quantities and types of vulcanizing agents, and the conditions required to achieve the desired vulcanization. Any known crosslinking system can be used, so long as it is suitable under the vulcanization conditions for the elastomer component and it is compatible with the thermoplastic olefin polymer component of the composition. Crosslinking (curing) agents include sulfur, sulfur donors, metal oxides, phenolic resin systems, maleimides, peroxide based systems, hydrosilylation systems, high energy radiation and the like, both with and without accelerators and co-agents.

The terms fully vulcanized or completely vulcanized as used herein mean that the olefin rubber component of the composition has been crosslinked to a state in which the elastomeric properties of the crosslinked rubber are similar to those of the rubber in its conventional vulcanized rubber state. The degree of crosslinking (or cure) of the rubber can also be expressed in terms of gel content, crosslink density or amount of uncrosslinked rubber that is extractable by a rubber solvent. All of these descriptions are well known in the art. A typical partially crosslinked composition will have less than about 50 to less than about 15 weight percent of the elastomer extractable by a rubber solvent, while a fully crosslinked composition will have less than about 5 weight percent, and preferably less than about 3 weight percent, of the elastomer extractable by a rubber solvent.

Usually about 5 to about 20 parts by weight of the crosslinking agent or system are used per 100 parts by weight of the rubber component to be vulcanized.

The thermoplastic vulcanizates are rubbery materials having tensile set values of about 50% or less, which meet the standards for rubber as defined by ASTM D1566.

Styrenic Block Copolymer

The composition of the invention includes a hydrogenated styrene block copolymer component. This component is generally described as a hydrogenated poly(styrene-*b*-isoprene-*b*-styrene) block copolymer (SEPS). One such material

particularly suited for use in the process of the invention is Septon™ 2002, available from Septon Company of America Inc., a Kuraray Group company.

The amount of the SEPS component in the composition of the invention generally ranges from about 1 to about 10 weight percent, based on the total weight of the composition including the thermoplastic resin component, the rubber component, additives and the SEPS component. The preferred amount of SEPS ranges from about 1 to about 10 weight percent, with about 2 to about 4 weight percent being most preferred. Alternatively, the amount of SEPS can be expressed in terms of parts per hundred parts of rubber (phr). The preferred amount of SEPS, expressed in this manner, ranges from about 3 to about 40 phr with a range of about 5 to about 15 phr being most preferred.

In the preparation of thermoplastic vulcanizates of the invention, the SEPS was generally incorporated directly into the blend of resin and rubber during production of the thermoplastic vulcanizate, in a one step process, so that it was an integral part of the composition. Since the SEPS block copolymer is hydrogenated it contains very low levels of unsaturation and does not co-cure with the elastomer component during dynamic vulcanization. It is believed that the SEPS remains as a discrete phase in the thermoplastic vulcanizate. Alternatively, the SEPS can be mechanically blended with a preformed thermoplastic vulcanizate composition, or it can be introduced into the foaming process simultaneously with the thermoplastic vulcanizate.

EXAMPLES

Dynamically vulcanized blends of resins, rubber components and SEPS were converted into foam products by being fed into an extruder or other mixing device capable of maintaining melt temperatures in the range of about 165°C to about 220°C. If the blowing agent was a solid material, it was also blended with the components prior to introduction into the mixing device. When the blowing agent was a gas or liquid, it was injected into the mixing device through an appropriate inlet. The blowing agent was thus thoroughly dispersed in the molten blend, and the

mixture was maintained at a pressure sufficient to prevent premature foaming. The mixture was passed through a die or other appropriate outlet, where foaming occurred. The foamed product was cooled in air or in a water mist.

In the following examples thermoplastic vulcanizates were prepared from blends of polypropylene thermoplastic resin and EPDM rubber, with common additives and processing aids. SEPS was incorporated into the blends and the rubber component was cross-linked by dynamic vulcanization using a phenolic resin cure system to prepare the vulcanizates. For the fabrication of foamed articles by an extrusion process, the thermoplastic vulcanizate was introduced into a single screw extruder and thoroughly melted. The blowing agent, water in the examples set forth in Table 1, was then injected under pressure into the molten thermoplastic elastomer at rates of 1.1 to 1.4 weight percent. The melt was mixed and conveyed, under pressure, to the extruder exit and through a shaping die. The hot and fragile foam was transferred to a conveyor belt where it was cooled by air and water mist. The foamed article may then be cut or shaped for specific applications. Foamed profiles can be either extruded alone as described or coextruded with a dense carrier.

The following measurement methods were used in evaluating the examples of the invention:

Tensile strength at break; tensile set; tensile modulus; elongation at break - ASTM D412 (ISO 37, type 2)

Shore hardness - ASTM D2240

Specific gravity - ASTM D792

Surface (Ra) - Surface finish was evaluated as the arithmetic average of roughness irregularities measured from a mean line with the sampling length, using a Surface Analyzer System from Federal Products Corporation, Providence, RI.

Compression set - The sample was compressed inside spaced sample holders to 40% of its initial height, and held at 100°C for 22 hours. The sample was removed and allowed to recover for 30 minutes at room temperature. Compression set was then determined as: $CS(\%) = (H_{\text{initial}} - H_{\text{final}}) / (H_{\text{initial}} - H_0) \times 100$, where H_0 is the gap of the sample holder (60% of H_{initial}).

Compression load deflection - The force necessary to compress a 100 mm sample to 40% of its original height, at room temperature.

Water absorption - Two test methods were used to measure water absorption. In the first method (A) a weighed foam profile 50 mm long was submerged in water at room temperature two inches below the surface of the water. The specimen was allowed to remain submerged for either 24 hours at atmospheric pressure, or for three minutes at 23 inches Hg vacuum (above the surface of the water). After the appropriate time, the specimen was removed, blotted dry, weighed and the percent change in mass was calculated. In the second method (B) a weighed foam profile 254 mm long was submerged in water at room temperature eight inches below the surface of the water, with a one inch section of the specimen located above the water at each end. The specimen was allowed to remain thus submerged for either 24 hours at atmospheric pressure, or for five minutes at 26 inches Hg vacuum (above the surface of the water). After the appropriate time, the specimen was removed, blotted dry, weighed and the percent change in mass was calculated.

TABLE 1

Example ⇒	Control A	Control B	1	2	3	4	5
<u>Components</u> ↓ (phr)							
Polypropylene	42	32	35	30	25	22	20
EPDM rubber	100	100	100	100	100	100	100
Process oil	150	150	150	150	150	150	150
Additives/Curative	63	63	63	63	63	63	63
Septon™ 2002	0	0	12	15	20	15	20
Branched polypropylene						8	
<u>TPE Properties</u> ↓							
Hardness (Shore A)	66	60	61	57	54	56	47
Ultimate tensile strength (MPa)	6.9	5.6	5.1	3.2	4.0	4.6	3.4
Modulus - 100 (MPa)	2.71	2.63	1.9	1.7	1.5	1.6	1.1
Ultimate elongation (%)	520	368	377	354	368	384	332
Weight gain (%)	117	83	92	96	114	106	131
Viscosity (poise)	353	310	516	637	458	472	780
<u>Foam Properties</u> ↓							
Specific gravity (1.4 wt% water)	0.45	0.40	0.42	0.40	0.41	0.41	0.41
Surface, Ra (microns)	9.1	8.5	6.8	8.4	7.4	7.6	8.8
H ₂ O absorption (test A) - Atmosphere (%)		6.1	6.1	5.5	7.6	6.2	9.5
Compression set (%) 100° C, 22 hrs.	52	39	32	32	31	30	29
Compression load deflection (kgF/100mm)	0.77	0.44	0.56	0.58	0.50	0.69	0.67

As can be seen from the examples in Table 1, the foamed thermoplastic elastomer of the invention provides a smooth surface, low water absorption, good compression set and improved compression load deflection. Visual inspection shows that foam cell density is high and the cells are uniform in structure.

Additional examples were prepared using the same thermoplastic elastomer formulation (with 35 phr polypropylene), but varying the amount of SEPS or

replacing the SEPS with other block copolymers. Foams were generated from the compositions thus prepared, using various levels of water as the blowing agent in order to obtain foams with various densities. The thermoplastic elastomer and foam properties were evaluated, and the results are set forth in Tables 2 and 3.

TABLE 2

Example⇒	6	7	8	9	10	11	12
Block Copolymer↓ (phr)							
SEPS	5						
SEPS		12					
SEEPS			12				
SEBS-1				12			
SEBS-2					12		
SEPS						25	
SEPS							40
TPE Properties↓							
Ultimate tensile strength (MPa)	5.37	5.43	6.08	5.68	5.72	3.74	3.15
Modulus - 100 (MPa)	2.12	1.97	1.99	1.96	2.24	1.72	1.75
Ultimate elongation (%)	375	364	425	381	353	330	350
Extensional viscosity (kPa)	265	143	266	259	224	79	49

SEPS = Septon™ 2002 – hydrogenated poly(styrene-*b*-isoprene-*b*-styrene)

SEEPS = Septon™ 4033 – hydrogenated poly(styrene-*b*-isoprene-co-butadiene-*b*-styrene)

SEBS-1 = Septon™ 8007 – hydrogenated poly(styrene-*b*-butadiene-*b*-styrene)

SEBS-2 = Kraton™ G1652 – hydrogenated poly(styrene-*b*-butadiene-*b*-styrene)

TABLE 3 – Foam Properties

Example ⇒ Property ↓	Example 6				Example 7				Example 8				Example 9			
	0.33	0.40	0.57	0.30	0.35	0.38	0.51	0.42	0.45	0.64	0.38	0.44	0.58	0.38	0.44	0.58
Specific gravity	4.2	3.1	2.6	3.1	1.9	2.9	3.1	2.1	1.8	2.0	4.3	2.0	1.4	4.3	2.0	1.4
H ₂ O absorption (atmosphere) %	0.54	1.1	0.74	0.83	1.3	0.83	1.7	0.6	0.37	0.41	0.63	0.39	0.48	0.63	0.39	0.48
H ₂ O absorption (vacuum) %	30	33	35	38	35	35	38	37	35	40	36	34	43	36	34	43
Compression set – 100°C, 22 hr. (%)	0.74	0.84	1.30	0.45	0.65	0.59	1.10	1.10	1.20	1.30	0.72	1.30	1.60	0.72	1.30	1.60
Compression load Deflection KgF/100 mm	0.33	0.40	0.57	0.30	0.35	0.38	0.51	0.42	0.45	0.64	0.38	0.44	0.58	0.38	0.44	0.58
Surface Ra (microns)																

TABLE 3 – Foam Properties (Cont'd)

Example ⇒ Property ↓	Example 10			Example 11			Example 12 ⁵		
	0.41	0.46	0.65	0.33	0.55	0.28	0.33	0.51	
Specific gravity	2.0	2.1	3.4	2.7	3.6	8.0	6.3	1.5	
H ₂ O absorption (atmosphere) %	0.42	0.42	0.3	0.85	1.4	1.0	1.5	2.1	
H ₂ O absorption (vacuum) %	32	36	41	40	20	61	58	49 ¹⁰	
Compression set – 100°C, 22 hr. (%)	1.0	1.20	1.50	0.56	1.20	0.40	0.60	1.30	
Compression load Deflection KgF/100 mm	0.41	0.46	0.65	0.33	0.55	0.28	0.33	0.51	
Surface Ra (microns)									

The materials used in the thermoplastic vulcanizates of Table 1 were EPDM rubber - Vistalon™ 3666 (ExxonMobil Chemical Co.); polypropylene - D008M™ (Aristech Chemical Corp.); process oil - Sunpar™ 150M; clay - Icecap K™ (Burgess); curative - SP-1045™ (Schenectady International); carbon black - Ampacet 49974 (Ampacet Corp.); branched polypropylene – HMS 130D (Borealis).

The foamed thermoplastic vulcanizate composition and the molded and shaped articles made therefrom are useful in a variety of applications such as handles and grips for tools or utensils, as well as weather strip for automotive and construction uses.

WHAT IS CLAIMED IS:

1. Process for preparing a foamed thermoplastic vulcanizate, comprising the steps of:

A) blending

A) polyolefin thermoplastic resin,

B) olefinic elastomer,

C) hydrogenated styrenic block copolymer,

D) crosslinking agent for said elastomer, and

E) optional additives;

B) processing the resulting blend under dynamic vulcanization conditions to at least partially crosslink said elastomer;

C) incorporating a blowing agent into the blend; and activating said blowing agent to form a foam.

2. The process of claim 1 wherein said blend contains from about 8 to about 90 weight percent polyolefin thermoplastic resin (A) and from about 92 to about 10 weight percent olefinic elastomer (B), based on the total weight of (A) + (B), and from about 1 to about 10 weight percent of block copolymer (C), based on the total weight of the blend.

3. The process of claim 1 wherein the dynamic vulcanization is carried out to the extent that said elastomer (B) is fully crosslinked.

4. The process of claim 1 wherein said polyolefin thermoplastic elastomer (A) is polypropylene, said olefinic elastomer (B) is EPDM rubber, and said block copolymer (C) is hydrogenated poly(styrene-*b*-isoprene-*b*-styrene) block copolymer.

5. The process of claim 1 wherein said crosslinking agent is a phenolic resin system.

6. A foamed thermoplastic vulcanizate produced by the process of claim 1.
7. A foamed thermoplastic vulcanizate comprising
 - A) polyolefin thermoplastic resin,
 - B) an at least partially crosslinked olefinic elastomer,
 - C) hydrogenated styrenic block copolymer, and
 - D) optional additives.
8. The thermoplastic vulcanizate of claim 7 wherein said composition contains from about 8 to about 90 weight percent polyolefin thermoplastic resin (A) and from about 92 to about 10 weight percent olefinic elastomer (B), based on the total weight of (A) + (B), and from about 1 to about 10 weight percent of block copolymer (C), based on the total weight of the blend.
9. The thermoplastic vulcanizate of claim 7 wherein said elastomer (B) is fully crosslinked.
10. The thermoplastic vulcanizate of claim 7 wherein said polyolefin thermoplastic elastomer (A) is polypropylene, said olefinic elastomer (B) is EPDM rubber, and said block copolymer (C) is hydrogenated poly(styrene-*b*-isoprene-*b*-styrene) block copolymer.
11. The use of a hydrogenated styrenic block copolymer block copolymer in the preparation of a soft thermoplastic foam, said foam comprising polyolefin thermoplastic resin, an at least partially crosslinked olefinic elastomer, and optional additives, for the purposes of improving processability and melt strength and increasing cell density and uniformity.
12. The use according to claim 11 wherein said block copolymer is hydrogenated poly(styrene-*b*-isoprene-*b*-styrene) block copolymer, said polyolefin thermoplastic elastomer is polypropylene, said olefinic elastomer is EPDM rubber.