

Description

This invention relates to stretched multilayer polypropylene films having excellent optical properties and also good heat sealability.

Stretched polypropylene films are widely used in various fields such as food wrapping and fiber wrapping because of their good optical properties (e.g. transparency and gloss), mechanical properties (e.g., tensile strength and stiffness), and moisture proofness.

Single layer-type stretched polypropylene films, however, are very poor in heat sealability, and if heat sealing of such films is attempted at temperatures at which they are heat sealable, heat shrinkage occurs; for practical purposes, therefore, such films are not heat sealable.

Various attempts have been made to overcome the above problem, including a method in which an adhesion layer is locally coated on a stretched polypropylene film constituting a base layer; a method in which an easily heat sealable resin is coated over the entire surface area; a method in which another film is laminated onto a stretched polypropylene film using an intermediate adhesive layer; and a method in which an easily heat sealable resin is laminated on a stretched polypropylene film by melt extrusion. Japanese Patent Publications No. 11353/66, for example, discloses a method in which an ethylene polymer having good heat sealability, such as an ethylene/vinyl acetate copolymer or a low or intermediate density polyethylene, is laminated in film form on at least one side of a stretched polypropylene film. It is known, however, that lamination of such an ethylene polymer gives rise to various problems, particularly in scratch resistance.

Japanese Patent Publication Nos. 31478/71 and 14343/74 disclose a method of producing a film having good scratch resistance and also relatively good heat sealability, in which a crystalline propylene/ethylene copolymer is laminated. It is also known that of crystalline propylene/ethylene copolymers, a propylene/ethylene random copolymer has relatively good transparency and has good heat sealability. The heat sealable temperature of the propylene/ethylene random copolymer drops as the ethylene content is increased. In order to increase the heat sealability of the stretched composite film, it is desirable to increase the ethylene content as much as possible. However, as the ethylene content of the propylene/ethylene random copolymer is increased, the scratch resistance of the film is reduced and, furthermore, antiblocking and sliding properties, which are important properties for films, are impaired. In general, therefore, an antiblocking agent, such as silica, is added to prevent the deterioration of antiblocking properties. The addition of such antiblocking agents, on the other hand, inhibits the optical properties such as transparency and gloss of the film and hence results in impairment of the inherent optical properties of a biaxially stretched polypropylene film. Thus the scope for improving heat sealability is limited.

In order to improve the heat sealability of the propylene/ethylene random copolymer, compound-

ing it with the above-described ethylene/vinyl acetate copolymer or low or intermediate density polyethylene, for example, can be considered. However, the compatibility of the propylene/ethylene random copolymer with the ethylene copolymer is poor. Thus if the ethylene copolymer is compounded even in a small amount, the transparency of the film is seriously reduced.

As a result of extensive investigations to provide good heat sealability without harming good optical properties such as transparency and gloss, which are great features of a stretched polypropylene film, it has been found that a stretched composite polypropylene film having good optical properties such as excellent see-through properties and also good heat sealability can be obtained by laminating on at least one side of a stretched layer of crystalline polypropylene being a homopolymer or a copolymer a layer of a composition (Composition B) comprising a homo- or co-polymer composed mainly of ethylene, propylene or butene-1, said homo- or co-polymer having a melting point of 150°C or less, preferably 150°C to 40°C, and further containing a vinylcycloalkane polymer (Polymer A) in an amount of 1 wt ppm to 10 wt% calculated as a vinylcycloalkane units on at least one side of a stretched crystalline polypropylene layer.

The content of the vinylcycloalkane polymer (Polymer A) in Composition B is not critical. In order not to change the inherent physical properties of Composition B, however it is preferred that the amount of the vinylcycloalkane polymer be as small as possible. For this reason, the content of Polymer A is preferably 0.05 wt ppm to 10 wt% and more preferably 1 wt ppm to 1 wt% calculated as vinylcycloalkane units.

Typical examples of the vinylcycloalkane include vinylcyclopentane, vinylcyclohexane and vinylcyclobornane. Of these compounds vinylcycloalkanes having at least 8 carbon atoms are preferred. Particularly preferred is vinylcyclohexane.

Polymer A is a homo- or co-polymer composed mainly of said vinylcycloalkane. Examples of Polymer A include a homopolymer of vinylcyclohexane, a copolymer of vinylcyclopentane and a small amount of propylene, and a copolymer of vinylcyclohexane and a small amount of vinylcyclopentane.

Composition B containing the vinylcycloalkane polymer can be prepared by, for example, the following methods.

(1) Using a Ziegler-Natta catalyst, a vinylcycloalkane is first polymerized, and subsequently propylene and ethylene are random copolymerized.

(2) The polymer as obtained in (1) above is mixed with a propylene/ethylene random copolymer.

(3) A vinylcycloalkane polymer and a propylene/ethylene random copolymer are mixed.

The formulation of Composition B comprising the homo- or co-polymer composed mainly of ethylene, propylene or butene-1 and having a melting point of 150°C or less is determined according to the degree of heat sealability required. Typical examples are a crystalline propylene/ethylene copolymer containing 10 wt% or less of ethylene, a crystalline propylene/butene-1 copolymer containing 50 wt% or less of butene-1, a propylene/ethylene/butene-1 copolymer

containing 10 wt% or less of ethylene and 50 wt% or less of butene-1, and their mixtures.

The melting point as referred to herein indicates an absorption peak resulting from the fusion of a sample when the sample is raised in temperature at a constant rate (temperature-raising rate: 4°C/min) by the use of a differential scanning calorimeter (DSC) (i.e., a point at which the amount of heat absorption is maximum).

Composition B may be blended with suitable amounts of known polymers such as an ethylene/propylene (EP) rubber.

In the above methods (2) and (3), the mixing process may be carried out by the usual procedure such as by the use of a roll extruder. To Composition B may be added various commonly used additives such as an antioxidant, a lubricant, an antistatic agent, and an antiblocking agent.

The crystalline polypropylene used in the present invention is a propylene polymer having at least 80 wt. of a boiling n-heptane-insoluble matter, an intrinsic viscosity $[\eta]$ of 1.3 to 4.2 dl/g, and containing at least 95 wt% of propylene. A copolymer containing 5 wt% or less of ethylene can be also used as the crystalline polypropylene.

With the crystalline polypropylene may be compounded various commonly used additives as in the case of Composition B.

In order to improve the optical properties, if desired, the vinylcycloalkane polymer (Polymer A) may be added to the crystalline polypropylene layers. Such a layer containing Polymer A can be prepared by the same methods as described for Composition B.

The stretched composite polypropylene film of the present invention can be produced by the following methods.

(1) Crystalline polypropylene as a base material and Composition B are coextruded and laminated and, thereafter, monoaxial stretching and successive or simultaneous biaxial stretching are applied thereto.

(2) Crystalline polypropylene as a base material is extruded in a molten state and monoaxially stretched either in the longitudinal direction or in the vertical direction and, thereafter, Composition B is extruded in a molten state, or alternatively laminated in the state of a solidified film and further stretched in a different direction.

(3) Crystalline polypropylene as a base material is extruded in a molten state and monoaxial stretching and biaxial stretching are applied separately or at the same time and, thereafter, Composition B is extruded in a molten state on the stretched film and laminated.

The degree of stretching in one direction of the crystalline polypropylene layer as a base layer of the stretched composite film of the present invention is 3 to 20 times and preferably 4 to 10 times.

Surface treatment such as a corona discharge treatment and flame treatment may be applied onto the stretched composite film of the present invention by techniques commonly employed on an industrial scale.

The stretched multilayer film of the present inven-

tion exhibits good optical properties, particularly excellent see-through properties as compared with conventional films, while maintaining good heat sealability. Thus the stretched composite film will undoubtedly find many applications in the field of wrapping.

The present invention is described in greater detail with reference to the following Examples, although it is not limited thereto.

The intrinsic viscosity $[\eta]$, melt index, light scattering index (LSI), haze, gloss, heat seal temperature, and melting point were measured by the following methods.

(1) Intrinsic Viscosity $[\eta]$

Measured at 135°C in tetralin by the use of an Ubbelohde's viscometer.

(2) Melt Index

Measured according to JIS K6758.

(3) Light Scattering Index (LSI)

Measured by an LSI tester (receiving scattering transmitting light at 1.2 to 3.6°; manufactured by Toyo Seiki Co., Ltd.). The LSI value was used as a measure of see-through properties because of its good agreement with the see-through properties as examined with the naked eye.

(4) Haze

Measured according to ASTM D1003.

(5) Gloss

Measured according to ASTM D2457.

(6) Heat Seal Temperature

Laminated films were superposed and heat sealed for 2 seconds at a pressure of 2 kg/cm², and a temperature at which a peeling strength (peeling speed: 200 mm/min) in a 25 mm width was 300 g was determined.

(7) Melting Point

Using DSC Model IB (manufactured by Perkin Elmer Co., Ltd.), a sample was preheated at 220°C for 5 minutes and, thereafter, manually lowered to 150°C and then from 150°C to 40°C at a constant rate of 4°C/min, and subsequently from 40°C at a constant rate of 4°C/min. In this way, the melting point was determined.

Example 1

In a flask which had been purged with argon gas were successively introduced 650 ml of dehydrated and purified n-heptane, 94 mmol of diethylaluminum chloride, and 27 g of a titanium trichloride catalyst (produced by Marubeni Solvay Co., Ltd.), and the resulting mixture was heated to 60°C. Subsequently, 40 ml of vinylcyclohexane was added and polymerized for 40 minutes. As a result, a catalyst in which 1.02 g of vinylcyclohexane was polymerized per gram of the titanium trichloride catalyst was obtained.

Using 45.7 g of the above vinylcyclohexane polymer containing catalyst, 180 g of diethylaluminum chloride and 150 l of n-heptane, propylene and ethylene were random copolymerized in a 300-liter stainless steel autoclave under the conditions of ethylene concentration of 2.4 vol%, pressure of 4 kg/cm² G, temperature of 50°C, and hydrogen concentration of 1.5 vol% for 5 hours. After completion of the

polymerization, 30 l of n-butanol was added to stop the polymerization and also to achieve deashing. Then the polymer powder was separated from the solvent by filtration. The dry weight of the polymer powder was 54.7 kg. The amount of the propylene polymerized was 2,350 g per gram of the titanium trichloride catalyst. The intrinsic viscosity $[\eta]$ of the polymer was 2.12 dl/g. The vinylcyclohexane content of the copolymer powder was 434 ppm when determined based on the amount of the polymer per the titanium trichloride catalyst. The ethylene content of the copolymer was 4.0 wt%, and the melting point of the copolymer was 144°C.

A mixture of 20 wt% of the above copolymer (a) and 80 wt% of a propylene/ethylene random copolymer (b) having a melt index of 8.0, an ethylene content of 3.9 wt%, and a melting point of 142°C was prepared. To this mixture were added as stabilizers 0.1 part by weight of calcium stearate and 0.2 part by weight of BHT (2,6-di-tert-butylhydroxytoluene), and 0.05 part by weight of Irganox 1010® (antioxidant produced by Ciba Geigy AG; tetrakis[methylene-3(3',5'-di-tert-butyl-4-hydroxyphenyl)propionate]methane), and the resulting mixture was mixed in a Henschel mixer and then pelletized by the use of a 65 mm ϕ extruder [composition (c)].

A crystalline polypropylene resin (Sumitomo Noblen FS2011D produced by Sumitomo Chemical Co., Ltd.; melt index: 2.6) was melt extruded at a resin temperature of 280°C by the use of a 65 mm ϕ sheet extruder and then cooled and solidified with a cooling roll maintained at 20°C to produce a 0.5 mm-thick sheet.

Composition (c) as prepared above was melt extruded at a resin temperature of 280°C through an extrusion laminator and laminated in a thickness of 50 μ on one side of the above crystalline polypropylene sheet. The laminate thus produced was stretched to 5 times in both the lengthwise and widthwise directions at the same time at a stretching temperature of 150°C by the use of a small-sized, biaxial stretching apparatus to produce a laminated film having a thickness of about 22 μ .

Example 2

A laminated film having a thickness of about 21 μ was produced in the same manner as in Example 1 except that the copolymer (b) was replaced with a propylene/ethylene copolymer having a melt index of 67.5, and ethylene content of 3.3 wt% and a melting point of 145°C.

Comparative Example 1

A laminated film having a thickness of about 22 μ was produced in the same manner as in Example 1 except that the copolymer (a)/copolymer (b) mixture was replaced with the copolymer (b) alone.

Example 3

A laminated film having a thickness of about 28 μ was produced in the same manner as in Example 1 except that the lamination thickness of the composition (c) by the extrusion laminator was changed to 200 μ .

Comparative Example 2

A laminated film having a thickness of about 28 μ was produced in the same manner as in Example 1 except that the copolymer (a)/copolymer (b) mixture was replaced with the copolymer (b) alone and the lamination thickness of the copolymer (b) on the crystalline polypropylene sheet was changed to 200 μ .

Example 4

In a flask which had been purged with argon gas were successively introduced 500 ml of dehydrated and purified n-heptane, 75 mmol of diethylaluminum chloride, and 24.9 g of a titanium trichloride catalyst (produced by marubeni Solvay Co., Ltd.), and the resulting mixture was heated to 60°C. Subsequently, 80 ml of vinylcyclohexane was dropped and polymerized for 150 minutes. As a result, a catalyst in which 4.7 g of vinylcyclohexane was polymerized per gram of the titanium trichloride catalyst was obtained.

Using 22.1 g of the above-prepared vinylcyclohexane polymer-containing catalyst, 180 g of diethylaluminum chloride and 150 l of n-heptane, propylene was polymerized in a 300-liter stainless steel autoclave under the conditions of pressure of 2 kg/cm² G, temperature of 60°C, and hydrogen concentration of 10 vol% for 5 hours. After completion of the polymerization, 30 l of n-butanol was added to stop the polymerization and also to achieve deashing. Then the polymer powder was separated from the solvent by filtration. The dry weight of the polymer powder was 19.2 kg. The amount of the propylene polymerized was 490 g per gram of the titanium trichloride catalyst. The intrinsic viscosity $[\eta]$ of the copolymer powder was 1.49 dl/g. The vinylcyclohexane content of the copolymer powder was 0.95 wt% as calculated from the amount of the polymer per the titanium trichloride.

Then, 1.0 part by weight of the copolymer (d) as obtained above and the same stabilizers and antioxidant was used in Example 1 were added to a propylene/butene-1 copolymer having a melt index of 4.0, a butene-1 content of 18.3 wt%, and a melting point of 139°C, pelletized in the same manner as in Example 1 [composition (e)], composition (e) was then laminated on the same crystalline polypropylene sheet as used in Example 1 to produce an about 22 μ -thick laminated film.

Comparative Example 3

A laminated film was produced in the same manner as in Example 4 except that only the propylene/butene-1 copolymer was laminated on the crystalline polypropylene sheet.

Example 5

To a crystalline polypropylene resin having a melt index of 2.0 were added 0.2 part by weight of the copolymer (d) containing 0.95 wt% of vinylcyclohexane as obtained in Example 4 and 0.1 part by weight of calcium stearate and 0.2 part by weight of BHT as stabilizers and 0.05 part by weight of Irganox 1010®. The resulting mixture was mixed in a Henschel mixer, pelletized by a 65 mm ϕ extruder, melt

extruded at a resin temperature of 280°C by the use of a 65 mm ϕ sheet extruder, and then cooled and solidified with a cooling roll maintained at 20°C to produce a sheet having a thickness of about 0.5 mm.

Then the composition (e) as obtained in Example 4 was laminated and stretched in the same manner as in Example 1 to produce a laminated film having a thickness of about 22 μ , in which both the heat sealability-providing layer and crystalline polypropylene layer contained vinylcyclohexane.

The laminated films as obtained in Examples 1 to 5 and Comparative Examples 1 to 3 were measured for physical properties. The results are shown in Table 1.

It can be seen from Table 1 that the stretched composite films of the present invention (Examples 1 to 4) are greatly improved in optical properties, particularly see-through properties as indicated by the LSI value while maintaining good heat sealability as compared with the conventional films.

TABLE 1
Physical Properties

	Haze (%)	LSI (%)	Gloss (%)	Heat Seal Temperature (°C)
Example 1	1.9	4.0	148	130
» 2	2.0	3.9	143	131
» 3	1.8	5.2	138	135
» 4	2.0	3.5	135	102
» 5	1.7	3.6	137	100
Comparative Example 1	2.0	20.1	152	129
» 2	0.7	36.4	128	134
» 3	1.5	17.4	132	105

Claims

1. A stretched multilayer polypropylene film comprising a stretched layer of crystalline polypropylene which is a homopolymer or a copolymer containing up to 5 wt% of ethylene and a layer of a heat-sealable composition laminated on at least one side of the stretched crystalline polypropylene layer, characterised in that the heat-sealable composition comprises a homo- or co-polymer composed mainly of ethylene, propylene or butene-1 and having a melting point, as determined with a differential scanning calorimeter, of 150°C or less, and from 0.05 wt ppm to 10 wt%, calculated as vinylcycloalkane units, of a vinylcycloalkane polymer being a homopolymer or a copolymer.

2. A film as claimed in Claim 1, wherein said composition comprises a crystalline propylene/ethylene copolymer containing 10 wt% or less of ethylene.

3. A film as claimed in Claim 1, wherein said composition comprises a crystalline propylene/butene-1 copolymer containing 50 wt% or less of butene-1.

4. A film as claimed in Claim 1, wherein said composition comprises a propylene/ethylene/butene-1 random copolymer containing 10 wt% or less of ethylene and 50 wt% or less of butene-1.

5. A film as claimed in any preceding claim, wherein said composition also contains a vinylcycloalkane polymer.

6. A film as claimed in any preceding claim, wherein the vinylcycloalkane polymer is a polymer of vinylcyclopentane, vinylcyclohexane or vinylnorbornane.

Patentansprüche

1. Verstreckte Polypropylen-Mehrschichtfolie, umfassend eine verstreckte Schicht aus kristallinem Polypropylen, das ein Homopolymerisat oder ein Copolymerisat mit bis zu 5 Gew.-% Äthylen ist, und eine Schicht aus einer hitzeverschweißbaren Masse umfaßt, die auf mindestens einer Seite der verstreckten Schicht aus kristallinem Polypropylen laminiert ist, dadurch gekennzeichnet, daß die hitzeverschweißbare Masse ein Homo- oder Copolymerisat umfaßt, das hauptsächlich aus Äthylen, Propylen oder Buten-1 zusammengesetzt ist und einen Schmelzpunkt, bestimmt mit einem Differential-Raster-Calorimeter, von 150°C oder weniger, und 0,05 ppm Gewicht bis 10 Gew.-%, berechnet als Vinylcycloalkan-Einheiten, eines Vinylcycloalkanpolymerisats aufweist, das ein Homopolymerisat oder ein Copolymerisat ist.

2. Folie nach Anspruch 1, in der die Masse ein kristallines Propylen/Äthylen-Copolymerisat mit höchstens 10 Gew.-% Äthylen umfaßt.

3. Folie nach Anspruch 1, in der die Masse ein kristallines Propylen/Buten-Copolymerisat mit höchstens 50 Gew.-% Buten-1 umfaßt.

4. Folie nach Anspruch 1, in der die Masse ein statistisches Propylen/Äthylen/Buten-1-Copolymerisat mit höchstens 10 Gew.-% Äthylen und höchstens 50 Gew.-% Buten-1 umfaßt.

5. Folie nach einem der vorangehenden Ansprüche, in der die Masse auch ein Vinylcycloalkanpolymer enthält.

6. Folie nach einem der vorangehenden Ansprüche, in der das Vinylcycloalkanpolymere ein Polymeres aus Vinylcyclopentan, Vinylcyclohexan oder Vinylnorbornan ist.

Revendications

1. Pellicule de polypropylène à plusieurs couches, étirées, comprenant une couche étirée de polypropylène cristallin qui est un homopolymère ou un copolymère contenant jusqu'à 5% en poids d'éthylène, et une couche d'une composition thermosoudable lamifiée au moins sur une face de la couche de polypropylène cristallin étirée, caractérisée en ce que la composition thermosoudable comprend un homopolymère ou un copolymère constitué principalement d'éthylène, de propylène ou de butène-1 et ayant un point de fusion, déterminé au moyen d'un calorimètre différentiel, de 150°C ou moins, ainsi

extruded at a resin temperature of 280°C by the use of a 65 mm ϕ sheet extruder, and then cooled and solidified with a cooling roll maintained at 20°C to produce a sheet having a thickness of about 0.5 mm.

Then the composition (e) as obtained in Example 4 was laminated and stretched in the same manner as in Example 1 to produce a laminated film having a thickness of about 22 μ , in which both the heat sealability-providing layer and crystalline polypropylene layer contained vinylcyclohexane.

The laminated films as obtained in Examples 1 to 5 and Comparative Examples 1 to 3 were measured for physical properties. The results are shown in Table 1.

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1. A stretched multilayer polypropylene film comprising a stretched layer of crystalline polypropylene which is a homopolymer or a copolymer containing up to 5 wt% of ethylene and a layer of a heat-sealable composition laminated on at least one side of the stretched crystalline polypropylene layer, characterized in that the heat-sealable composition comprises a homo- or co-polymer composed mainly of ethylene, propylene or butene-1 and having a melting point, as determined with a differential scanning calorimeter, of 150°C or less, and from 0.05 wt ppm to 10 wt%, calculated as vinylcycloalkane units, of a vinylcycloalkane polymer being a homopolymer or a copolymer.

2. A film as claimed in Claim 1, wherein said composition comprises a crystalline propylene/ethylene copolymer containing 10 wt% or less of ethylene.

3. A film as claimed in Claim 1, wherein said composition comprises a crystalline propylene/butene-1 copolymer containing 50 wt% or less of butene-1.

4. A film as claimed in Claim 1, wherein said composition comprises a propylene/ethylene/butene-1 random copolymer containing 10 wt% or less of ethylene and 50 wt% or less of butene-1.

5. A film as claimed in any preceding claim, wherein said composition also contains a vinylcycloalkane polymer.

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1. Verstreckte Polypropylen-Mehrschichtfolie, umfassend eine verstreckte Schicht aus kristallinem Polypropylen, das ein Homopolymerisat oder ein Copolymerisat mit bis zu 5 Gew.-% Äthylen ist, und eine Schicht aus einer hitzeverschweißbaren Masse umfaßt, die auf mindestens einer Seite der verstreckten Schicht aus kristallinem Polypropylen laminiert ist, dadurch gekennzeichnet, daß die hitzeverschweißbare Masse ein Homo- oder Copolymerisat umfaßt, das hauptsächlich aus Äthylen, Propylen oder Buten-1 zusammengesetzt ist und einen Schmelzpunkt, bestimmt mit einem Differential-Raster-Calorimeter, von 150°C oder weniger, und 0,05 ppm Gewicht bis 10 Gew.-%, berechnet als Vinylcycloalkan-Einheiten, eines Vinylcycloalkanpolymerisats aufweist, das ein Homopolymerisat oder ein Copolymerisat ist.

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que de 0,05 ppm en poids à 10% en poids, calculés en unités de vinylcycloalcane, d'un polymère de vinylcycloalcane, étant un homopolymère ou un copolymère.

2. Pellicule suivant la revendication 1, caractérisée en ce que la composition comprend un copolymère cristallin de propylène/éthylène contenant 10% en poids ou moins d'éthylène.

3. Pellicule suivant la revendication 1, caractérisée en ce que la composition comprend un copolymère cristallin de propylène/butène-1 contenant 50% en poids ou moins de butène-1.

4. Pellicule suivant la revendication 1, caractérisée en ce que la composition comprend un copolymère statistique de propylène/éthylène/butène-1 contenant 10% en poids ou moins d'éthylène et 50% en poids ou moins de butène-1.

5. Pellicule suivant l'une quelconque des revendications précédentes, caractérisée en ce que la composition contient également un polymère de vinylcycloalcane.

6. Pellicule suivant l'une quelconque des revendications précédentes, caractérisée en ce que le polymère de vinylcycloalcane est un polymère de vinylcyclopentane, vinylcyclohexane ou vinylnorbornane.

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