

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property
Organization
International Bureau

(43) International Publication Date
21 June 2018 (21.06.2018)



(10) International Publication Number
WO 2018/109747 A1

(51) International Patent Classification:
C08G 18/40 (2006.01)

(21) International Application Number:
PCT/IB2017/058027

(22) International Filing Date:
16 December 2017 (16.12.2017)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
201621043153 17 December 2016 (17.12.2016) IN

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(81) Designated States (unless otherwise indicated, for every
kind of national protection available): AE, AG, AL, AM,
AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ,
CA, CH, CL, CN, CO, CR, CU, CZ, DE, DJ, DK, DM, DO,
DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN,
HR, HU, ID, IL, IN, IR, IS, JO, JP, KE, KG, KH, KN, KP,
KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME,
MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ,
OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA,
SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN,
TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every
kind of regional protection available): ARIPO (BW, GH,
GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ,

UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ,
TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK,
EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV,
MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM,
TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW,
KM, ML, MR, NE, SN, TD, TG).

Declarations under Rule 4.17:

- as to the identity of the inventor (Rule 4.17(i))
- of inventorship (Rule 4.17(iv))

Published:

- with international search report (Art. 21(3))

(54) Title: A BARRIER FILM AND PROCESS OF PREPARATION THEREOF

(57) Abstract: The present disclosure relates to a barrier film and a process of preparation thereof. The barrier film comprises at least one layer of solid state compacted, hot stretched ultra-high molecular weight polyethylene. The barrier film has high barrier performance and high strength with thickness in the range of 5.0 μ to 300 μ . The barrier film has low permeation rates for oxygen, nitrogen and moisture vapor. The barrier film also has high tensile strength and tensile modulus. The barrier film of the present disclosure finds application in the areas of food and electronic goods packaging requiring high barrier for gas and moisture.

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A BARRIER FILM AND PROCESS OF PREPARATION THEREOF

FIELD

The present disclosure relates to a barrier film.

DEFINITIONS

As used in the present disclosure, the following terms are generally intended to have the
5 meaning as set forth below, except to the extent that the context in which they are used indicate otherwise.

Ultra-high molecular weight polyethylene (UHMWPE): Ultra-high molecular weight polyethylene is a polyethylene with a viscosity average molecular weight (molecular weight) in the range of 0.5 million g mol⁻¹ to 15 million g mol⁻¹, bulk density in the range of 0.03 g
10 cm⁻³ to 0.2 g cm⁻³, crystallinity in the range of 85% to 97% and melt temperature in the range of 141 °C to 145 °C.

A barrier film can be a monolayer barrier film or a multilayer barrier film. A monolayer barrier film comprises a single layer of solid-state, hot stretched ultra-high molecular weight polyethylene. A multilayer barrier film comprises multiple layers of solid-state compacted,
15 hot stretched ultra-high molecular weight polyethylene.

Solid State compaction: Solid state compaction is a compaction process of a polymeric material below its melt temperature.

Barrier Performance: Barrier performance of a polymeric film means its ability to resist permeation of small molecules like O₂, N₂, CO₂, moisture, organic fluid and other gases such
20 as inert gases. The barrier performance is assessed in terms of permeation rates of O₂, N₂, CO₂, moisture, organic fluid and other gases such as inert gases across the film.

Machine direction: Machine direction is a direction parallel to the direction of the input and the output in a calendering machine.

Stretching ratio in the machine direction: Stretching ratio in the machine direction is the
25 relative change in the length of a film caused by stretching in the machine direction.

Stretching ratio transverse to the machine direction: Stretching ratio transverse to the machine direction is the relative change in the width of a film caused by stretching transverse to the machine direction at an angle in the range of 1° to 179°.

BACKGROUND

- 5 Plastic products are widely used for general purpose and engineering applications, due to their light weight, ease of processability and an excellent strength-to-weight ratio. There has been an increasing demand for plastic products with properties that comprise odor/fragrance barrier and resistant to the permeation of gases and liquids, especially, in the field of packaging material for food and other industrial products.
- 10 The barrier performance of a polymeric film means its ability to resist permeation of small molecules like O₂, N₂, CO₂, moisture, organic fluid and other gases such as inert gases. Permeation rate is the volume or mass of small molecules passing through a plastic film of certain thickness at a given pressure, temperature and humidity conditions, per unit time per unit area. A low permeation rate of a small molecule through a barrier film indicates better
- 15 barrier performance of the film, thereby making it a suitable candidate for specific applications. As an example, a film of a polymeric material having a low oxygen permeation rate is an excellent candidate for use as a packaging material for food products that are prone to decomposition in the presence of oxygen. Similarly, a low CO₂ permeation rate is needed for a polymeric film to be used as a material for packaging carbonated beverages.
- 20 In addition to high barrier performance, it is required for a barrier film to have good mechanical properties like high tensile strength and high tensile modulus, so as to prolong the life of the barrier film and avoid any damage.

Conventionally, in order to achieve a combination of properties like, high gas permeation resistance, low moisture permeation, good mechanical properties, good optical clarity and

25 high sealing strength, different types of polymeric materials are used and multi-layered laminates are produced. However, multi-layered laminates in the form of thin films made from different polymeric materials are difficult to manufacture and require expensive machinery. The skill required to produce such laminates is also very high as the melt rheological properties of the polymeric materials vary with their type. Moreover, a barrier

30 film derived from a combination of polymeric materials is difficult to process and recycle.

There is, therefore, felt a need to develop a barrier film having high barrier performance and high strength, and it is desired that the barrier film overcomes the above mentioned drawbacks.

OBJECTS

- 5 Some of the objects of the present disclosure, which at least one embodiment herein satisfies, are as follows:

It is an object of the present disclosure to ameliorate one or more problems of the prior art or to at least provide a useful alternative.

- 10 An object of the present disclosure is to provide a barrier film having high barrier performance.

Another object of the present disclosure is to provide a barrier film having high strength.

Still another object of the present disclosure is to provide a multilayer barrier film made from a single polymeric material.

- 15 Still another object of the present disclosure is to provide a barrier film which is easy to manufacture, process and recycle.

Still another object of the present disclosure is to provide a process for preparing a barrier film having high barrier performance and high strength.

Other objects and advantages of the present disclosure will be more apparent from the following description, which is not intended to limit the scope of the present disclosure.

20 SUMMARY

- In one aspect of the present disclosure, a barrier film comprising at least one layer of solid state compacted, hot stretched ultra-high molecular weight polyethylene is provided. The ultra-high molecular weight polyethylene has molecular weight in the range of 0.5 million g.mol⁻¹ to 15 million g.mol⁻¹, bulk density in the range of 0.03 g cm⁻³ to 0.2 g.cm⁻³ and
25 crystallinity in the range of 85% to 97%, wherein the barrier film has a thickness in the range of 5.0 μ to 300 μ.

Typically, the barrier film comprises at least one layer of solid state compacted, hot stretched ultra-high molecular weight polyethylene having molecular weight in the range of 3.5 million g.mol⁻¹ to 5.5 million g.mol⁻¹, bulk density in the range of 0.05 g cm⁻³ to 0.1 g.cm⁻³ and crystallinity in the range of 90% to 95%, wherein the barrier film has a thickness in the range of 35 μ to 250 μ.

In accordance with the embodiments of the present disclosure, the barrier film has an oxygen permeation rate in the range of 1.0 cm³.m⁻².day⁻¹ to 75 cm³.m⁻².day⁻¹, nitrogen permeation rate in the range of 1.0 cm³.m⁻².day⁻¹ to 20 cm³.m⁻².day⁻¹, moisture vapor permeation rate in the range of 0.01 g.m⁻².day⁻¹ to 0.7 g.m⁻².day⁻¹, tensile strength in the range of 500 kg.cm⁻² to 2500 kg.cm⁻² and tensile modulus in the range of 1000 kg.cm⁻² to 40000 kg.cm⁻².

Typically, the barrier film has an oxygen permeation rate in the range of 40 cm³.m⁻².day⁻¹ to 55 cm³.m⁻².day⁻¹, nitrogen permeation rate in the range of 8.0 cm³.m⁻².day⁻¹ to 12 cm³.m⁻².day⁻¹, moisture vapor permeation rate in the range of 0.2 g.m⁻².day⁻¹ to 0.6 g.m⁻².day⁻¹, tensile strength in the range of 1000 kg.cm⁻² to 2100 kg.cm⁻² and tensile modulus in the range of 20000 kg.cm⁻² to 30000 kg.cm⁻².

In another aspect of the present disclosure, the barrier film is a multilayer barrier film comprising at least two layers of solid state compacted, hot stretched ultra-high molecular weight polyethylene.

Typically, at least two layers of solid state compacted ultra-high molecular weight polyethylene are stacked one on the top of the other in the machine direction, fused and hot stretched.

Typically, the ultra-high molecular weight polyethylene comprises at least one stabilizer in an amount in the range of 1000 ppm to 8000 ppm.

Typically, the stabilizer is selected from the group consisting of substituted phenols, amines, alkyl phosphites/phosponites, aryl phosphites/phosponites, alkyl-aryl phosphites/phosponites, alkyl phosphates, aryl phosphates, alkyl-aryl phosphates, lactones, thioesters, thio compounds containing oxidizable sulphur and aryl nitroso compounds,

Preferably, the stabilizer is selected from the group consisting of tetrakis(2,4-di-tert-butylphenyl)-4, 4'-bisphenylene diphosponite, bis(2,4-di-t-butylphenyl)pentaerythritol diphosphate, tris(2,4-di-t-butylphenyl)phosphate, bis(2,2,6,6-tetramethyl-4-

piperidinyl)sebacate, 2,4,6-tri-*t*-butylphenyl 2-butyl-2-ethyl-1,3-propanediol phosphate,
 octadecyl 3,5 di-*tert*-butyl-4-hydroxyhydrocinnamate, 5,7-di-*tert*-butyl-3-(3,4-
 dimethylphenyl)-3H-benzofuran-2-one, pentaerythritol tetrakis(3,5-di-*tert*-butyl-4-
 hydroxyhydrocinnamate), 5,7-di-*t*-butyl-3-(3,4-dimethylphenyl)-3H-benzofuran-2-one,
 5 tetrakis(methylene (3,5-di-*t*-butyl-4-hydroxyhydrocinnamate)methane and
 disterythiodropinate.

In yet another aspect of the present disclosure, a process for preparing the barrier film is provided. The process comprises the following steps:

- 10 (a) solid state compacting ultra-high molecular weight polyethylene having molecular weight in the range of 0.5 million g.mol⁻¹ to 15 million g.mol⁻¹, bulk density in the range of 0.03 g.cm⁻³ to 0.2 g.cm⁻³ and crystallinity in the range of 85% to 97%, at a first predetermined temperature to obtain a layer of solid state compacted ultra-high molecular weight polyethylene, wherein the layer has a thickness in the range of 100 μ to 2000 μ; and
- 15 (b) hot stretching the layer of solid state compacted ultra-high molecular weight polyethylene in the machine direction and hot stretching transverse to the machine direction at an angle in the range of 1° to 179° at a second predetermined temperature to obtain a barrier film having thickness in the range of 5.0 μ to 300 μ.
- 20 Typically, the step of solid state compacting comprises a process selected from the group consisting of compression molding and calendaring.

Typically, the first predetermined temperature and the second predetermined temperature are temperatures independently selected from temperatures greater than 100° C and less than the melt temperature of the ultra-high molecular weight polyethylene.

- 25 Typically, in step (b), the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are achieved in batch mode or continuous mode.

- 30 Typically, in step (b), the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done sequentially.

Typically, in step (b), the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done simultaneously.

Typically, the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done iteratively.

Typically, in step (b), the ratio of stretching ratio in the machine direction to the stretching ratio transverse to the machine direction at an angle in the range of 1° to 179° is in the range of 1:0.11 to 1:1.

In yet another aspect of the present disclosure, a process for preparing the barrier film is provided, wherein the barrier film is a multilayer barrier film. The process comprises the following steps:

- obtaining at least two layers of solid state compacted ultra-high molecular weight polyethylene by solid state compacting ultra-high molecular weight polyethylene at the first predetermined temperature;
- stacking the at least two layers of solid state compacted ultra-high molecular weight polyethylene in the machine direction to obtain a stacked layer of solid state compacted ultra-high molecular weight polyethylene;
- fusing the stacked layer by passing the stacked layer through a pair of calendar rolls at a temperature greater than 100°C and less than the melt temperature of the ultra-high molecular weight polyethylene to obtain a fused layer of solid state compacted ultra-high molecular weight polyethylene; and
- hot stretching the fused layer in the machine direction and hot stretching transverse to the machine direction at an angle in the range of 1° to 179° at the second predetermined temperature to obtain a multilayer barrier film having thickness in the range of $5.0\text{ }\mu$ to $300\text{ }\mu$.

DETAILED DESCRIPTION

An ideal polymeric barrier film requires a combination of properties such as gas and liquid permeation resistance, low moisture permeation, good mechanical properties like high tensile strength and high tensile modulus, optical clarity and good sealing strength which are critical for packaging applications. In order to prepare barrier film having this combination of properties, several multilayered laminates comprising layers prepared from different

polymers are prepared and used in such packaging applications. However, such multilayered laminates prepared from different polymers are difficult to manufacture and require expensive machinery. Therefore, it is desirable to obtain a barrier film having these desired properties, and it is desired that the barrier film is easy to manufacture, process and recycle.

- 5 The present disclosure, therefore envisages a barrier film having high barrier performance and high strength.

In one aspect, the present disclosure provides a barrier film comprising at least one layer of solid state compacted, hot stretched ultra-high molecular weight polyethylene having molecular weight in the range of 0.5 million g.mol⁻¹ to 15 million g.mol⁻¹, bulk density in the
10 range of 0.03 g cm⁻³ to 0.2 g.cm⁻³ and crystallinity in the range of 85% to 97%, wherein the barrier film has a thickness in the range of 5.0 μ to 300 μ.

Typically, the barrier film comprises at least one layer of solid state compacted, hot stretched ultra-high molecular weight polyethylene having molecular weight in the range of 3.5 million g.mol⁻¹ to 5.5 million g.mol⁻¹, bulk density in the range of 0.05 g cm⁻³ to 0.1 g.cm⁻³ and
15 crystallinity in the range of 90% to 95%, wherein the barrier film has a thickness in the range of 35 μ to 250 μ.

In accordance with the embodiments of the present disclosure, the barrier film has an oxygen permeation rate in the range of 1.0 cm³.m⁻².day⁻¹ to 75 cm³.m⁻².day⁻¹, nitrogen permeation rate in the range of 1.0 cm³.m⁻².day⁻¹ to 20 cm³.m⁻².day⁻¹, moisture vapor permeation rate in
20 the range of 0.01 g.m⁻².day⁻¹ to 0.7 g.m⁻².day⁻¹, tensile strength in the range of 500 kg.cm⁻² to 2500 kg.cm⁻² and tensile modulus in the range of 1000 kg.cm⁻² to 40000 kg.cm⁻².

Typically, the barrier film has an oxygen permeation rate in the range of 40 cm³.m⁻².day⁻¹ to 55 cm³.m⁻².day⁻¹, nitrogen permeation rate in the range of 8.0 cm³.m⁻².day⁻¹ to 12 cm³.m⁻².day⁻¹, moisture vapor permeation rate in the range of 0.2 g.m⁻².day⁻¹ to 0.6 g.m⁻².day⁻¹,
25 tensile strength in the range of 1000 kg.cm⁻² to 2100 kg.cm⁻² and tensile modulus in the range of 20000 kg.cm⁻² to 30000 kg.cm⁻².

In an embodiment, the oxygen permeation rate of the barrier film is 53.4 cm³ m⁻² day⁻¹. In another embodiment, the oxygen permeation rate of the barrier film is 45 cm³ m⁻² day⁻¹.

In an embodiment, the barrier film has a nitrogen permeation rate of 10.7 cm³ m⁻².day⁻¹. In
30 another embodiment, the barrier film has a nitrogen permeation rate of 9.0 cm³ m⁻².day⁻¹.

In an embodiment, the barrier film has a moisture vapor permeation rate of $0.5 \text{ g m}^{-2} \text{ day}^{-1}$. In another embodiment, the barrier film has a moisture vapor permeation rate of $0.3 \text{ g m}^{-2} \text{ day}^{-1}$.

In an embodiment, the crystallinity of the barrier film is in the range of 95 % to 98%.

5 It is observed that the permeation rates for oxygen, nitrogen and moisture of the barrier film are inversely related to the thickness of the barrier film. The thicker barrier films have lower oxygen permeation rate, nitrogen permeation rate and moisture vapor permeation rate than relatively thinner barrier films. Thus, the barrier performance of the barrier film improves with the thickness of the barrier film.

10 It is observed that the tensile strength and tensile modulus of the barrier film are inversely related to the thickness of the barrier film. The thinner barrier films have higher tensile strength and tensile modulus than relatively thicker barrier films as the stretch ratio of a thin film is higher than a thick film. Thus, the mechanical properties of the barrier film improve with decrease in thickness of the barrier films.

15 In an embodiment, the barrier film is a monolayer barrier film comprising a single layer of ultra-high molecular weight polyethylene that is solid state compacted and hot stretched.

In another embodiment, the barrier film is a multilayer barrier film comprising multiple layers of ultra-high molecular weight polyethylene that are solid state compacted and hot stretched.

20 In an embodiment, the barrier film is a multilayer film comprising at least two layers of solid state compacted, hot stretched ultra-high molecular weight polyethylene, wherein the at least two layers of solid state compacted ultra-high molecular weight polyethylene are stacked one on the top of the other in the machine direction, fused and hot stretched.

In an embodiment of the present disclosure, the ultra-high molecular weight polyethylene comprises at least one stabilizer in an amount in the range of 1000 ppm to 8000 ppm.

25 In accordance with the embodiments of the present disclosure, the stabilizer is at least one selected from the group consisting of substituted phenols, amines, alkyl phosphites/phosphonites, aryl phosphites/phosphonites, alkyl-aryl phosphites/phosphonites, alkyl phosphates, aryl phosphates, alkyl-aryl phosphates, lactones, thioesters, thio compounds containing oxidizable sulphur and aryl nitroso compounds.

Preferably, the stabilizer is preferably selected from the group consisting of tetrakis(2,4-di-tert-butylphenyl)-4, 4'-bisphenylene diphosphonite, bis(2,4-di-t-butylphenyl)pentaerythritol diphosphate, tris(2,4-di-t-butylphenyl)phosphate, bis(2,2,6,6-tetramethyl-4-piperidiny)sebacate, 2,4,6-tri-t-butylphenyl 2-butyl-2-ethyl-1,3-propanediol phosphate, octadecyl 3,5 di-tert-butyl-4-hydroxyhydrocinnamate, 5,7-di-tert-butyl-3-(3,4-dimethylphenyl)-3H-benzofuran-2-one, pentaerythritol tetrakis(3,5-di-tert-butyl-4-hydroxyhydrocinnamate), 5,7-di-t-butyl-3-(3,4-dimethylphenyl)-3H-benzofuran-2-one, tetrakismethylene (3,5-di-t-butyl-4-hydroxyhydrocinnamate)methane and disterythiodropinate.

- 10 The stabilizer protects the film from atmospheric degradation that can occur upon contact with agents such as air and light. Thus stabilizers improve the stability of barrier film and extend the material's longevity.

In an embodiment, the ultra-high molecular weight polyethylene comprises at least one filler selected from the group consisting of carbon black, talc, calcium carbonate and carbon nanotubes.

The barrier film prepared without the use of filler is transparent.

In another aspect, the present disclosure provides a process for preparing the barrier film. The process comprises the following steps:

- (a) solid state compacting ultra-high molecular weight polyethylene having molecular weight in the range of 0.5 million g.mol^{-1} to 15 million g.mol^{-1} , bulk density in the range of 0.03 g.cm^{-3} to 0.2 g.cm^{-3} and crystallinity in the range of 85% to 97%, at a first predetermined temperature to obtain a layer of solid state compacted ultra-high molecular weight polyethylene, wherein the layer has a thickness in the range of 100 μ to 2000 μ ; and
- (b) hot stretching the layer of solid state compacted ultra-high molecular weight polyethylene in the machine direction and hot stretching transverse to the machine direction at an angle in the range of 1° to 179° at a second predetermined temperature to obtain a barrier film having thickness in the range of 5.0 μ to 300 μ .

In accordance with the embodiments of the present disclosure, the step of solid state compacting comprises a process selected from the group consisting of compression molding and calendaring.

5 The compression molding is carried out at a pressure in the range of 50 bar to 250 bar. In an exemplary embodiment, the compression molding is carried out at a pressure of 200 bar.

In accordance with the embodiments of the present disclosure, the first predetermined temperature and the second predetermined temperature are temperatures independently selected from temperatures greater than 100° C and less than the melt temperature of the ultra-high molecular weight polyethylene.

10 In one embodiment of the present disclosure, the first predetermined temperature is 128 °C. In another embodiment of the present disclosure, the first predetermined temperature is 125 °C.

In an embodiment of the present disclosure, the second predetermined temperature is 135 °C. In another embodiment of the present disclosure, the second predetermined temperature is 125 °C. In yet another embodiment of the present disclosure, the second predetermined temperature is 138 °C.

20 In an embodiment of the present disclosure, the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° is achieved in batch mode. In another embodiment of the present disclosure, the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° is achieved in continuous mode.

In one embodiment of the present disclosure, the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done sequentially.

25 In another embodiment of the present disclosure, the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done simultaneously.

In an embodiment, the hot stretching is achieved by means of a roller assembly. Other suitable devices can also be used for hot stretching in place of the roller assembly.

In an embodiment, the hot stretching in the machine direction and the hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done iteratively.

Typically, the number of iterations is in the range of 2 to 5 until a barrier film with thickness in the range of $5.0\ \mu$ to $300\ \mu$ is obtained.

- 5 The ratio of stretching ratio in the machine direction to stretching ratio transverse to the machine direction at an angle in the range of 1° to 179° is in the range of 1:0.11 to 1:1.

In yet another aspect of the present disclosure, a process for preparation of the barrier film is provided, wherein the barrier film is multilayer barrier film. The process comprises the following steps:

- 10 • obtaining at least two layers of solid state compacted ultra-high molecular weight polyethylene by solid state compacting ultra-high molecular weight polyethylene at the first predetermined temperature;
- stacking the at least two layers of solid state compacted ultra-high molecular weight polyethylene in the machine direction to obtain a stacked layer of solid state compacted ultra-high molecular weight polyethylene;
- 15 • fusing the stacked layer by passing the stacked layer through a pair of calendar rolls at a temperature greater than 100°C and less than the melt temperature of the ultra-high molecular weight polyethylene to obtain a fused layer of solid state compacted ultra-high molecular weight polyethylene; and
- 20 • hot stretching the fused layer in the machine direction and hot stretching transverse to the machine direction at an angle in the range of 1° to 179° at the second predetermined temperature to obtain a multilayer barrier film having thickness in the range of $5.0\ \mu$ to $300\ \mu$.

25 The present disclosure provides a barrier film having high barrier performance, which is observed from the low permeation rates for oxygen, nitrogen and moisture vapor. The barrier film also displays high strength which is observed from high values of tensile strength and tensile modulus.

Moreover, the barrier film of the present disclosure is prepared from a single polymer i.e. UHMWPE. Due to the use of a single polymer, the barrier film is homogeneous and easy to use. This property is in contrast to the conventional barrier films prepared from two or more

30

polymers. These conventional barrier films are non-homogeneous. Due to homogeneity, the barrier films of the present disclosure are easy to manufacture and recycle as it avoids the use of multiple polymeric materials. The required processing machine and tools for processing film as per the present disclosure are simple and are relatively low cost.

- 5 The present disclosure is further described in light of the following experiments which are set forth for illustration purpose only and not to be construed for limiting the scope of the disclosure. The following experiments can be scaled up to industrial/commercial scale and the results obtained can be extrapolated to industrial scale.

Experiment:

- 10 The present disclosure provides a barrier film and a process of preparation of barrier film from ultra-high molecular weight polyethylene (UHMWPE). The crystallinity of UHMWPE was measured using x-ray technique. The elastic modulus of UHMWPE was measured in melt form, at 180°C by strain controlled dynamic rheometer using parallel plate geometry in time sweep mode. The oxygen and nitrogen permeability of the barrier film were measured
15 by ASTM D1434 and moisture vapor permeation of the barrier film was measured by ASTM F1249. The tensile properties of the barrier film were measured by ASTM D882 and were restricted to the machine direction only.

Experiment 1: Preparation of the barrier film by calendaring

- UHMWPE was subjected to solid state compacting by calendaring. UHMWPE powder
20 having molecular weight (M_v) 5.16 million g mol^{-1} , bulk density of 0.063 g cm^{-3} and crystallinity of 95.2% was homogeneously mixed with pentaerythritol tetrakis(3,5-di-tert-butyl-4-hydroxyhydrocinnamate) (2000 ppm) to obtain UHMWPE mixture. The UHMWPE mixture was subjected to solid state compacting at 128° C by passing through a pair of calendar rolls at a linear speed of 2.69 m/min to obtain a layer of solid state compacted ultra-
25 high molecular weight polyethylene having thickness of 150 μ and width of 160 mm.

- The layer of solid state compacted ultra-high molecular weight polyethylene was then stretched first in the machine direction and then at an angle of 90 ° to the machine direction at roller temperatures of 135° C, and at a linear speed of 3.7 m/min to obtain a barrier film with ratio of stretch as 1:0.33. The roller gap setting was set in such a way the barrier film of
30 thickness 50 μ was obtained.

The barrier film was then analyzed for oxygen permeation rates and nitrogen permeation rates at 0.1 MPa and 23° C, wherein humidity was 0 %. The barrier film was also analyzed for moisture vapor permeation rate test (MVTR) and tensile properties such as tensile strength and tensile modulus.

- 5 It was found that the oxygen gas permeation rate of the barrier film was $53.4 \text{ cm}^3 \cdot \text{m}^{-2} \cdot \text{day}^{-1}$, the nitrogen gas permeation rate was $10.7 \text{ cm}^3 \cdot \text{m}^{-2} \cdot \text{day}^{-1}$ and the moisture vapor permeation rate of the barrier film was $0.5 \text{ g} \cdot \text{m}^{-2} \cdot \text{day}^{-1}$.

The tensile strength and tensile modulus of the barrier film was 1350 kg cm^{-2} and 25000 kg cm^{-2} respectively.

- 10 It is inferred that the barrier film obtained using the process of the present disclosure has good mechanical properties and low permeation rates for oxygen, nitrogen and moisture vapor. Therefore, the barrier film of the present disclosure is a suitable candidate for packaging of food material and electronic goods.

Experiment 2: Preparation of the barrier film by compression molding

- 15 UHMWPE was subjected to solid state compacting by using compression molding. UHMWPE powder having molecular weight of 5.2 million $\text{g} \cdot \text{mol}^{-1}$, bulk density of 0.055 g cm^{-3} and crystallinity of 94.4% was homogeneously mixed with pentaerythritol tetrakis(3,5-di-tert-butyl-4-hydroxyhydrocinnamate) (2000 ppm) to obtain UHMWPE mixture. The UHMWPE mixture was subjected to solid state compacting by compression molding at
20 125°C and a pressure of 200 bar to obtain a layer of solid state compacted ultra-high molecular weight polyethylene having a thickness of 1000μ and diameter of 120 mm.

- The layer of solid state compacted ultra-high molecular weight polyethylene was first hot stretched in the machine direction by feeding the film at the nip of a calendaring roller unit at a temperature of 125°C and a linear roller speed of 0.19 m/min to stretch the film in the
25 machine direction and then hot stretched at an angle of 90° to the machine direction to obtain a unidirectionally stretched film. The unidirectionally stretched film so obtained was then stretched at 90° to the machine direction with the help of another calendaring roller unit at 125°C and a linear roller speed of 0.19 m/min. These steps were repeated 3 times while
30 progressively reducing the gap between the rollers to obtain a barrier film having thickness of 220μ .

The barrier film was then analyzed for oxygen permeation rates and nitrogen permeation rates at 0.1 MPa and 23° C, wherein humidity was 0 %. The barrier film was also analyzed for moisture vapor permeation rate test (MVTR) and tensile properties such as tensile strength and tensile modulus.

- 5 The nitrogen permeation rate of the barrier film was $9.0 \text{ cm}^3 \text{ day}^{-1} \text{ m}^{-2}$ and the oxygen permeation rate of the barrier film was $45 \text{ cm}^3 \text{ day}^{-1} \text{ m}^{-2}$. The moisture vapor permeation rate (MVTR) value was $0.3 \text{ g m}^{-2} \text{ day}^{-1}$.

The tensile strength in the machine direction was 1100 kg cm^{-2} and the tensile modulus in the machine direction was 21000 kg cm^{-2} .

10 **Experiment 3:** Preparation of the barrier film by using two roll mill

- UHMWPE powder having molecular weight of 3.9 million g mole^{-1} , bulk density of 0.058 g cm^{-3} and crystallinity of 91% was homogeneously mixed with pentaerythritol tetrakis(3,5-di-tert-butyl-4-hydroxyhydrocinnamate) (2000 ppm) to obtain UHMWPE mixture. The UHMWPE mixture was subjected to solid state compacting at 128 °C by feeding in the nip of two roll mill at a linear speed of 2.69 m/min to obtain a layer of solid state compacted ultra-high molecular weight polyethylene having thickness of 110 μ and width of 23 cm.

The layer of solid state compacted ultra-high molecular weight polyethylene was fed at 90° in the nip of two roll mill at a temperature of 135 °C maintaining the linear speed as 3.75 m/min to obtain a barrier film having thickness of 40 μ .

- 20 The barrier film was then analyzed for tensile properties such as tensile strength and tensile modulus.

The tensile strength of the barrier film was 2000 kg cm^{-2} and the tensile modulus of the barrier film was 26800 kg cm^{-2} .

Experiment 4: Preparation of multilayer barrier film

- 25 Two different UHMWPE powder mixtures were prepared as detailed out in example 1 and example 3. These two stabilized UHMWPE powder mixtures were subjected to solid state compacting at 125 °C by feeding each UHMWPE mixture separately in the nip of the two roll mill at a linear speed of 2.69m/min to obtain two layers of solid state compacted ultra-high molecular weight polyethylene. These two layers of solid state compacted ultra-high

molecular weight polyethylene were stacked in the machine direction to obtain a stacked layer of solid state compacted ultra-high molecular weight polyethylene. The stacked layer was fused at 135 °C by feeding at 90° in the nip of the rollers which are maintained at a linear speed of 3.75 m/min, to obtain a fused layer of solid state compacted ultra-high molecular weight polyethylene. The fused layer was hot stretched at 138 °C in the machine direction and at an angle of 90° to the machine direction. The hot stretching steps were repeated while reducing the nip gap to obtain a multilayer barrier film having thickness of 50μ.

The multilayer barrier film was then analyzed for tensile properties such as tensile strength and tensile modulus.

The tensile strength of the multilayer barrier film was 1850 kg cm⁻² and the tensile modulus of the multilayer barrier film was 22000 kg cm⁻².

It is observed that the barrier film obtained using the process of the present disclosure has good physical strength and low permeation rates for oxygen, nitrogen and moisture. Therefore, the barrier film of the present disclosure is a suitable candidate for packaging of food material and electronic goods.

TECHNICAL ADVANCEMENTS

The present disclosure described herein above has several technical advantages including, but not limited to, the realization of a barrier film comprising ultra-high molecular weight polyethylene having:

- high barrier performance with low gas and moisture permeation rates; and
- excellent mechanical properties.

Throughout this specification the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the invention to achieve one or more of the desired objects or results. While certain embodiments of the inventions have been described, these embodiments have been presented by way of example only, and

are not intended to limit the scope of the inventions. Variations or modifications to the formulation of this invention, within the scope of the invention, may occur to those skilled in the art upon reviewing the disclosure herein. Such variations or modifications are well within the spirit of this invention.

- 5 The numerical values given for various physical parameters, dimensions and quantities are only approximate values and it is envisaged that the values higher than the numerical value assigned to the physical parameters, dimensions and quantities fall within the scope of the invention unless there is a statement in the specification to the contrary.

- 10 While considerable emphasis has been placed herein on the specific features of the preferred embodiment, it will be appreciated that many additional features can be added and that many changes can be made in the preferred embodiment without departing from the principles of the disclosure. These and other changes in the preferred embodiment of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of
15 the disclosure and not as a limitation.

CLAIMS:

1. A barrier film comprising at least one layer of solid state compacted, hot stretched ultra-high molecular weight polyethylene having molecular weight in the range of 0.5 million g.mol^{-1} to 15 million g.mol^{-1} , bulk density in the range of 0.03 g cm^{-3} to 0.2 g.cm^{-3} and crystallinity in the range of 85% to 97%, wherein said barrier film has a thickness in the range of 5.0μ to 300μ .
2. The barrier film as claimed in claim 1, wherein said barrier film comprises at least one layer of solid state compacted, hot stretched ultra-high molecular weight polyethylene having molecular weight in the range of 3.5 million g.mol^{-1} to 5.5 million g.mol^{-1} , bulk density in the range of 0.05 g cm^{-3} to 0.1 g.cm^{-3} and crystallinity in the range of 90% to 95%, wherein said barrier film has a thickness in the range of 35μ to 250μ .
3. The barrier film as claimed in claim 1, wherein said barrier film has an oxygen permeation rate in the range of $1.0 \text{ cm}^3.\text{m}^{-2}.\text{day}^{-1}$ to $75 \text{ cm}^3.\text{m}^{-2}.\text{day}^{-1}$, nitrogen permeation rate in the range of $1.0 \text{ cm}^3.\text{m}^{-2}.\text{day}^{-1}$ to $20 \text{ cm}^3.\text{m}^{-2}.\text{day}^{-1}$, moisture vapor permeation rate in the range of $0.01 \text{ g.m}^{-2}.\text{day}^{-1}$ to $0.7 \text{ g.m}^{-2}.\text{day}^{-1}$, tensile strength in the range of 500 kg.cm^{-2} to 2500 kg.cm^{-2} and tensile modulus in the range of 1000 kg.cm^{-2} to 40000 kg.cm^{-2} .
4. The barrier film as claimed in claim 1, wherein said barrier film has an oxygen permeation rate in the range of $40 \text{ cm}^3.\text{m}^{-2}.\text{day}^{-1}$ to $55 \text{ cm}^3.\text{m}^{-2}.\text{day}^{-1}$, nitrogen permeation rate in the range of $8.0 \text{ cm}^3.\text{m}^{-2}.\text{day}^{-1}$ to $12 \text{ cm}^3.\text{m}^{-2}.\text{day}^{-1}$, moisture vapor permeation rate in the range of $0.2 \text{ g.m}^{-2}.\text{day}^{-1}$ to $0.6 \text{ g.m}^{-2}.\text{day}^{-1}$, tensile strength in the range of 1000 kg.cm^{-2} to 2100 kg.cm^{-2} and tensile modulus in the range of 20000 kg.cm^{-2} to 30000 kg.cm^{-2} .
5. The barrier film as claimed in claim 1, wherein the barrier film is a multilayer barrier film comprising at least two layers of solid state compacted, hot stretched ultra-high molecular weight polyethylene, wherein said at least two layers of solid state compacted ultra-high molecular weight polyethylene are stacked one on the top of the other in the machine direction, fused and hot stretched.
6. The barrier film as claimed in claim 1, wherein said ultra-high molecular weight polyethylene comprises at least one stabilizer in an amount in the range of 1000 ppm to 8000 ppm, wherein said stabilizer is selected from the group consisting of

- substituted phenols, amines, alkyl phosphites/phosphonites, aryl phosphites/phosphonites, alkyl-aryl phosphites/phosphonites, alkyl phosphates, aryl phosphates, alkyl-aryl phosphates, lactones, thioesters, thio compounds containing oxidizable sulphur and aryl nitroso compounds, wherein said stabilizer is preferably selected from the group consisting of tetrakis(2,4-di-tert-butylphenyl)-4,4'-bisphenylene diphosphonite, bis(2,4-di-t-butylphenyl)pentaerythritol diphosphate, tris(2,4-di-t-butylphenyl)phosphate, bis(2,2,6,6-tetramethyl-4-piperidiny)sebacate, 2,4,6-tri-t-butylphenyl 2-butyl-2-ethyl-1,3-propanediol phosphate, octadecyl 3,5 di-tert-butyl-4-hydroxyhydrocinnamate, 5,7-di-tert-butyl-3-(3,4-dimethylphenyl)-3H-benzofuran-2-one, pentaerythritol tetrakis(3,5-di-tert-butyl-4-hydroxyhydrocinnamate), 5,7-di-t-butyl-3-(3,4-dimethylphenyl)-3H-benzofuran-2-one, tetrakismethylene (3,5-di-t-butyl-4-hydroxyhydrocinnamate)methane and disterythiodropinate.
7. A process for preparing the barrier film as claimed in claim 1, said process comprising the following steps:
- (a) solid state compacting ultra-high molecular weight polyethylene having molecular weight in the range of 0.5 million g.mol^{-1} to 15 million g.mol^{-1} , bulk density in the range of 0.03 g.cm^{-3} to 0.2 g.cm^{-3} and crystallinity in the range of 85% to 97%, at a first predetermined temperature to obtain a layer of solid state compacted ultra-high molecular weight polyethylene, wherein said layer has a thickness in the range of 100 μ to 2000 μ ; and
 - (b) hot stretching said layer of solid state compacted ultra-high molecular weight polyethylene in the machine direction and hot stretching transverse to the machine direction at an angle in the range of 1° to 179° at a second predetermined temperature to obtain a barrier film having thickness in the range of 5.0 μ to 300 μ .
8. The process as claimed in claim 7, wherein said step of solid state compacting comprises a process selected from the group consisting of compression molding and calendaring.
9. The process as claimed in claim 7, wherein said first predetermined temperature and said second predetermined temperature are temperatures independently selected from

temperatures greater than 100° C and less than the melt temperature of the ultra-high molecular weight polyethylene.

10. The process as claimed in claim 7, wherein in step (b), said hot stretching in the machine direction and said hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are achieved in batch mode or continuous mode.
11. The process as claimed in claim 7, wherein in step (b), said hot stretching in the machine direction and said hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done sequentially.
12. The process as claimed in claim 7, wherein in step (b), said hot stretching in the machine direction and said hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done simultaneously.
13. The process as claimed in claim 7, wherein said hot stretching in the machine direction and said hot stretching transverse to the machine direction at the angle in the range of 1° to 179° are done iteratively.
14. The process as claimed in claim 7, wherein in step (b), the ratio of stretching ratio in the machine direction to stretching ratio transverse to the machine direction at an angle in the range of 1° to 179° is in the range of 1:0.11 to 1:1.
15. A process for preparing the barrier film as claimed in claim 5, wherein the barrier film is a multilayer barrier film, said process comprising the following steps:
- obtaining at least two layers of solid state compacted ultra-high molecular weight polyethylene by solid state compacting ultra-high molecular weight polyethylene at the first predetermined temperature;
 - stacking said at least two layers of solid state compacted ultra-high molecular weight polyethylene in the machine direction to obtain a stacked layer of solid state compacted ultra-high molecular weight polyethylene;
 - fusing said stacked layer by passing said stacked layer through a pair of calendar rolls at a temperature greater than 100° C and less than the melt temperature of the ultra-high molecular weight polyethylene to obtain a fused layer of solid state compacted ultra-high molecular weight polyethylene; and

- hot stretching said fused layer in the machine direction and hot stretching transverse to the machine direction at an angle in the range of 1° to 179° at the second predetermined temperature to obtain a multilayer barrier film having thickness in the range of 5.0 μ to 300 μ .

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2017/058027

A. CLASSIFICATION OF SUBJECT MATTER
C08G18/40 Version=2018.01

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C08G

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

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Patseer, IPO Internal Database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP2544841 A1 (CELGARD LLC[US]) 16 January 2013 (2013-01-13) abstract, page 15 lines 1-6, page 17 line 1-5, page 38 lines 30-41, claims 1-6	1-15
Y	EP0550116 A (DSM N.V.[NL]) 07 July 1993 (1993-07-07) abstract, paragraph [0020],[0031]; Claim 9, examples 1 and 2	1-15
Y	EP1497364A1 (DARAMIC INC [US]) 19 January 2005 (2005-01-19) abstract, page 1, claim 1	1-15
Y	JP2938613 B2 (NIPPON OIL CO LTD ET AL.) 23 August 1999 (1999-08-23) abstract, page 1, paragraph [0046], [0052]-[0054], [0073]-0074], [0078], [0086]; claims 1-2	1-15

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:	"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
"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"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	

Date of the actual completion of the international search

16-03-2018

Date of mailing of the international search report

16-03-2018

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/IB2017/058027

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