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

(19) **World Intellectual Property Organization**
International Bureau

(43) **International Publication Date**
15 April 2010 (15.04.2010)

PCT

(10) **International Publication Number**
WO 2010/042335 A1

(51) **International Patent Classification**:
C08J 5/18 (2006.01) ***B61D 39/00*** (2006.01)
A41D 13/00 (2006.01) ***B63H 9/00*** (2006.01)
B32B 27/32 (2006.01) ***B64D 17/00*** (2006.01)
B60R 21/00 (2006.01)

(21) **International Application Number**:
PCT/US2009/058530

(22) **International Filing Date**:
28 September 2009 (28.09.2009)

(25) **Filing Language**: English

(26) **Publication Language**: English

(30) **Priority Data**:
61/104,029 9 October 2008 (09.10.2008) US
61/118,021 26 November 2008 (26.11.2008) US

(71) **Applicant** *(for all designated States except US)*: **DOW GLOBAL TECHNOLOGIES INC.** [US/US]; 2040 Dow Center, Midland, MI 48674 (US).

(72) **Inventors; and**
(75) **Inventors/Applicants** *(for US only)*: **VAN DUN, Jozef** [BE/BE]; Pater Damiaanlaan 76, B-2240 Zandhoven (BE). **MCGEE, Robert** [US/US]; 3606 Mary Jane Drive, Midland, MI 48642 (US). **BATRA, Ashish** [IN/US]; 514 That Way, Apt. 225, Lake Jackson, TX 77566 (US). **DE WINDE, Peter** [NL/NL]; Haven Westzijde 9, NL-4511AR Breskens (NL).

(74) **Agent**: **XU, Hong**; 2030 Dow Center, Midland, Michigan 48674 (US).

(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, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PE, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, 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, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (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, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— *with international search report (Art. 21(3))*

(54) **Title**: POLYOLEFIN FILM HAVING AN IMPROVED ADHESIVE LAYER FOR AIRBAG APPLICATIONS

(57) **Abstract**: The present invention provides a two layer polyolefin film including a polymer barrier layer, and an adhesive layer including a polyolefin grafted with maleic anhydride and ungrafted polyolefins. The adhesive layer has a maleic anhydride surface concentration (cm) from about.025 g/m2 to about.250 g/m2 calculated according to the surface concentration formula cm = fm x fg x fa x pff x tf wherein fm is the weight fraction of the maleic anhydride in the grafted polyolefins, fg is the weight fraction of the grafted polyolefins in the adhesive layer, fa is the weight fraction of the adhesive layer in the film, pf is the density of the film, and tf is the total thickness of the film. The two layer polyolefin film is bonded to synthetic fabric, such as nylon or polyester, and useful for airbag applications.

WO 2010/042335 A1

POLYOLEFIN FILM HAVING AN IMPROVED ADHESIVE LAYER FOR AIRBAG APPLICATIONS

Field of the Invention

[0001] A polyolefin film for adhering to synthetic fabric.

Background of the Invention

[0002] For over ten years, the National Highway Traffic Safety Administration (NHTSA) has required automobiles to include inflatable airbags. In recent years, automobile manufacturers have installed head airbags having a curtain design in addition to traditional airbags. Inflatable airbags typically comprise a synthetic fabric, such as nylon, with a thin barrier layer over the fabric for providing an airtight seal. The barrier layer is designed to have low permeability and enough flexibility to assist in the folding and unfolding of the airbag. The barrier layer must strongly adhere to the synthetic fabric so that it is capable of providing these desirable features. An adhesive coating can be used to bond the barrier layer to the fabric. Adhesion between the barrier layer and fabric has a significant impact on the utility of the airbag.

[0003] Lamination films including a barrier layer and an adhesive for bonding to synthetic fabric exist in the prior art. The adhesive layer of the film has an especially significant impact on the permeability and peel strength, as well as on the thickness, overall strength, flexibility and adhesive strength of the multilayer film. The film compositions are difficult to formulate because each application has unique and specific requirements for the permeability, thickness, overall strength, flexibility, and adhesive strength of the film. For example, multilayer films useful for bonding automotive interior trim applications are generally not adequate for inflatable airbag applications. Existing films are not ideal for adhering to synthetic fabric in airbag applications because they are costly to manufacture, their permeabilities are too high and their peel strengths are too low.

SUMMARY OF THE INVENTION

[0004] The subject invention provides a polyolefin film including an adhesive layer for providing improved adhesion to synthetic fabric, such as nylon, polyester, polyethylene, polypropylene, aramides (such as KEVLAR), or another fibrous fabric.

[0005] The polyolefin film is ideal for inflatable airbag applications. The adhesive layer comprises at least one polyolefin grafted with maleic anhydride. The adhesive layer has a maleic anhydride surface concentration (c_m) from about .025 g/m^2 to about .250 g/m^2. The maleic anhydride surface concentration is calculated according to the surface concentration formula $c_m = f_m \text{ x } f_g \text{ x } f_a \text{ x } \rho_f \text{ x } t_f$ wherein f_m is the weight fraction of the maleic anhydride in the grafted polyolefins, f_g is the weight fraction of the grafted polyolefins in the adhesive layer, f_a is the weight fraction of the adhesive layer in the film, ρ_f is the density of the film and t_f is the total thickness of the film.

[0006] The polyolefin film provides an economical film for inflatable airbag applications having improved adhesive properties including a high peel strength and low permeability, which are ideal for inflatable airbag applications, especially those having a curtain design. The peel strength of the subject invention is greater than about 100 N/m and can be up to about 600 N/m and more. This is a significant improvement over comparative polyolefin films, which typically have peel strengths around 35 N/m. The permeability of the subject invention is about .02 cm^3/sec/cm^2, which is also a significant improvement over comparative polyolefin films, which typically have permeabilities lower than about .1 cm^3/sec/cm^2. The film also provides good hydrolytic and UV stability. When the polyolefin film is bonded to nylon fabric, the reactive chemistry between the nylon and the film can enable a more effective adhesive bond between the film and fabric, compared to existing films non-reactive to nylon fabric, such as thermoplastic polyurethane (TPU), which rely on mechanical interlocking only. The effective bond allows the film to perform better over time and after exposure to aggressive media or weathering, compared to non-reactive films, which can de-bond from the fabric if exposed to moisture. Specifically, the bond contributes to the high peel strength and can reduce the amount of required film penetration depth into the fabric, thus reducing stiffness, thickness, and weight. Also, the structure of the polyolefins allow high levels of additives or fillers, such as an $Mg(OH)_2$ flame retardant, to be added to the film.

[0007] These physical properties make the polyolefin film ideal for inflatable airbag applications, especially those having a curtain design. The film can be used in other automotive airbag applications, such as first impact airbags and knee airbags, located in front of either the driver or passenger. The polyolefin film can also be used in motorcycle airbags, rucksack avalanche airbags, tarpaulins, sails, parachutes, clothing, and architectural fabrics.

[0008] The subject invention also provides a polyolefin film having a long storage life. The film has a melting temperature greater than 105° C, and therefore temperature control and ventilation is not required for storage of the film at temperatures under 70° C. The storage costs of the subject invention are significantly less than existing silicone coating raw materials, which must be stored in sealed containers, away from moisture, and in a ventilated area under 35° C. Also, unlike silicone coating raw materials, which typically have storage limitations of about 15 months, and a life of about only 24 hours when mixed, the film of the subject invention can be stored up to about three years.

[0009] The inventive polyolefin film can be produced with cost effective raw materials and extrusion processes so that the total production costs are generally lower than prior art films.

The polyolefins used in the subject invention are less expensive than TPU, copolyesters, and other exotic materials used in many existing films. Also, the film of the subject invention does not require mixing and curing chemistry in a chemical plant. The film can be applied to the synthetic fabric in one step, such as by an extrusion process. Also, the film can be produced, transported, and stored without expensive packaging, such as drums, that must be returned to a supplier.

[0010] The polyolefin film can also be efficiently recycled. Unlike existing laminates having a silicone coating, which must be stripped of the airbag prior to re-use, the polyolefin film of the subject invention can be recycled without expensive stripping or other preparation. Further, unlike existing TPU films laminated to nylon, which typically form an unstable compound that cannot be re-used, the polyolefins used in the film of the subject invention enable a stable, versatile, and re-usable compound when laminated to nylon.

DETAILED DESCRIPTION OF THE INVENTION

[0011] The polyolefin film includes an adhesive layer for providing improved adhesion to synthetic fabric, such as nylon, polyester, polyethylene, polypropylene, aramides (such as KEVLAR), or another fibrous fabric for inflatable airbag applications. The polyolefin film typically includes two layers, the adhesive layer and a barrier layer.

[0012] The adhesive layer includes at least one polyolefin grafted with maleic anhydride (MAH grafted polyolefin), but can include more than one MAH grafted polyolefin. The polyolefins chosen for the MAH grafted polyolefins preferably comprise copolymers of ethylene and at least one C3 to C10 alpha-olefin. The polyolefins chosen for the MAH grafted polyolefins preferably have melting temperatures greater than 105° C or less than 85° C, tested using the ASTM D 1525 method. At least one of the MAH grafted polyolefins can have a melting temperature higher than the highest temperature of use of the airbag. Each of the MAH grafted polyolefins is elastomeric and can have a glass transition temperature preferably from about -35° C to about -60° C. Each of the MAH grafted polyolefins can have a density preferably from about .860 g/cm^3 to about .910 g/cm^3, tested using the ASTM D 792 method. Maintaining the density below about .910 g/cm^3 allows the glass transition temperature to remain below -35° C. The density of the MAH grafted polyolefins depends in part on whether the polyolefin film is formed with the barrier layer. If the polyolefin film does not include the barrier layer, the adhesive layer can include a barrier function, in which case the MAH grafted polyolefins of the adhesive layer may have a higher density than MAH grafted polyolefins of a film including the barrier layer.

[0013] A preferred MAH grafted polyolefin is OBC-g-MAH, an ethylene-octene block copolymer grafted with .8 wt% MAH, available from The Dow Chemical Company. Another preferred grafted polyolefin is AMPLIFY GR 216, a MAH grafted ethylene octene random copolymer, available from the Dow Chemical Company. Other elastomeric MAH grafted polyolefin materials may also be used, such as FUSABOND MN493D, available from DuPont. Other suitable MAH grafted polyolefins preferably include copolymers of ethylene and at least one C3 to C10 alpha olefin, such as an ethylene-octene block copolymer, an ethylene-octene random copolymer, or an ethylene-butene random copolymer. The most preferred MAH grafted polyolefins include from about .4 wt% to about .8 wt% maleic anhydride (MAH).

[0014] The overall adhesive layer can also be a blend of the MAH grafted polyolefins and other materials. The adhesive layer preferably includes ungrafted polyolefins each having melting temperatures greater than about 85° C, and most preferably greater than 105° C, but the ungrafted polyolefins are not required. The preferred ungrafted polyolefins are elastomeric and have densities from about .86 g/cm^3 to about .91 g/cm^3 and glass transition temperatures from about -35° C to about -60° C. The preferred ungrafted polyolefins include ethylene-octene block copolymers, such as INFUSE 9000 or INFUSE 9500, available from The Dow Chemical Company. Other suitable ungrafted polyolefins include, but are not limited to, copolymers of ethylene and at least one C3 to C10 alpha-olefin, such as ethylene-octene random copolymers and low density polyethylene or linear low density polyethylene. The adhesive layer can also include anti-block additives for preventing the polyolefins from adhering to one another and to processing equipment, and additional stabilization additives for protecting the film during processing or from light and pigments.

[0015] The adhesive layer has a maleic anhydride surface concentration (c_m) from about .025 g/m^2 to about .250 g/m^2 for improving the adhesion of the polyolefin film to the synthetic fabric. An adhesive layer having a maleic anhydride surface concentration (c_m) from about .025 g/m^2 to about .250 g/m^2 allows the film to have a high peel strength, with preferably cohesive failure. When the preferred cohesive failure is observed during peel strength testing, the film will break first, rather than peel off. If the maleic anhydride surface concentration (c_m) is above about .250 g/m^2, the peel strength typically decreases. In addition, if the maleic anhydride surface concentration (c_m) is below about .025 g/m^2 or above about .250 g/m^2, the film typically exhibits adhesive failure, i.e. the polyolefin film will peel off the synthetic fabric without breaking first. Therefore, films having a maleic anhydride surface concentration (c_m) below about .025 g/m^2 or above about .250 g/m^2 are not generally suitable for inflatable airbag applications.

[0016] The maleic anhydride surface concentration (c_m) is calculated according to the surface concentration equation:

$$c_m = f_m \times f_g \times f_a \times \rho_f \times t_f \qquad \textbf{Equation 1}$$

wherein f_m is the weight fraction of the maleic anhydride in the grafted polyolefins, f_g is the weight fraction of the grafted polyolefins in the adhesive layer, f_a is the weight fraction of the adhesive layer in the film, ρ_f is the density of the film, and t_f is the total thickness of the film.

[0017] The weight fraction of the maleic anhydride in the MAH grafted polyolefins can be determined by a Fourier Transform Infrared Spectroscopy (FTIR) Analysis. The weight fraction of the grafted polyolefins in the adhesive layer (f_g), weight fraction of the adhesive layer in the film (f_a), density of the film (ρ_f) and total thickness of the film (t_f) can be determined according to various physical testing procedures and standards known in the field.

[0018] The weight percent of MAH (MAH wt%) can be determined by the ratio of peak heights of the MAH ($FTIR_{MAH}$) and the peak heights of maleic acid ($FTIR_{ma}$) to the peak heights of the polymer reference ($FTIR_{ref}$). The peak heights of MAH are at wave number 1791 cm^{-1}, the peak heights of maleic acid are at 1721 cm^{-1} and the peak heights of polyethylene, which can be used as the polymer reference, are at 2019 cm^{-1}. The ratio of peak heights are multiplied by the appropriate calibration constants (A and B), and the products of the ratios and calibration constants are added together to equal the MAH wt%. When polyethylene is the reference polymer, the MAH wt% is calculated according to the following MAH wt% formula:

$$\text{MAH wt\%} = A\left(\frac{FTIR_{MAH}\ @\,1791cm^{-1}}{FTIR_{ref}\ @\,2019cm^{-1}}\right) + B\left(\frac{FTIR_{ma}\ @\,1721cm^{-1}}{FTIR_{ref}\ @\,2019cm^{-1}}\right) \qquad \textbf{Equation 2}$$

[0019] The calibration constant A can be determined using C13 NMR standards, which are known in the field. The actual calibration constant may differ slightly depending on the instrument and the polymers. The peak heights of maleic acid account for the presence of maleic acid in the polyolefins, which is negligible for freshly grafted polyolefins. However, over time and in the presence of moisture, maleic anhydride is converted to maleic acid. For MAH grafted polyolefins having a high surface area, significant hydrolysis can occur under ambient conditions in just a few days. The calibration constant B in Equation 2 is a correction for the difference in extinction coefficients between the anhydride and acid groups, which can be determined by standards known in the field. The MAH wt% formula takes different sample thicknesses into account to normalize the data.

[0020] A sample of the adhesive layer or MAH grafted polyolefin can be prepared for FTIR analysis in a heating press. The sample of the adhesive layer is about .05 millimeters to about .15 millimeters in thickness and is placed between suitable protective films, such MYLAR™ or TEFLON™, to protect it from the platens of the heating press. Aluminum foil should not be used as a protective film because MAH reacts with aluminum. The sample is then placed in the heating press at about 150-180° C and the platens should be under about 10 tons of pressure for about five minutes. The sample remains in the heating press for about one hour and then is allowed to cool to room temperature before being scanned in the FTIR.

[0021] A background scan should be run on the FTIR before each sample is scanned, or as needed. The sample is placed in an appropriate FTIR sample holder and then scanned in the FTIR. The FTIR will typically display an electronic graph providing the peak heights of MAH at wave number 1791 cm^{-1}, the peak heights of maleic acid at 1721 cm^{-1} and the peak heights of polyethylene at 2019 cm^{-1}. The FTIR test should have an inherent variability less than +/- 5%.

[0022] The adhesive layer including MAH grafted polyolefins and ungrafted polyolefins and anti-block additives preferably has a melting temperature (**T_a**) greater than about 105° C, tested using the ASTM D 3418 method. If the polyolefin film does not include the barrier layer, the adhesive layer can include a barrier function, in which case the MAH grated polyolefins of the adhesive layer may have a higher density than MAH grated polyolefins of a film including the barrier layer. In such a case, the melting temperature of the adhesive layer (**T_a**) should be higher than the highest temperature of use of the airbag to prevent blocking. To achieve the high melting temperature (**T_a**), at least one of the MAH grafted polyolefins can also have a melting temperature higher than the highest temperature of use of the airbag. The adhesive layer can also include an elastomeric MAH grafted polyolefin combined with low-density polyethylene (LDPE) having a melting temperature higher than the melting temperature of the MAH grafted polyolefin.

[0023] The adhesive layer preferably has a melt flow rate (**m_r**) less than about 20 g/min, tested using the ASTM D 1238 method. For airbag applications, the adhesive layer should preferably have an adhesive layer thickness (**t_a**) from about 5 microns to about 100 microns, and most preferably from about 20 microns to about 35 microns. If the polyolefin film includes the barrier layer, the adhesive layer preferably comprises less than about eighty-five percent (85 %) by volume of the polyolefin film. If the film does not include a barrier layer, the adhesive layer preferably comprises a greater percent of the film.

[0024] As alluded to above, the polyolefin film typically includes the barrier layer, which preferably comprises from about 15 % to about 60 % of the volume of the film, and most preferably from about 30 % to about 50 %. The barrier layer comprises at least one polymer, typically a polymer resin. Numerous different polymers or polymer blends can be used for the barrier layer, such as those available from the Dow Chemical company, as long as the polyolefin film has a preferred tensile modulus (2% secant modulus) from about 15 MPa to about 30 MPa, tested using the ASTM D 882 method, and a preferred 100% tensile modulus from about 1 MPa to about 3 MPa, tested using the ASTM D 638 method. The barrier layer should have a high enough melting temperature and low enough tensile modulus so that it can be coextruded with the adhesive layer, laminated onto the synthetic fabric and used for inflatable airbag applications. It should provide optimal strength, flexibility and permeability to the film. The barrier layer prevents blocking between layers of the synthetic fabric and prevents blocking of the film to itself when the airbag is in a rolled-up position and the vehicle is exposed to high temperatures. The barrier layer also prevents excessive penetration of the adhesive layer into the fabric. Films including separate adhesive and barrier layers, not coextruded with one another, require more complex manufacturing processes. The barrier layer can also include additives, such as a flame retardant, pigment, or lubricant.

[0025] The polyolefin film preferably has a total film thickness from about 25 microns to about 100 microns, and most preferably about 50 microns. The film typically has a density from about .893 to about .904 g/cm^3, tested using the ASTM D 792 method. The polyolefin film typically has a melting temperature greater than about 107° C. The film should have a melting temperature greater than the highest temperature of use of the airbag so that the film does not block at the highest temperature. The polyolefin film preferably has a peel strength greater than about 100 N/m at 23° C. The film may exhibit a peel failure mode being adhesive or cohesive, depending on the specific composition of the barrier layer and adhesive layer. The film preferably has a 1% secant tensile modulus from about 200 MPa to about 500 MPa. The film preferably has a permeability from about .015 cm^3/sec/cm^2 to about .100 cm^3/sec/cm^2.

[0026] In an alternative embodiment, the polyolefin film includes at least one additional layer, for example a tie layer or a recycle layer between the barrier layer and adhesive layer. The most preferred tie layers preferably comprise a polyolefin having a density less than about .920 g/cm^3. The most preferred tie layers, for excellent bonding to the adhesive layer and the barrier layer, comprise either linear low density polyethylene, ultra low density polyethylene, or metallocene based polyethylene elastomers. The preferred recycle layers may comprise these same polyolefins combined with excess material from the adhesive layer and barrier layer. The tie layers and recycle layers may reduce the manufacturing costs of the film.

[0027] The polyolefin film can be prepared by extrusion processes known in the field. First, a pelletized form of the adhesive layer can be prepared by a twin screw extruder. If the film is formed without the barrier layer, extrusion coating can be used to apply the adhesive layer to the synthetic fabric. If the polyolefin film includes the barrier layer, the two layer film can be prepared by a blown or cast film extrusion process. The film can then be laminated onto the synthetic fabric, for example nylon 6, 6, polyester, polyethylene, polypropylene, or aramides (such as KEVLAR) with a fusing machine. An advantage of using polyester over nylon as the synthetic fabric is the lower cost of the polyester fibers. However, for the same fiber tenacity, polyester fibers have a smaller diameter fiber than nylon 6,6, which leads to a looser weave at the same strength. The inventive polyolefin film is superior to the traditional silicone coating in retaining the gas content in the airbag. In addition, the MAH of the adhesive layer can form polar bonds with the polyester, ensuring high peel strengths of the film to the polyester fabric.

[0028] The polyolefin film should be stored properly so that the MAH surface concentration remains between about .025 g/m^2 to about .250 g/m^2, which directly impacts the improved peel strength and other preferred properties of the film. Moisture content of the film can be about .1 wt% under ambient conditions, which causes the MAH to convert to maleic acid. Therefore, the film should be stored with a desiccant to prevent excessive hydrolysis. The conversion of anhydride to acid is reversible by placing the polyolefin film in a vacuum oven having a vacuum level near 30 inches of mercury (Hg) at about 150° C. Complete reversion back to maleic anhydride may take up to one week.

Specific Embodiments

[0029] The following examples are given as particular embodiments of the invention and to demonstrate the practice and advantages thereof. The examples are given by way of illustration and are not intended to limit the specification or the claims in any manner.

Example 1

[0030] The adhesive layer includes one MAH grafted polyolefin being an experimental ethylene-octene block copolymer grafted with .8 wt% MAH, such as OBC-g-MAH, available from the Dow Chemical Company. The MAH grafted polyolefin accounts for 20 wt% of the adhesive layer. It has a melt index of 3.1 g/10 minutes at 190° C and 2.16 kg force, tested using the ASTM D 1238 method, density of .866 g/cm^3, tested using the ASTM D 792 method. The ethylene-octene block copolymer on which grafting is carried out has a hard segment wt% of 15 wt% and a soft segment wt% of 85 wt%. The MAH grafted polyolefin also has a peak melting point of 120° C and a glass transition temperature of -60° C.

[0031] The adhesive layer also includes two ungrafted polyolefins. The first ungrafted polyolefin is a commercial ethylene-octene block copolymer, such as INFUSE™ D-9000, available from the Dow Chemical Company. The first ungrafted polyolefin accounts for 60.75 wt% of the adhesive layer. It has a melt index of 0.5 g/10 minutes at 190° C and 2.16 kg force, density of .877 g/cm^3. The first ungrafted polyolefin has a peak melting point of 120° C and a glass transition temperature of -60° C.

[0032] The second ungrafted polyolefin is a commercial low density polyethylene, such as LDPE 662i, available from the Dow Chemical Company. The second ungrafted polyolefin accounts for 14.25 wt% of the adhesive layer. It has a melt index of .47 g/10 minutes at 190° C and 2.16 kg force, density of .919 g/cm^3 and peak melting point of 108° C. The adhesive layer also includes an anti-block additive. The additive is a commercial anti-block master-batch, such as Ampacet 10063, available from the Ampacet Corporation. The adhesive layer has a flexural modulus of about 21.3 MPa when tested on a compression molded part, according to the ASTM D 790 method.

[0033] The adhesive layer is prepared by a twin screw extruder having two weight loss feeders and eight heating zones, such as a WP-ZSK 25, available from Werner and Pfleider. The MAH grafted polyolefin, anti-block additive and second ungrafted polyolefin are dry-blended and fed through a first weight loss feeder. The first ungrafted polyolefin, comprising the majority of the wt% of the adhesive layer, is fed through a second weight loss feeder. The extruder speed is about 500 rpm. The first heating zone is about 140° C and the second through eighth heating zones are about 170° C. The adhesive layer is extruded as a strand and pelletized to form compound pellets upon exiting the extruder.

[0034] Next, a two layer polyolefin film being 50 microns thick and including the adhesive layer is prepared on a small blown film extrusion line, such as a DS 075 HM extruder, available from Davis Standard. The extrusion line has three extruders each having a 46 cm length and a 1.0 cm diameter (L/D 24:1). The film is extruded through a die having a 50.8 mm diameter and a 8.5 mm die gap. The extruders have three controlled heating zones and the die has two heating zones. The three extruders operate simultaneously and have a nip speed of 3.3 m/min.

[0035] Extruder 1 accounts for the barrier layer comprising 30 % of the polyolefin film. The barrier layer includes 100 wt% of random polypropylene resin, such as PP R131-02A, available from the Dow Chemical Company. Extruder 1 operates at a speed of 75 rpm and a pressure of 2095 psi. The three controlled heating zones are at 188° C, 193° C, and 204° C and both die heating zones are at 204° C.

[0036] Extruders 2 and 3 account for the adhesive layer comprising 70 % of the volume of the polyolefin film. Each extruder accounts for 35 % and contains the same pelletized adhesive layer described above. Extruder 2 operates at a speed of 79 rpm and a pressure of 2380 psi. The three controlled heating zones are at 188° C, 193° C, and 204° C and both die heating zones are at 204° C. The melt temperature of the adhesive layer in Extruder 2 is about 211° C. Extruder 3 operates at a speed of 79 rpm and a pressure of 2100 psi. The three controlled heating zones and die heating zones are the same as Extruder 2. The melt temperature of the adhesive layer in Extruder 3 is about 202° C.

[0037] The polyolefin film is laminated onto commercially available synthetic fabric, such as nylon fabric Style 28147-7726, available from Safety Components Inc. The lamination process occurs on a fusing machine, such as an Astex Model 3024, available from Advanced Innovation Technologies LLC. The lamination process includes cutting a sample of the polyolefin film, placing the film onto the fabric and then placing the film and fabric between two fluropolymer coated glass release sheets. The glass sheets are placed between two heated moving belts of the fusing machine with a 30 second dwell time and 30 psi gauge pressure. The film and nylon in the glass sheets are heated to temperatures between 160° C and 177° C. The finished laminates are then prepared for testing. Table 1 provides an overview of testing details. Table 2 provides testing results for the examples.

Example 2

[0038] The adhesive layer includes one MAH grafted polyolefin being a commercial ethylene-octene random copolymer grafted with .8 wt% MAH, such as Amplify™ GR216, available from the Dow Chemical Company. The MAH grafted polyolefin accounts for 20 wt% of the adhesive layer. It has a melt index of 1.25 g/10 minutes at 190° C and 2.16 kg force, density of .870 g/cm^3 and flexural modulus of 14 MPa. The MAH grafted polyolefin also has a peak melting point of 63° C and a glass transition temperature of -53° C.

[0039] The adhesive layer also includes two ungrafted polyolefins. The first ungrafted polyolefin is a commercial ethylene-octene block copolymer, such as OBC D9000, available from the Dow Chemical Company. The first ungrafted polyolefin accounts for 60.75 wt% of the adhesive layer. It has a melt index of .5 g/10 minutes at 190° C and 2.16 kg force, density of .877 g/cm^3 and flexural modulus of 21.3 MPa. The first ungrafted polyolefin has a peak melting point of 120° C and a glass transition temperature of -60° C. The ethylene-octene block copolymer on which grafting is carried out has a 30 wt% hard segment and a 70 wt% soft segment.

[0040] The second ungrafted polyolefin is a commercial low density polyethylene, such as LDPE 662i, available from the Dow Chemical Company. The second ungrafted polyolefin accounts for 14.25 wt% of the adhesive layer. It has a melt index of .47 g/10 minutes at 190° C and 2.16 kg force, density of .919 g/cm^3 and peak melting point of 108° C. The properties are tested according to the test methods disclosed in Example 1.

[0041] The adhesive layer also includes an anti-block additive, accounting for 5 wt% of the adhesive layer. The additive is a commercial anti-block master-batch, such as Ampacet. The adhesive layer, in a pelletized form, is prepared by a twin screw extrusion process, as described in Example 1.

[0042] Next, a two layer polyolefin film being 50 microns thick and including the adhesive layer is prepared on a small blown film extrusion line having three extruders, as described in Example 1. The three extruders operate simultaneously and have a nip speed of 3.3 m/min.

[0043] Extruder 1 accounts for the barrier layer comprising 30 % of the polyolefin film. The barrier layer includes 100 wt% random polypropylene resin, such as PP R131-02A, available from the Dow Chemical Company. Extruder 1 operates at a speed of 75 rpm and a pressure of 1675 psi. The three controlled heating zones are at 188° C, 193° C, and 204° C, and both die heating zones are at 201° C.

[0044] Extruders 2 and 3 account for the adhesive layer comprising 70 % of the film. Each extruder accounts for 35 % and contains the same pelletized adhesive layer described above. Extruder 2 operates at a speed of 79 rpm and a pressure of 2515 psi. The three controlled heating zones are at 188° C, 193° C, and 206° C and both die heating zones are at 204° C. The melt temperature of the adhesive layer in Extruder 2 is about 211° C. Extruder 3 operates at a speed of 79 rpm and a pressure of 2170 psi. The three controlled heating zones are at 188° C, 193° C, and 204° C and both die heating zones are at 204° C. The melt temperature of the adhesive layer in Extruder 2 is about 202° C.

[0045] The polyolefin film is bonded to the fabric on a fusing machine, as described in Example 1. The finished laminates are then prepared for testing. Table 1 provides an overview of testing details. Table 2 provides testing results for the examples.

Example 3

[0046] The adhesive layer includes one MAH grafted polyolefin being a commercial linear low density polyethylene resin grafted with .4 wt% MAH, such as Amplify™ GR-208, available from the Dow Chemical Company. The MAH grafted polyolefin accounts for 100 wt% of the adhesive layer. It has a melt index of 3.3 g/10 minutes at 190° C and 2.16 kg force, density of .902 g/cm^3 and flexural modulus of 90 MPa, tested using the ASTM D 790A method. The MAH grafted polyolefin also has a peak melting point of 117° C and a glass transition temperature of -47° C. The properties are tested according to the test methods disclosed in Example 1.

[0047] The adhesive layer is prepared by a twin screw extruder having two weight loss feeders and eight heating zones, as described in Example 1. The MAH grafted polyolefin is fed through the two weight loss feeders. The extruder speed is about 500 rpm. The first heating zone is about 140° C and the second through eighth heating zones are about 170° C. The adhesive layer is extruded as a strand and pelletized to form compound pellets upon exiting the extruder.

[0048] Next, a two layer polyolefin film being 50 microns thick and including the adhesive layer is prepared on a small blown film extrusion line having three extruders, as described in Example 1. The three extruders operate simultaneously and have a nip speed of 3.3 m/min.

[0049] Extruder 1 accounts for the barrier layer comprising 30 % of the polyolefin film. The barrier layer includes 100 wt% random polypropylene resin, such as PP R131-02A, available from the Dow Chemical Company. Extruder 1 operates at a speed of 75 rpm and a pressure of 1710 psi. The three controlled heating zones are at 188° C, 193° C, and 203° C and both die heating zones are at 203° C.

[0050] Extruders 2 and 3 account for the adhesive layer comprising 70 % of the polyolefin film. Each extruder accounts for 35 % and contains the same pelletized adhesive layer described above. Extruder 2 operates at a speed of 79 rpm and a pressure of 1205 psi. The three controlled heating zones are at 188° C, 193° C, and 203° C and both die heating zones are at 204° C. The melt temperature of the adhesive layer in Extruder 2 is about 210° C. Extruder 3 operates at a speed of 79 rpm and a pressure of 1065 psi. The three controlled heating zones are at 188° C, 193° C, and 204° C, and both die heating zones are at 204° C. The melt temperature of the adhesive layer in Extruder 3 is about 202° C.

[0051] The polyolefin film is bonded to synthetic fabric on a fusing machine, as described in Example 1. The finished laminates are then prepared for testing. Table 1 provides an overview of testing details. Table 2 provides testing results for the examples.

Example 4

[0052] The adhesive layer includes one MAH grafted polyolefin being an experimental ethylene-octene block copolymer grafted with .8 wt% MAH, such as OBC-g-MAH, available from the Dow Chemical Company. The MAH grafted polyolefin accounts for 20 wt% of the adhesive layer. It has a melt index of 3.1 g/10 minutes at 190° C and 2.16 kg force, density of .866 g/cm^3. The MAH grafted polyolefin also has a peak melting point of 120° C and a glass transition temperature of -60° C.

[0053] The adhesive layer also includes two ungrafted polyolefins. The first ungrafted polyolefin is a commercial ethylene-octene block copolymer, such as INFUSE™ D-9000, available from the Dow Chemical Company. The first ungrafted polyolefin accounts for 60.75

wt% of the adhesive layer. It has a melt index of 0.5 g/10 minutes at 190°C and 2.16 kg force, density of .877 g/cm^3 and flexural modulus of 21.3 MPa. The first ungrafted polyolefin has a peak melting point of 120°C and a glass transition temperature of -60°C.

[0054] The second ungrafted polyolefin is a commercial low density polyethylene, such as LDPE 662i, available from the Dow Chemical Company. The second ungrafted polyolefin accounts for 14.25 wt% of the adhesive layer. It has a melt index of .47 g/10 minutes at 190°C and 2.16 kg force, density of .919 g/cm^3 and peak melting point of 108°C. The properties are tested according to the test methods disclosed in Example 1.

[0055] The adhesive layer also includes a commercial anti-block additive, such as Ampacet 10063, accounting for 5 wt% of the adhesive layer. The adhesive layer is prepared by a twin screw extruder having two weight loss feeders and eight heating zones, as described in Example 1. The adhesive layer is extruded as a strand and pelletized to form compound pellets upon exiting the extruder.

[0056] Next, a two layer polyolefin film being 50 microns thick and including the adhesive layer is prepared on a small blown film extrusion line having three extruders, as described in Example 1. The three extruders operate simultaneously and have a nip speed of 3.4 m/min.

[0057] Extruder 1 accounts for the barrier layer comprising 30 % of the polyolefin film. The barrier layer includes 100 wt% random polypropylene resin, such as PP R131-02A, available from the Dow Chemical Company. Extruder 1 operates at a speed of 75 rpm and a pressure of 1495 psi. The three controlled heating zones are at 188°C, 193°C, and 204°C and both die heating zones are at 204° C. Extruders 2 and 3 account for the adhesive layer comprising 70 % of the film. Each extruder accounts for 35 % and contains the same pelletized adhesive layer described above. Extruder 2 operates at a speed of 79 rpm and a pressure of 2335 psi. The three controlled heating zones are at 188° C, 193° C, and 203° C and both die heating zones are at 204° C. The melt temperature of the adhesive layer in Extruder 2 is about 204° C. Extruder 3 operates at a speed of 79 rpm and a pressure of 1890 psi. The three controlled heating zones are at 188° C, 193° C, and 204° C and both die heating zones are at 204° C. The melt temperature of the adhesive layer in Extruder 2 is about 202° C.

[0058] The polyolefin film is bonded to synthetic fabric on a fusing machine, as described in Example 1. The finished laminates are then prepared for testing. Table 1 provides an overview of testing details. Table 2 provides testing results for the examples.

TABLE 1

Test:	**Air Permeability**	**Peel Strength**
Sample No.	5	10 x 2
Directions	Circular	Length Direction (L) & Cross Direction (X)
Dimensions	Clamp Diameter 11 cm	2.5 x 30 cm
Total Surface Area	Diameter 11 x 5 cm	50 x 30 cm
Method No.	ASTM D737:2004	ASTM D1876
Test Equipment	Air Permeability Tester	Tensile Equip. & Suitable Grips, Loadcell 2kN
Test Conditions	Air Pressure 500 Pa Test Surface Area 10 cm^2 Test Temperature 20° C Relative Humidity 65%	Test Speed 200 mm/min Length and cross direction of film/airbag
Measurement	Air pressures is applied on the fabric side, air flow through fabric is measured.	Peel Strength: Avg. of 10 load readings at fixed crosshead distances
Range	passing air/time/surface area	After initial peak
Units	cm^3/sec/cm^2	N

TABLE 2

Example No.	wt% Adh./ wt% Bar.	Adh. Melt Temp. DSC 1st Scan, °C	Film density, g/cm^3	Thickness, microns	MAH g/m^2	Permeability	Peel Strength	Peel Failure Mode
Ex. 1	70/30	120	.903	50	.054	.020	----	cohesive
Ex. 2	70/30	121	.893	50	.057	.034	211	adhesive
Ex. 3	70/30	121	.903	55	.139	.168	----	partly cohesive
Ex. 4	70/30	121	.903	52	.053	.020	109	adhesive

[0059] Obviously, many modifications and variations of the present invention are possible in light of the above teachings and may be practiced otherwise than as specifically described while within the scope of the appended claims. These antecedent recitations should be interpreted to cover any combination in which the inventive novelty exercises its utility.

CLAIMS

What is claimed is:

1. A polyolefin film for adhering to synthetic fabric comprising:

an adhesive layer including at least one polyolefin;

at least one of said polyolefins of said adhesive layer being a grafted polyolefin which includes maleic anhydride; and

said adhesive layer having a maleic anhydride surface concentration (c_m) from about .025 g/m^2 to about .250 g/m^2 calculated according to the surface concentration formula $c_m = f_m \text{ x } f_g \text{ x } f_a \text{ x } \rho_f \text{ x } t_f$ wherein f_m is the weight fraction (wt%) of said maleic anhydride in said grafted polyolefins, f_g is the weight fraction of said grafted polyolefins in said adhesive layer, f_a is the weight fraction of said adhesive layer in said film, ρ_f is the density of said film and t_f is the total thickness of said film.

2. A polyolefin film as set forth in claim 1 wherein said weight fraction (wt%) of said maleic anhydride in at least one of said grafted polyolefins (f_m) is from about .14 wt% to about 1.2 wt%.

3. A polyolefin film as set forth in claim 1 or 2 wherein at least one of said grafted polyolefins has a polyolefin density from about .860 g/cm^3 to about .910 g/cm^3, tested using the ASTM D 792 method.

4. A polyolefin film as set forth in any of claims 1-3 wherein at least one of said grafted polyolefins has a glass transition temperature from about -35° C to about -60° C.

5. A polyolefin film as set forth in any of claims 1-4 wherein at least one of said grafted polyolefins comprises ethylene.

6. A polyolefin film as set forth in claim 5 wherein at least one of said grafted polyolefins comprises a copolymer of ethylene and at least one C3 to C10 alpha-olefin.

7. A polyolefin film as set forth in claim 5 wherein at least one of said grafted polyolefins comprises an ethylene-octene random copolymer.

8. A polyolefin film as set forth in claim 5 wherein at least one of said grafted polyolefins is an ethylene-octene block copolymer.

9. A polyolefin film as set forth in any of claims 1-8 wherein at least one of said grafted polyolefins including maleic anhydride of said adhesive layer has a melting temperature greater than about 105° C or less than 85° C, tested using the ASTM D 1525 method.

10. A polyolefin film as set forth in any of claims 1-9 wherein said adhesive layer includes at least one ungrafted polyolefin.

11. A polyolefin film as set forth in claim 10 wherein at least one of said ungrafted polyolefins comprises ethylene.

12. A polyolefin as set forth in any of claims 1-11 wherein at least one of said ungrafted polyolefins has a melting temperature greater than about 105° C or less than 85° C.

13. A polyolefin film as set forth in claim 1 wherein said adhesive layer has a melting temperature greater than about 105° C.

14. A polyolefin film as set forth in claim 1 wherein said adhesive layer has a melt flow rate less than about 20 g/min.

15. A polyolefin film as set forth in claim 1 wherein said adhesive layer comprises less than about 86 % percent (f_a) of said polyolefin film.

16. A polyolefin film as set forth in any of claims 1-15 wherein said adhesive layer has a thickness from about 5 microns to about 100 microns.

17. A polyolefin film as set forth in claim 1 wherein said adhesive layer includes at least one anti-block additive.

18. A polyolefin film as set forth in claim 1 wherein said peel strength of said polyolefin film is greater than about 100 N/m at 23° C.

19. A polyolefin film as set forth in claim 1 including a barrier layer comprising a polymer resin.

20. A polyolefin film for adhering to synthetic fabric comprising:

an adhesive layer comprising a percent of said polyolefin film (f_a);

said percent of said adhesive layer (f_a) being from about 30 % to about 85 wt%;

said adhesive layer including a grafted polyolefin;

said grafted polyolefin of said adhesive layer comprising ethylene and including maleic anhydride grafted therein;

said grafted polyolefin having a density from about .860 g/cm^3 to about .910 g/cm^3;

said grafted polyolefin having a melting temperature greater than about 105° C;

said adhesive layer having a maleic anhydride surface concentration (**c_m**) from about .025 g/m^2 to about .250 g/m^2 calculated according to the surface concentration formula $c_m = f_m \times f_g \times f_a \times \rho_f \times t_f$ wherein f_m is the weight fraction (wt%) of said maleic anhydride in said grafted polyolefins, f_g is the weight fraction of said grafted polyolefin in said adhesive layer, f_a is the weight fraction of said adhesive layer in said film**,** ρ_f is the density of said film and t_f is the total thickness of said film;

said adhesive layer having a melting temperature greater than about 105° C;

said adhesive layer having a melt flow rate less than about 20 g/min.;

said adhesive layer including an anti-block additive;

a barrier layer comprising from about 15 % to about 30 % of said film;

said barrier layer comprising a polymer resin;

said polyolefin film having a total film thickness being about 50 microns;

said polyolefin film having a permeability from about .0150 $cm^3/sec/cm^2$ to about .2 $cm^3/sec/cm^2$;

said polyolefin film having a 1% tensile modulus from about 200 MPa to about 500 MPa; and

said polyolefin film having a peel strength greater than about 100 N/m at 23° C.

21. A polyolefin film as set forth in claim 20 wherein said weight fraction of said maleic anhydride in said grafted polyolefin is about .8 wt% (f_m).

22. A polyolefin film as set forth in claim 21 wherein,

said grafted polyolefin is an ethylene-octene block copolymer having maleic anhydride grafted therein;

said grafted polyolefin comprises about 20 wt% (f_g) of said adhesive layer;

said grafted polyolefin has a melting index of about 3.1 g/10 minutes at 190° C and 2.16 kg force and a density of about .866 g/cm^3 and a peak melting point of about 120° C and a glass transition temperature of about -60° C;

said adhesive layer further includes a first ungrafted polyolefin comprising an ethylene-octene block copolymer;

said first ungrafted polyolefin comprises about 61 wt% of said adhesive layer;

said first ungrafted polyolefin has a melting index of about .5 g/10 minutes at 190° C and 2.16 kg force and a density of about .877 g/cm^3 and a flexural modulus of about 21.3 MPa and a peak melting point of about 120° C and a glass transition temperature of about -60° C;

said adhesive layer further includes a second ungrafted polyolefin comprising a low density polyethylene;

said second ungrafted polyolefin comprises about 14 wt% of said adhesive layer;

said second ungrafted polyolefin has a melting index of about .47 g/10 minutes at 190° C and 2.16 kg force and a density of about .919 g/cm^3 and a peak melting point of about 108° C; and

said adhesive layer further includes an anti-block additive comprising about 5 wt% of said adhesive layer.

23. A polyolefin film as set forth in claim 20 wherein said weight fraction of said maleic anhydride in said grafted polyolefin is about .4 wt% (f_m).

24. A polyolefin film as set forth in claim 20 wherein said adhesive layer further comprises at least one ungrafted polyolefin.

25. A polyolefin film as set forth in claim 24 wherein at least one of said ungrafted polyolefins comprises a copolymer of ethylene and at least one C3 to C10 alpha-olefin.

26. A polyolefin film as set forth in claim 24 wherein at least one of said ungrafted polyolefins comprises an ethylene-octene block copolymer.

27. A polyolefin film as in any of claims 1-26 including a tie layer between said adhesive layer and said barrier layer.

28. A polyolefin film as set forth in any of claims 1-26 including a recycle layer between said adhesive layer and said barrier layer.

29. A polyolefin film as set forth in any of claims 1-28 wherein said barrier layer includes a stabilizer.

30. A polyolefin film as set forth in any of claims 1-29 wherein said barrier layer includes an anti-block additive.

31. A polyolefin film as set forth in any of claims 1-30 wherein said barrier layer includes a lubricant.

32. A polyolefin film as set forth in any of claims 1-31 wherein said barrier layer includes and a pigment for coloring said polyolefin film.

33. A polyolefin film as set forth in any of claims 1-15 wherein said polyolefin film has a total thickness from about 40 microns to about 60 microns.

34. A multilayer material including a polyolefin film as set forth in any of claims 1-33 and a fabric layer adhering to said polyolefin film.

35. A multilayer material as set forth in claim 34 wherein said fabric layer includes at least one of a synthetic material and a fibrous material.

36. A multilayer material as set forth in claim 34 wherein said fabric layer includes at least one of nylon, polyester, polyethylene, polypropylene, and aramides.

37. An inflatable airbag including a polyolefin film as set forth in any of claims 1-33.

38. An inflatable airbag including a multilayer material as set forth in any of claims 34-36.

39. An inflatable airbag as set forth in claim 37 or 38 wherein said inflatable airbag is further defined as a curtain airbag for an automotive vehicle.

40. An inflatable airbag as set forth in claim 37 or 38 wherein said inflatable airbag is further defined as a first impact airbag for an automotive vehicle.

41. An inflatable airbag as set forth in claim 37 or 38 wherein said inflatable airbag is further defined as a knee airbag for an automotive vehicle.

42. An inflatable airbag as set forth in claim 37 or 38 wherein said inflatable airbag is further defined as a motorcycle airbag.

43. An inflatable airbag a set forth in claim 37 or 38 wherein said inflatable airbag is further defined as a rucksack avalanche airbag.

44. A tarpaulin material including a polyolefin film as set forth in any of claims 1-33.

45. A tarpaulin material including a multilayer material as set forth in any of claims 34-36.

46. A fabric for catching wind including a polyolefin film as set forth in any of claims 1-33.

47. A fabric for catching wind including a multilayer material as set forth in any of claims 34-36.

48. A parachute including a polyolefin film as set forth in any of claims 1-33.

49. A parachute including a multilayer material as set forth in any of claims 34-36.

50. A clothing fabric including a polyolefin film as set forth in any of claims 1-33.

51. A clothing fabric including a multilayer material as set forth in any of claims 34-36.

52. An architectural fabric including a polyolefin film as set forth in any of claims 1-33.

53. An architectural fabric including a multilayer material as set forth in any of claims 34-36.

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2009/058530

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08J5/18 A41D13/00 B32B27/32 B60R21/00 B61D39/00
B63H9/00 B64D17/00

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)
C08J C08L A41D B32B B60R B61D B63H B64D

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 practical, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	THE DOW CHEMICAL COMPANY: "AMPLIFY GR 208"[Online] May 2006 (2006-05), XP002562164 Retrieved from the Internet: URL:http://www.dow.com/PublishedLiterature/dh_0050/0901b803800501d0.pdf?filepath=amplify/pdfs/noreg/305-02916.pdf&fromPage=GetDoc> [retrieved on 2010-01-07] the whole document -----	1-6
A	GB 2 076 832 A (CELANESE CORP) 9 December 1981 (1981-12-09) page 3, lines 47-57 -----	1-53
	-/--	

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search 8 January 2010	Date of mailing of the international search report 20/01/2010
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer olde Scheper, Bernd

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2009/058530

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 434 386 A (DOW CHEMICAL CO [US]) 26 June 1991 (1991-06-26) claims 1-29; examples 1-6	1-36,52, 53
A		37-45, 48,49
X	US 2002/068182 A1 (KELCH ROBERT H [US] ET AL) 6 June 2002 (2002-06-06)	1-36,52, 53
A	claims 1-19	37-45, 48,49
X	WO 2007/001694 A (EXXONMOBIL CHEM PATENTS INC [US]; TSE MUN FU [US]; LI FENG [US]; MATHE) 4 January 2007 (2007-01-04)	1-36,46, 47,50-53
A	paragraphs [0003], [0191], [0197]; claims 1,41	37-45, 48,49

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2009/058530

Patent document cited in search report		Publication date	Patent family member(s)			Publication date
GB 2076832	A	09-12-1981	BR	8103274	A	16-02-1982
			DE	3120898	A1	01-04-1982
			FR	2483442	A1	04-12-1981
			JP	57018755	A	30-01-1982
EP 0434386	A	26-06-1991	AU	630532	B2	29-10-1992
			AU	6831890	A	27-06-1991
			BR	9006456	A	01-10-1991
			CA	2032851	A1	22-06-1991
			DE	69032993	D1	15-04-1999
			DE	69032993	T2	08-07-1999
			ES	2128298	T3	16-05-1999
			FI	906301	A	22-06-1991
			JP	4305447	A	28-10-1992
			NO	905518	A	24-06-1991
			US	5045401	A	03-09-1991
US 2002068182	A1	06-06-2002	NONE			
WO 2007001694	A	04-01-2007	CN	101248133	A	20-08-2008
			EP	1896537	A1	12-03-2008
			JP	2008546885	T	25-12-2008
			KR	20080031734	A	10-04-2008