

COMMONWEALTH of AUSTRALIA
Patents Act 1952

636715

APPLICATION FOR A STANDARD PATENT

I/We

Shell Oil Company

of

900 Louisiana Street, Houston, Texas, 77002, United States of America

hereby apply for the grant of a Standard Patent for an invention entitled:

Polymer blends for use in providing peelable heat seals

which is described in the accompanying complete specification.

Details of basic application(s):-

<u>Number</u>	<u>Convention Country</u>	<u>Date</u>
486561	United States of America	28 February 1990

The address for service is care of DAVIES & COLLISON, Patent Attorneys, of 1 Little Collins Street, Melbourne, in the State of Victoria, Commonwealth of Australia.

DATED this TWENTY SIXTH day of FEBRUARY 1991

To: THE COMMISSIONER OF PATENTS


.....
a member of the firm of
DAVIES & COLLISON for
and on behalf of the
applicant(s)

Davies & Collison, Melbourne

0250 260201

PATENTS ACT 1952-1973

In support of the Application made for a Patent ~~XXXXXXXXXXXX~~ for an invention entitled "Polymer Blends for use in Providing peelable Heat Seals"

Subject matter of invention

[illegible]

DOUGLAS BALDWIN, Assistant General Counsel

of: SHELL OIL COMPANY, a Corporation organised and existing under the laws of the State of Delaware, United States of America of:900, Louisiana, Houston, Texas 77001, United States of America.

These two sentences of Paragraph 9
1(a) or 1(b) does not apply

do solemnly and sincerely declare as follows :—

2(c) false in application made
by individual(s)

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2(b) relates to application made by company; insert name of applicant company.

er (b) I am authorized by
SHELL OIL COMPANY.

Cross out whichever of paragraphs 2(a) or 2(b) does not apply

the applicant..... for the patent ~~patent of addition~~ to make this declaration on its behalf ~~then~~

2(a) relates to application made
by inventor(s)

~~XX~~

3. (b) relates to application made by company(s) or person(s) who are not inventor(s). Insert full name(s), and address(es) of inven-

or (b) .

Robert W. Lee, of 3202 Ashton Park Drive, Houston
Texas 77082, United States of America and
Charles C. Hwo, of 2710 Sugarwood Drive, Sugarland
Texas 77478, United States of America

~~X~~ are the actual inventor. S..... of the invention and the facts upon which the applicant ~~X~~.....
is entitled to make the application are as follows :-

"The Applicant would, if a patent were granted upon an application made by the Inventors, be entitled to have the patent assigned to it"

State manner in which applicant(s)
derive title from inventor(s)

*Info on paragraphs 3 and 4 for non-employment applications. For employment applications, insert basic security (a) followed by state(s) and basic application(s).

3. The basic application..... as defined by Section 141 of the Act ^{was} ~~made~~ ^{made}
in U.S.A. (No. 486,561)..... on the 28th day of February 1990
by Robert W. Lee and Charles C. Hwo
.....
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6. The basic application..... referred to in paragraph 3 of this Declaration ^{was} ~~were~~ the first application..... made in a Convention country in respect of the invention the subject of the application.

ನಿಮ್ಮ ಪರೀಕ್ಷೆ ಮಾಡಿ ಪೂರೈಕೆ ಆಗಿಲ್ಲವೆಂದು.

Declared at Houston, Texas this 5th day of April 1991

វិទ្យាស្ថាន ៩៧ វិទ្យាស្ថាន(១) (គណនេយ្យសាស្ត្រ)

Douglas Baldwin
DOUGLAS BALDWIN

Atch: Initial all discussion.

ASSISTANT GENERAL COUNSEL



AU9171393

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(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 636715

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POLYMER BLENDS FOR USE IN PROVIDING PEELABLE HEAT SEALS
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C08L 053/02
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- (71) Applicant(s)
SHELL OIL COMPANY
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- (74) Attorney or Agent
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- (56) Prior Art Documents
US 4550141
US 4111895
AU 493845 14171/76 C08L 23/20 C08L 53/02
- (57) Claim

1. A polymer blend for use in providing a peelable heat seal between substrates which comprises

(a) 5 to 50 weight percent of a butene-1 homopolymer or copolymer having a melt index of 0.01 to 1000;

(b) 95 to 50 weight percent of a styrenic-olefinic block copolymer; and

(c) 0 to 30 percent by weight of polypropylene, the weight percentages of (a), (b) and (c) being based on their combined weight.

10. A laminar structure comprising a coextrudate of a backing material and a blend as claimed in any one of the preceding claims.

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13. A peelably sealed rigid package comprising a rigid container having a lid sealed thereon with a film comprising a blend as claimed in any one of claims 1 to 9 or laminar structure as claimed in claim 10, 11 or 12.

636715

COMMONWEALTH OF AUSTRALIA
PATENTS ACT 1952
COMPLETE SPECIFICATION

NAME & ADDRESS
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NAME(S) OF INVENTOR(S):

Robert W LEE
Charles C HWO

ADDRESS FOR SERVICE:

DAVIES & COLLISON
Patent Attorneys
1 Little Collins Street, Melbourne, 3000.

COMPLETE SPECIFICATION FOR THE INVENTION ENTITLED:

Polymer blends for use in providing peelable heat seals

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

The present invention relates to polymer blends for use in providing a peelable heat seal between substrates, such as a rigid container and a peelable lid therefor, or in a flexible film package made from films and/or laminates. Seals produced using these blends are characterised by an adequate, nearly constant peel strength when used on rigid containers including styrenic containers and polyvinyl chloride (PVC) containers. The seals can be formed over a wide range of heat seal temperatures and are easily peelable. The term peelable refers to a seal failure of which occurs primarily at the interface of the sealing surfaces, rather than by film tearing.

A widely used package comprises a laminated lid material peelably bonded to a rigid container. The lid material is typically made from a backing material coated with a heat sealable material so that it can be easily peeled from the rigid container. The backing material may be any material strong enough to close said container and also to provide any barrier properties which may be needed. The backing material may be, for example, metal foil, polypropylene film, polyester film, nylon film or paper. The package is opened by peeling off the lid material, and this must generally be done without destroying the integrity of the lid itself. Accordingly, to provide a

satisfactory package of this type, a balance of properties is necessary, i.e. the seal strength must be adequate to maintain the package in a closed and protective condition, while also exhibiting such release properties as will

5 permit opening by peeling when desired. In the past there have been no inexpensive and effective peel seal closures for use with rigid styrenic containers or with PVC-based containers because no single peelable adhesive could be used with both types of container.

10 Most commercial peel seal materials are olefin-based copolymers that can not be used for containers of polystyrene or PVC since there is no adhesion between the former and the latter two types of polymer.

At the present time, in order to use the easy open, 15 peelable lid concept on polystyrene or PVC rigid containers, the manufacturer must coextrude polystyrene or PVC with high density polyethylene (HDPE) to form a container with a HDPE surface which can be sealed with peel seal material. This is a costly process.

20 One type of peel seal material currently available for polystyrene containers is an ethylene/vinyl acetate copolymer (EVA) based liquor. Use of this material involves venting and recovering of organic solvent while EVA often produces organoleptic problems in food which is 25 packaged in such containers.

Various references teach heat sealable films capable of forming peel seals, such as US-A-4,550,141;

US-A-4,539,263; US-A-4,414,053 and US-A-3,879,492.

However, these references do not appear to teach a composition which will provide a peelable seal for rigid styrenic, PVC or olefinic containers.

5 The invention provides a polymer blend which can be used to provide peelable seals which will adhere to a variety of substrates and can be used with chemically compatible backing materials to provide peel sealable packages, such as polystyrene, PVC or olefinic containers.

10 The invention thus provides a blend which will provide a peelable seal for a wide variety of rigid containers.

 According to the invention there is provided a polymer blend for use in providing a peelable heat seal between substrates which comprises

15 (a) 5 to 50, preferably 10 to 40, especially 25 to 35, weight percent of a butene-1 homopolymer or copolymer having a melt index of 0.01 to 1000, preferably 0.1 to 500;

 (b) 95 to 50, preferably 90 to 60, especially 75 to 65, weight percent of a styrenic-olefinic block copolymer; and

20 (c) 0 to 30 percent by weight of polypropylene, the weight percentages of (a), (b) and (c) being based on their combined weight.

 This blend, when laminated between a backing material and a rigid container at a heat sealing
25 temperature of between 177°C and 232°C (350°F and 450°F) as determined at sealing jaws, affords a peel strength of 0.18 to 2.3 kg per 2.54 cm (0.4 to 5 pounds per inch) between

the backing material and the rigid container.

The invention also comprises a laminar structure suitable for use as a lid for a rigid container, said laminar structure comprising a backing material and a layer
5 of the blend of the invention. The laminar structure can be a coextrudate of the backing material and the blend or can comprise the backing material coated with the blend. Such a laminar structure, e.g. using aluminum foil as backing material, yields an easy open, peelable lid
10 material for styrenic, PVC or olefinic rigid containers. The laminar structure can be heat sealed onto PVC, crystal polystyrene, or impact modified polystyrene or olefinic containers. Lid stock made from the blend has a broad heat seal temperature range. The backing material is suitably
15 selected from polystyrene, polypropylene film, aluminum foil, nylon film, polyester film, polycarbonate film, poly-4-methyl pentene-1 film and paper.

The invention further provides a peelably sealed rigid package comprising a rigid container having a lid
20 sealed thereon with a film comprising the blend of the invention or a laminar structure according to the invention. Preferably the rigid container is of a styrenic polymer, nylon polyester, polyvinyl chloride or an olefinic polymer.

25 The polymer blend of the invention may be formed into unoriented or oriented film by a casting or film blowing method. After fabrication the films can be heat

sealed by sealing jaws at a preset temperature, pressure and dwell. These manufacturing techniques apply to film, although the blends can be formed into sheeting. Film refers to shaped plastics that are comparatively thin and
5 have a maximum thickness of 0.245 mm (0.010 inches or 10 mils). Sheetting is a shaped plastic having a thickness greater than 0.254 mm (0.010 inches).

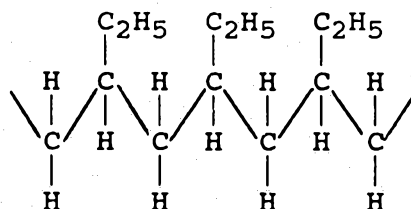
The laminar structure of the invention can be made by making two separate films that are then laminated, as by
10 heat lamination. The films may be prefabricated by either film blowing (melt extrusion with a circular die) or the casting method (a flat die or slot die-melt extrusion process). Alternatively the laminar structure can be prepared by coextrusion. Coextrusion techniques that can
15 be used are described in US-A-2,480,998.

The extrusion process starts with a polymer in a form that can be fed continuously into an extruder by means of a screw or pneumatic tube. Sometimes the polymers are combined with materials such as plasticizers, lubricants,
20 stabilizers, and colorants by means of Banbury mixers. The resulting mix is extruded through rod shaped dies and chirped into pellets. Pelletized polymer is fed into a screw conveyor into the end of a screw-type extruder and is heated and made into viscous fluid in a cylinder by means
25 of a revolving, helical screw. The sheet emitting from the die may be quenched on a temperature controlled chill roll. Finished films may be subject to a two-way stretching using

continuous tenter-frame operations in biaxial orientation.

The term "polybutylene" is used herein to denote component (a) and refers to butene-1 homopolymer and butene-1 copolymers such as butene-1-ethylene copolymer and butene-1-propylene copolymer.

The polybutylene which is a basic component of the blends of this invention suitably is a product of a stereospecific polymerization procedure and is referred to as an "isotactic" or "stereospecific" polymer in that the repeating units of its polymeric chain all possess the same stereochemical configuration along the chain. This is to be contrasted with "atactic" polymers wherein the repeating units of the polymeric chain vary in a random configuration along the chain. The stereochemical nature of isotactic polybutylene required for use in novel adhesives may be readily observed from the following structural depiction thereof:



High molecular weight, isotactic polybutylenes utilized in the blends of this invention are prepared by the stereospecific polymerization of butene-1 monomer. Methods for conducting such polymerization procedures are

well known to those skilled in the art; typical procedures being disclosed in US-A-3,197,452, and in NL-A-6507546.

These procedures generally involve the use of polymerization initiators or catalysts for the

5 polymerization of the butene-1 monomer to polymers of high molecular weight. Among the preferable catalytic systems utilized in such procedures are the reaction products of metal alkyl compounds, such as aluminum triethyl, and a heavy metal compound, such as the trihalides of Groups IV--
10 VI metals in the Periodic Table, e.g. titanium, vanadium, chromium, zirconium, molybdenum and tungsten. The formation of polymers exhibiting substantial isotactic properties as well as the variations in the molecular weight thereof will thus depend on the nature of the
15 polymerization catalyst, the nature of the co-reactants in the system and the reaction conditions being utilized.

Suitable isotactic polybutylenes are relatively rigid at normal temperatures but flow readily upon being heated. As noted, they must exhibit a high molecular
20 weight. Thus, expressing molecular weight in terms of "melt flow," the applicable isotactic polybutylenes to be used in these novel adhesives should exhibit a melt flow in the range of from 0.1 to 500, preferably 0.2 to 300, more preferably from 0.4 to 40.0, still more preferably 0.4 to
25 7, especially 1 to 4. Such melt flow values are determined by the method described in ASTM D 1336-62 Tc and are inversely related to molecular weight, i.e. the lower the

melt flow value, the higher the molecular weight. In addition, it should be noted that minor amounts, i.e. up to 5% by weight, of alpha-olefin comonomers, such as ethylene and propylene, may be present in the butene-1

5 polymerization system without any substantial loss of the herein-described desirable properties displayed by the resultant butene-1 polymer. Preferably the butene-1 polymer is a high molecular weight, flexible, polymer having a density of about 0.91 g/cm^3 .

10 The optional polypropylene component (c) of the blends of the invention is used to modify melt index or adhesive properties.

The polybutylene employed herein is preferably of a film-forming grade, and can be obtained commercially from
15 the Shell Company or prepared as described in US-A-3,362,940.

The block copolymer component (b) of the blends of the present invention is well known and can be produced by conventional methods. The block copolymer can be produced
20 in geometric forms referred to as linear, branched and radial. Preferred methods of production of the block copolymers involve the use of a lithium polymerization initiator, particularly a lithium alkyl. US-A-3,592,942 describes block copolymers and methods of their production.
25 Linear block copolymers are typically produced by sequential addition of suitable monomers into a polymerization reactor in the presence of the lithium alkyl initiator. The living polymer chain grows through sequential polymerization. Linear block copolymers are



also illustratively produced by coupling of two living polymer chains with a difunctional coupling agent such as a dihaloalkane. Branched block copolymer structures are obtained through coupling of living polymers chains with a coupling agent having a functionality of three or more. Use of a coupling agent which is non-symmetrical results in block polymer of a structure termed branched. The precise nature of any coupling agent does not contribute substantially to the properties of the block copolymer and may be ignored so far as a description of the polymers is concerned.

The block copolymer suitably has at least two blocks, one designated A of a polymerized vinyl aromatic compound and a second designated B of a polymerized conjugated alkadiene. The block copolymer can comprise 8% to 55% by weight, preferably 10% to 30% by weight, of block(s) A with the remainder being block(s) B. Each A block preferably has an average molecular weight of 5,000 to 125,000, preferably 7,000 to 60,000. Each B block preferably has an average molecular weight of 10,000 to 300,000, preferably 30,000 to 150,000.

The A blocks are produced by polymerization of a vinyl aromatic compound such as styrene or analogs of styrene such as α -methylstyrene or ring methylated styrenes including p-methylstyrene and α -methylstyrene and styrene is particularly preferred.

A B block is preferably a homopolymeric block of

butadiene or isoprene but copolymers with a vinyl aromatic compound are also suitable so long as the B block predominantly comprises conjugated alkadiene units.

Aliphatic unsaturation in the block copolymer (b) can be
5 hydrogenated, while aromatic unsaturation is not hydrogenated to a significant extent. A particularly preferred non-hydrogenated B block is homopolymeric butadiene. In the case of butadiene, polymerization to
10 give polymer units having a 1,2-structure or a 1,4-structure is possible. Preferred B blocks produced from butadiene have from 35% to 55% of units having a 1,2-structure with the remainder being units of a 1,4-structure.

Illustrative of the block copolymers which can be
15 used as such or as precursors of hydrogenated block copolymers are block copolymers of the following types:

- polystyrene-polybutadiene-polystyrene (SBS)
- polystyrene-polyethylene-polybutadiene-polystyrene (SEBS)
- polystyrene-polyisoprene-polystyrene (SIS)
- 20 poly(α -methylstyrene)-polybutadiene-poly(α -methylstyrene)
- poly(α -methylstyrene)-polyisoprene-poly(α -methylstyrene).

A hydrogenated block copolymer can be produced by substantially complete hydrogenation of the aliphatic unsaturation of the B block while very little of the
25 aromatic unsaturation of the A blocks is affected. Such selective hydrogenation is conventional.

Hydrogenation of the block copolymer may be

effected in conventional manner, for example using a catalyst formed by reaction of an aluminum alkyl with a nickel or cobalt carboxylate or alkoxide. Such a catalyst serves to partially or selectively hydrogenate the block

5 copolymer in that at least 80% of the aliphatic unsaturation is hydrogenated while hydrogenating no more than 25% of the aromatic unsaturation. Preferably at least 99% of the aliphatic carbon-carbon double bonds are hydrogenated while less than 5% of the aromatic

10 unsaturation is hydrogenated. Such selective hydrogenation is now conventional and is described in US-A-3,595,942 and US-B-27,145. For a general description of block copolymers and their production before and after hydrogenation see US-A-4,111,895.

15 The hydrogenated block copolymers can have an average molecular weight of from 25,000 to 350,000. Preferred hydrogenated block copolymers have a molecular weight from 35,000 to 300,000. Such hydrogenated block copolymers are commercially available and are sold by Shell

20 Chemical Company under the tradename "KRATON" (RTM) G Thermoplastic Rubber.

The blends of the invention may further contain up to 25% by weight, preferably 10-20% by weight, of a plasticizing or extending oil in order to provide wetting

25 action and/or viscosity control. The plasticizing oil can be, for example, a paraffinic or naphthenic oil. Petroleum derived oils which may be employed are relatively high

boiling materials containing only a minor proportion of aromatic hydrocarbons (preferably less than 30%, more particularly less than 15%, by weight of the oil). Alternatively, the oil may be totally nonaromatic.

5 Other additives such as plasticizers, pigments, dye-stuffs, slip agents, or antiblock agents conventionally added to hot melt adhesives for various end uses contemplated may also be incorporated in minor amounts into the blends of the present invention.

10 The following Examples will serve to illustrate the present invention. As will become evident from the Examples, the blends of the present invention provide seals having a nearly constant peel strength over extended heat seal temperature ranges when used with a wide variety of
15 container compositions.

EXAMPLES

Approximately 4.5 kg (ten pounds) of each two component formulation was dry blended and is listed in Table 1. Percentages are by weight.

TABLE 1

BLEND NO.	% BLOCK COPOLYMER	% POLYBUTYLENE
1	65 KRATON G1726X Rubber	35 DP1560
2	70 " " "	30 "
3	75 " " "	25 "
4	100 " " "	-----
5	70 KRATON G1726X Rubber	30 PB1710
6	70 " " "	30 PB0300
7	65 KRATON D1102 Rubber	35 DP1560
8	75 " " "	25 "
9	100 " " "	-----
10	65 KRATON D1107 Rubber	35 DP1560
11	75 " " "	25 "
12	100 " " "	-----
13	75 " " "	25 PB0300
14	65 " " "	35 PB0300
15	75 " " "	25 PB1710
16	65 " " "	35 PB1710
17	65 KRATON D1111 Rubber	35 DP1560
18	65 " " "	35 PB0300
19	65 " " "	35 PB1710
20	65 KRATON D1112 Rubber	35 DP1560
21	65 " " "	35 PB0300
22	65 " " "	35 PB1710
23	65 KRATON D1117 Rubber	35 DP1560
24	65 " " "	35 PB0300
25	65 " " "	35 PB1710

KRATON G1726X rubber is an SEBS triblock copolymer with a 30 wt% styrene content.

KRATON D1102 rubber is an SBS triblock copolymer with a 28 wt% styrene content.

KRATON D1107 rubber is an SIS triblock copolymer with a 14 wt% styrene content.

KRATON D1111 rubber is an SIS triblock copolymer with a 21 wt% styrene content.

KRATON D1112 rubber is an SIS triblock copolymer with a 14 wt% styrene content.

KRATON D1117 rubber is an SIS triblock copolymer with a 17 wt% styrene content.

DP1560 is a butene-1-ethylene copolymer containing 0.75% ethylene with a melt index of 4.0 which has been blended with 20 wt% polypropylene.

PB1710 is a butene-1-ethylene copolymer containing 0.75% ethylene and having a melt index of 1.0 which has been blended with 7 wt% polypropylene.

PB0300 is a homopolymer polybutylene with a melt index of 4.0. All of the above polymers are available from Shell Chemical Company.

("KRATON" is a Registered Trade Mark)

Except for the 100% KRATON rubber blends (Nos. 4, 9 and 12) the blends were melt compounded using a 4.4 cm (1½ inch) Brabender extruder equipped with a two stage screw containing a Maddox mixing section at the end of the first 5 stage. The extrusion conditions are listed below in Tables 2 and 2A.

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TABLE 2

FORMULATIONS	1	2	3	5	6	7*	8*	10*	11*	13	14
ZONE 1 TEMP., °C	190	190	190	180	180	170	170	170	170	170	170
" 2 " °C	200	200	200	190	190	170	170	170	180	170	180
" 3 " °C	200	200	200	190	190	170	170	170	180	170	170
" 4 " °C	200	200	200	190	180	170	170	170	170	180	180
MELT " °C	206	210	210	204	195	180	180	180	187	200	195
RPM	76	70	70	70	63	125	125	125	123	98	91
BACK PRESSURE kg	204	204	204	159	181	408	408	408	454	318	272
(lbs.)	(450)	(450)	(450)	(350)	(400)	(900)	(900)	(900)	(1000)	(700)	(600)
CUTTER FEED SETTING	80	80	80	75	40	2	2	2	2	2	2
BLADE SPEED SETTING	90	90	90	90		70					

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* Used Cumberland pelletizer instead of "Farrs" customized pelletizer.

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TABLE 2A

FORMULATIONS	15	16	17	18	19	20	21	22	23	24	25
ZONE 1 TEMP., °C	170	170	170	170	170	170	170	170	170	170	170
" 2 " °C	170	170	170	170	170	180	170	170	170	170	170
" 3 " °C	170	170	170	170	170	180	170	170	170	170	170
" 4 " °C	180	180	180	180	180	180	180	180	180	180	180
MELT " °C	196	195	200	200	200	204	205	205	199	203	203
RPM	80	80	125	125	125	90	110	110	102	105	108
BACK PRESSURE kg	272	272	227	318	408	272	272	272	227	227	227
(lbs.)	(600)	(600)	(500)	(700)	(900)	(600)	(600)	(600)	(500)	(500)	(500)
CUTTER* FEED SET	2	2	3	3	3	3	3	3	2.5	2.5	2.5

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* Used Cumberland Pelletizer.

All compounding extrusions were made using an 80/20 screen pack and a single strand die. The strand was double strung in a water bath so as to provide additional cooling time.

5 An approximately 0.0254 mm (1 mil) thick layer of the various blends was coextruded onto a 0.076 mm (3 mil) thick film of polypropylene 5C08, available from the Shell Chemical Company, using a three layer die. Polypropylene was extruded in both the Brabender and Haake extruders.

10 The blends were extruded in the Killion extruder. Table 3 shows the extrusion conditions used for the polypropylene film extruded in the Brabender and Haake extruders and each of the blends which was extruded in the Killion extruder.

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TABLE 3

EXTRUSION CONDITIONS

FORMULATION	1	2	3	4	5	6	7	8	9	10	11	12
BRABENDER ZONE 1, °C	190	190	190	190	190	190	190	190	190	190	190	190
" " 2, °C	210	210	210	210	210	210	210	210	210	210	210	210
" " 3, °C	"	"	"	"	"	"	"	"	"	"	"	"
" " 4, °C	"	"	"	"	"	"	"	"	"	"	"	"
" DIE, °C	"	"	"	"	"	"	"	"	"	"	"	"
" ADAPTER, °C	"	"	"	"	"	"	"	"	"	"	"	"
" RPM	29	29	29	29	29	29	29	29	29	29	29	29
" BACK PRES., kg	453	453	453	453	453	453	453	453	453	453	453	453
(lbs.)	(1000)	(1000)	(1000)	(1000)	(1000)	(1000)	(1000)	(1000)	(1000)	(1000)	(1000)	(1000)
HAAKE ZONE 1, °C	180	180	180	180	180	180	180	180	180	180	180	180
" " 2, °C	210	210	210	210	210	210	210	210	210	210	210	210
" " 3, °C	"	"	"	"	"	"	"	"	"	"	"	"
" ADAPTER, °C	"	"	"	"	"	"	"	"	"	"	"	"
" RPM	76	76	76	76	76	76	76	76	76	76	76	76
" BACK PRES., kg	408	408	403	408	408	408	408	408	408	408	408	408
(lbs.)	(900)	(900)	(900)	(900)	(900)	(900)	(900)	(900)	(900)	(900)	(900)	(900)
KILLION ZONE 1, °C	177	177	177	177	177	177	177	177	177	177	177	177
(°F)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)
" " 2, °C	204	204	204	204	204	204	182	177	177	177	177	177
(°F)	(400)	(400)	(400)	(400)	(400)	(400)	(360)	(350)	(350)	(350)	(350)	(350)
" " 3, °C	207	207	207	207	207	207	182	204	204	204	204	204
(°F)	(405)	(405)	(405)	(405)	(405)	(405)	(360)	(400)	(400)	(400)	(400)	(400)
" ADAPTER, °C	204	204	204	204	204	204	188	191	191	191	191	191
(°F)	(400)	(400)	(400)	(400)	(400)	(400)	(370)	(375)	(375)	(375)	(375)	(375)
" RPM	23	24	24	27	27	27	30	42	39	42	42	45
" AMPS	3.4	3.5	3.5	4.2	5.8	4.0	7.2	5.9	5.9	3.5	6.0	7.0
" MELT, °C	218	216	214	218	219	225	225	224	196	202	193	202
(°F)	(425)	(420)	(418)	(425)	(427)	(437)	(437)	(435)	(385)	(395)	(380)	(395)
" BACK PRES., kg	181	227	227	272	318	318	318	340	340	318	318	499
(lbs.)	(400)	(500)	(500)	(600)	(700)	(700)	(700)	(750)	(750)	(700)	(700)	(1100)

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Chill water temperature was set at 15°C, and an air knife was used for all runs.

Depending on film thickness measurement the chill roll speed was adjusted between 0.0975 m/s (19.2 ft/min) and 0.107 m/s (21.0 ft/min.)

Formulations # 4, 9 and 12 pellets were very sticky and required hand-feeding of the pellets into the Killion extruder feed section.

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TABLE 3A

EXTRUSION CONDITIONS

FORMULATION	13	14	15	16	17	18	19	20	21	22	23	24	25
BRABENDER ZONE 1, °C	190	190	190	190	190	190	190	190	190	190	190	190	190
" " 2, °C	210	210	210	210	210	210	210	210	210	210	210	210	210
" " 3, °C	"	"	"	"	"	"	"	"	"	"	"	"	"
" " 4, °C	"	"	"	"	"	"	"	"	"	"	"	"	"
" DIE, °C	"	"	"	"	"	"	"	"	"	"	"	"	"
" ADAPTER, °C	"	"	"	"	"	"	"	"	"	"	"	"	"
" RPM	29	29	29	29	29	29	29	29	29	29	29	29	29
" BACK PRES., kg (lbs.)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)	453 (1000)
HAAKE ZONE 1, °C	180	180	180	180	180	180	180	180	180	180	180	180	180
" " 2, °C	210	210	210	210	210	210	210	210	210	210	210	210	210
" " 3, °C	"	"	"	"	"	"	"	"	"	"	"	"	"
" ADAPTER, °C	"	"	"	"	"	"	"	"	"	"	"	"	"
" RPM	76	76	76	76	76	76	76	76	76	76	76	76	76
" BACK PRES., kg (lbs.)	408 (900)	408 (900)	408 (900)	408 (900)	177 (350)	177 (350)	177 (350)	177 (350)	177 (350)	177 (350)	177 (350)	177 (350)	177 (350)
KILLION ZONE 1, °C	177	177	177	177	177	177	177	177	177	177	177	177	177
" (°F)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)
" " 2, °C	204	204	204	204	177	177	177	177	177	177	177	177	177
" (°F)	(400)	(400)	(400)	(400)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)
" " 3, °C	204	204	204	204	177	177	177	177	177	177	177	177	177
" (°F)	(400)	(400)	(400)	(400)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)	(350)
" ADAPTER, °C	204	204	204	204	204	204	204	210	210	210	210	210	210
" (°F)	(400)	(400)	(400)	(400)	(400)	(400)	(400)	(410)	(410)	(410)	(410)	(410)	(410)
" RPM	28	28	30	30	20	20	20	20	20	20	20	22	20
" AMPS	4.2	5.0	3.5	4.0	6.0	4.2	6.2	3.0	3.0	3.0	3.0	2.8	2.5
" MELT, °C	218	218	216	214	218	216	221	232	232	232	232	232	235
" (°F)	(425)	(425)	(420)	(418)	(425)	(420)	(430)	(450)	(450)	(450)	(450)	(450)	(455)
" BACK PRES., kg (lbs.)	227 (500)	272 (600)	318 (700)	318 (700)	363 (800)	363 (800)	431 (950)	318 (700)	318 (700)	318 (700)	177 (350)	149 (300)	232 (250)

Chill water set at 15°C, and an air knife was used for all runs.

Depending on film thickness measurement the chill roll speed was adjusted between 0.0808 m/s (15.9 ft/min) and 0.0838 m/s (16.5 ft/min.)



Each of the coated films were heat sealed to 0.13 mm (5 mil) thick high impact polystyrene (HIPS) film. All seals were made on a Packaging Industries, Inc. (Sentinel Machinery Div. Model No. 12-1249) heat sealer fitted with 2.54 cm (1 inch) wide jaws. Except for the sealing temperature all seals were made using "standard conditions" of 0.28 MPa (40 psi) air pressure and a duration of $\frac{1}{2}$ second. The sealing temperatures were varied over a range of 177 to 232°C (350°F to 450°F).

10 The term "peel sealability" refers to the sealed area of a film where the two parts are joined together. A film's peel sealability is measured by the amount of force which must be applied to peel the layers apart, such sealability can be measured by ASTM tensile strength
15 testing method D-882 which was used in the Examples.

TABLE 4

PEEL SEAL STRENGTH TO HIPS Kg/2.54 cm (LB/IN)

SEALING TEMPERATURE

	BLEND NO.	177°C (350°F)	204°F (400°F)	232°C (450°F)
5	1	-----	0.616 (1.36)	0.670 (1.48)
	2	-----	0.453 (1.48)	1.065 (2.35)
	3	-----	-----	0.870 (1.92)
	4	0.453 (0.70)	0.589 (1.30)	0.458 (1.01)
10	5	0.326 (0.72)	0.331 (0.73)	0.503 (1.11)
	6	0.575 (1.27)	0.657 (1.45)	0.811 (1.79)
15	7	0.140 (0.31)	0.258 (0.57)	0.824 (1.82)
	8	0.317 (0.70)	0.911 (2.01)	1.572 (3.47)
	9	0.589 (1.30)	1.395 (3.08)	1.459 (3.22)
20	10	1.327 (2.93)	1.237 (2.73)	1.604 (3.54)
	11	0.784 (1.73)	0.775 (1.71)	1.101 (2.45)
	12	0.938 (2.07)	1.386 (3.06)	1.572 (3.47)
25	13	0.983 (2.17)	1.069 (2.36)	1.132 (2.50)
	14	0.172 (0.38)	0.666 (1.47)	0.417 (0.92)
	15	0.725 (1.60)	0.947 (2.09)	1.191 (2.63)
	16	0.494 (1.09)	1.241 (2.74)	1.196 (2.64)
30	17	0.498 (1.10)	0.652 (1.44)	0.883 (1.95)
	18	0.091 (0.20)	0.630 (1.39)	0.974 (2.15)
	19	0.045 (0.10)	0.045 (0.10)	0.181 (0.40)
	20	0.231 (0.51)	0.344 (0.76)	0.421 (0.93)
35	21	0.236 (0.52)	0.313 (0.69)	0.435 (0.96)
	22	0.267 (0.59)	0.322 (0.71)	0.467 (1.03)
	23	0.199 (0.44)	0.299 (0.66)	0.349 (0.77)
	24	0.657 (1.45)	0.322 (0.71)	0.602 (1.33)
	25	0.199 (0.44)	0.344 (0.76)	0.453 (1.00)

Samples made from Blends Nos. 6 and 10 were also heat sealed onto 0.254 mm (10 mil) plasticized clear PVC, HIPS, polypropylene homopolymer and high density polyethylene sheets under the above-mentioned sealing conditions. The peel seal strengths measured are shown in Tables 5 and 6.

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TABLE 5

PEEL SEAL STRENGTH OF BLEND NO.6 kg/2.54 cm (LB/IN)

HEAT SEAL TEMP. °C (°F)	163 (325)	177 (350)	191 (375)	204 (400)	218 (425)
onto HIPS	0.65 (1.44)	0.54 (1.19)	0.63 (1.39)	0.72 (1.58)	1.00 (2.21)
onto PVC	0.99 (2.19)	1.20 (2.65)	1.51 (3.34)	1.45 (3.22)	—
onto HDPE	—	1.08 (2.38)	1.30 (2.88)	3.07 (6.78)	—
onto PP	—	2.25 (4.96)	2.29 (5.06)	2.58 (5.69)	—

TABLE 6

PEEL SEAL STRENGTH OF BLEND NO.10 kg/2.54 cm (LB/IN)

HEAT SEAL TEMP. °C (°F)	163 (325)	177 (350)	191 (375)	204 (400)	218 (425)
onto HIPS	1.48 (3.27)	1.54 (3.39)	1.27 (2.81)	1.20 (2.65)	1.73 (3.83)
onto PVC	1.36 (3.01)	1.39 (3.07)	2.34 (5.17)	2.84 (6.27)	—
onto HDPE	—	1.63 (3.59)	1.99 (4.39)	2.31 (5.09)	—
onto PP	—	1.62 (3.57)	2.20 (4.85)	2.05 (4.52)	—

All seal strength data obtained on the experimental formulations significantly exceeded the minimum target value typical for commercial lidding stock.

In summary, it has been unexpectedly found that a blend
5 formulated in accordance with the invention can be used to provide good peel seal strength, has good processability and will bond by extrusion lamination to a polystyrene, PVC or olefinic rigid container in a laminar structure without the need for an adhesive to achieve bonding.

The claims defining the invention are as follows:

1. A polymer blend for use in providing a peelable heat seal between substrates which comprises
 - (a) 5 to 50 weight percent of a butene-1 homopolymer or copolymer having a melt index of 0.01 to 1000;
 - 5 (b) 95 to 50 weight percent of a styrenic-olefinic block copolymer; and
 - (c) 0 to 30 percent by weight of polypropylene, the weight percentages of (a), (b) and (c) being based on their combined weight.
- 10 2. A blend according to claim 1 which contains from 10 to 40 percent by weight of (a); and from 90 to 60 percent by weight of (b).
3. A blend according to claim 1 or 2 wherein said butene-1 homopolymer or copolymer is a crystalline
- 15 isotactic polymer having a melt ^{Flow}index of 0.4 to 20 (ASTM D 1236-62 Te).
4. A blend according to claim 3 wherein said butene-1 homopolymer or copolymer is a high molecular weight, flexible, polymer having a density of about 0.91 g/cm³.
- 20 5. A blend according to any one of the preceding claims wherein the styrenic-olefinic block copolymer (b) is an A-B-A block copolymer wherein the A blocks are of a monoalkenyl arene polymer and the B block is of a homopolymeric conjugated diene.
- 25 6. A blend according to claim 5, wherein the B block is of polybutadiene or polyisoprene.



7. A blend according to claim 5 or 6, wherein the A block is of polystyrene or poly(α -methylstyrene).

8. A blend according to any one of the preceding claims, wherein the styrenic-olefinic block copolymer (b)
5 has been hydrogenated so that at least 80% of the aliphatic unsaturation is hydrogenated but no more than 25% of the aromatic unsaturation is hydrogenated.

9. A blend according to claim 1, substantially as hereinbefore described with reference to the Examples.

10 10. A laminar structure comprising a coextrudate of a backing material and a blend as claimed in any one of the preceding claims.

11. A laminar structure comprising a backing material coated with a blend as claimed in any one of
15 claims 1 to 9.

12. A laminar structure according to claim 10 or 11, wherein said backing material is selected from polystyrene, polypropylene film, aluminum foil, nylon film, polyester film, polycarbonate film, poly-4-methyl pentene-1
20 film and paper.

13. A peelably sealed rigid package comprising a rigid container having a lid sealed thereon with a film comprising a blend as claimed in any one of claims 1 to 9 or laminar structure as claimed in claim 10, 11 or 12.

25 14. A package according to claim 13 wherein the rigid container is of a styrenic polymer, nylon polyester, polyvinyl chloride or an olefinic polymer.



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15. The herein described invention in all its new
and useful aspects.

DATED this TWENTY SIXTH day of FEBRUARY 1991

Shell Oil Company

by DAVIES & COLLISON
Patent Attorneys for the applicant(s)

