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(54) **Title**: REACTIVELY PROCESSED, HIGH HEAT RESISTANT COMPOSITION OF POLYPROPYLENE AND AN OLEFINIC INTERPOLYMER

(57) **Abstract**: A propylene polymer is coupled with an olefinic interpolymer by a process comprising contacting under reactive processing conditions at least: A. 10 wt% of at least one propylene polymer; B. 10 wt% of at least one olefinic interpolymer; C. 35 wt % of at least one metal hydrate; and D. A coupling amount of a coupling agent, each weight percent based on the combined weight of the propylene polymer, olefinic interpolymer and metal hydrate. Wire and cable insulation sheaths made from compositions comprising the coupled polymer exhibit desirable heat resistance.

REACTIVELY PROCESSED, HIGH HEAT RESISTANT COMPOSITION OF POLYPROPYLENE AND AN OLEFINIC INTERPOLYMER

CROSS REFERENCE TO RELATED APPLICATION

The present application claims priority to U.S. patent application serial no. 61/059,356, filed on June 6, 2008, the entire content of which is incorporated by reference herein.

FIELD OF THE INVENTION

[0001] This invention relates to compositions comprising a polypropylene polymer and an olefinic interpolymer. In one aspect, the invention relates to compositions comprising polypropylene and an olefinic interpolymer that have been reactively processed and which display characteristics of high heat resistance suitable for wire and cable coatings. In another aspect, the invention relates to power cables comprising an insulation layer while in still another aspect, the invention relates to a power cable in which the insulation layer comprises a composition comprising polypropylene and an olefinic interpolymer that have been reactively processed.

BACKGROUND OF THE INVENTION

[0002] Polymeric compositions are used extensively as primary insulation materials for wire and cable. As an insulator the composition should exhibit various physical and electrical properties, such as heat resistance, resistance to mechanical cut through, stress crack resistance and dielectric failure. Insulation materials for electric conductors often require crosslinking to achieve the desired heat resistance.

[0003] Crosslinks can be introduced between different molecular chains of a polymer by a number of mechanisms, one of which is to graft to the individual polymer backbones or chains that constitute the bulk polymer a chemically reactive compound in such a manner that the grafted compound on one backbone may subsequently react with a similar grafted compound on another backbone thus forming the crosslink. Exemplary of this process is the "silane crosslinking" process.

[0004] The silane crosslinking process employs a silane-containing compound that crosslinks the polymer molecules. Silanes can be grafted to a suitable polymer by the use of a suitable quantity of organic peroxide or other free radical initiator, either before or during a shaping or molding operation. Additional ingredients such as stabilizers, pigments, fillers, catalysts, processing aids and the like may also be included in the mixture.

[0005] When using silane-peroxide blends for polymer crosslinking, a compromise must be made between grafting efficiency and process efficiency, such as extrusion rate and run times. The formation of a crosslinkable material by this means is difficult to carry out since it requires critical control of the process. If the process is conducted at too high a temperature, for example, the polymer may partially cross-link in the processing apparatus, e.g., an extruder, with consequent difficulties in achieving a consistently good quality product. Delays in the process may also occur as a result of the need to remove partially crosslinked product from the processing equipment. Care must also be exercised to ensure that articles prepared from the polymer retain their shape during subsequent heating to bring about the crosslinking process.

[0006] Moreover gel formation, screw-build up and scorching may result when using highly reactive silane-peroxide blends. This is particularly significant for processes using conditions and processing equipment that impose severe melting and mixing conditions leading to high shear stresses in the polyolefin. These problems generally arise due to early, and eventually complete, activation of the peroxide during the initial melting and homogenization process. Traditionally, this problem has been dealt with by using less reactive silane blends, but this approach can diminish the grafting efficiency of the crosslinking reaction.

[0007] Another method of crosslinking is the use of radiation. Radiation crosslinking requires complex equipment and is thus relatively costly to perform. Furthermore, radiation can cause polymer degradation by oxidation and/or chain scission thus requiring special stabilization. Furthermore, the sizes of cable that can be handled by commercial radiation equipment are limited, both in terms of jacket thickness and overall diameter of the cable. This limitation is typically manifested as non-uniform crosslinking of the jacket and a

resultant variation in physical properties around the circumference of the cable or within the material wall of the jacket.

[0008] Alternative materials, such as, polyurethane and fluorinated ethylene propylene elastomers, are expensive, and the materials are sensitive to water. In addition, halogens often are required in order to make the materials flame resistant, and this reduces the attractiveness of these compounds.

[0009] Thus, the need for polyolefin compositions that can be extruded without the need for further crosslinking, and that exhibit heat resistance and resistance to ignition and flame spread and, preferably, good flexibility, remain of interest to the wire and cable industry.

SUMMARY OF THE INVENTION

[0010] In one embodiment, the invention relates to compositions comprising a polypropylene polymer that has been reactively processed with an olefinic interpolymer such that the composition exhibits heat resistance and resistance to ignition and flame spread. In addition, the composition may also exhibit good flexibility.

[0011] In one embodiment the invention is a process for coupling a propylene polymer with an olefinic interpolymer, the process comprising contacting under reactive processing conditions at least:

A. 10 wt% of at least one propylene polymer;

B. 10 wt% of at least one olefinic interpolymer;

C. 35 wt% of at least one metal hydrate; and

D. A coupling amount of a coupling agent,

each weight percent based on the combined weight of the propylene polymer, olefinic interpolymer and metal hydrate. Typically the coupling agent is (i) a silane having a vinyl group, or (ii) a poly(azide).

[0012] The polypropylene polymer can be a homopolymer or a copolymer. The olefinic interpolymers include, but are not limited to, very low density polyethylene (VLDPE),

homogeneously branched, linear ethylene/α-olefin copolymers, homogeneously branched, substantially linear ethylene/α-olefin copolymers, linear medium density polyethylene, linear low density polyethylene (LLDPE), ultra low density polyethylene (ULDPE), and multi-block olefin polymers. The metal hydrates used in the present invention include, but are not limited to, aluminum hydroxide and magnesium hydroxide.

[0013] In one embodiment the invention is a cable comprising an insulation layer that comprises a composition comprising a reactively processed polypropylene polymer, olefinic interpolymer and metal hydrate.

DESCRIPTION OF THE PREFERRED EMBODIMENT

[0014] The numerical ranges in this disclosure are approximate, and thus may include values outside of the range unless otherwise indicated. Numerical ranges include all values from and including the lower and the upper values, in increments of one unit, provided that there is a separation of at least two units between any lower value and any higher value. As an example, if a compositional, physical or other property, such as, for example, molecular weight, viscosity, melt index, etc., is from 100 to 1,000, it is intended that all individual values, such as 100, 101, 102, etc., and sub ranges, such as 100 to 144, 155 to 170, 197 to 200, etc., are expressly enumerated. For ranges containing values which are less than one or containing fractional numbers greater than one (e.g., 1.1, 1.5, etc.), one unit is considered to be 0.0001, 0.001, 0.01 or 0.1, as appropriate. For ranges containing single digit numbers less than ten (e.g., 1 to 5), one unit is typically considered to be 0.1. These are only examples of what is specifically intended, and all possible combinations of numerical values between the lowest value and the highest value enumerated, are to be considered to be expressly stated in this disclosure. Numerical ranges are provided within this disclosure for, among other things, the amount of polypropylene, the amount of olefinic copolymer, and the amount of metal hydrate in the composition.

[0015] "Cable," "power cable," and like terms means at least one wire or optical fiber within a protective jacket or sheath. Typically, a cable is two or more wires or optical fibers bound together, typically in a common protective jacket or sheath. The individual wires or fibers inside the jacket may be bare, covered or insulated. Combination cables may contain

both electrical wires and optical fibers. The cable, etc., can be designed for low, medium and high voltage applications.

[0016] "Polymer" means a polymeric compound prepared by polymerizing monomers, whether of the same or a different type. The generic term polymer thus embraces the term homopolymer, usually employed to refer to polymers prepared from only one type of monomer, and the term interpolymer as defined below.

[0017] "Interpolymer", "copolymer" and like terms means a polymer prepared by the polymerization of at least two different types of monomers. These generic terms refer both to polymers prepared from two different types of monomers, and polymers prepared from more than two different types of monomers, e.g., terpolymers, tetrapolymers, etc.

[0018] "Polyolefin", "PO" and like terms mean a polymer derived from simple olefins. Many polyolefins are thermoplastic and for purposes of this invention, can include a rubber phase. Representative polyolefins include polyethylene, polypropylene, polybutene, polyisoprene and their various interpolymers.

[0019] "Blend," "polymer blend" and like terms mean a composition of two or more polymers. Such a blend may or may not be miscible. Such a blend may or may not be phase separated. Such a blend may or may not contain one or more domain configurations, as determined from transmission electron spectroscopy, light scattering, x-ray scattering, and any other method known in the art.

[0020] "Composition", "formulation" and like terms means a mixture or blend of two or more components. In the context of a mix or blend of materials from which a cable sheath or other article of manufacture is fabricated, the composition includes all the components of the mix, e.g., coupled propylene polymer and olefinic interpolymer, metal hydrate, uncoupled polymers and coupling agent, and any other additives such as processing agents, antioxidants, etc.

[0021] "Molecular melt" means an at least partially amorphous blend, at room temperature, of a coupling agent (modifying agent) and an antioxidant, optionally also containing other polymer additives as, for example, described in WO 2003/040229 A1. Both the coupling

agent and the antioxidant are at least partially contained in the amorphous phase of the blend. Also, preferably the coupling agent and the antioxidant form a complex where the Raman spectra relating to the groups forming the nitrene groups are shifted compared to the Raman spectra exhibited by the groups forming the nitrene groups of the coupling agent alone.

[0022] "Coupled" and like terms mean that one polymer strand is joined to another polymer strand by a coupling agent.

[0023] "Coupling agent" and like terms means a chemical compound that contains at least two reactive groups that are each capable of forming a carbene or nitrene group that are capable of inserting into the carbon hydrogen bonds of CH, CH_2, or CH_3 groups, both aliphatic and/or aromatic, of a polymer chain. The reactive groups together can couple or cross-link polymer chains. The coupling agent may require activation with heat, sonic energy, radiation or chemical activating energy, before it can effectively couple polymer chains.

[0024] "Coupling amount" and like terms mean, in the context of this invention, an amount of coupling agent sufficient to couple a propylene polymer and olefinic interpolymer under reactive processing conditions and in the presence of a metal hydrate such that the heat resistance of the composition in the form of a cable or wire insulation sheath is improved over the heat resistance of a similar cable or wire insulation sheath made from a composition alike in all aspects except that the propylene polymer and olefinic interpolymer are not coupled.

[0025] "Nitrene group" means a compound having a structure R--N in which N is nitrogen capable of reacting with a polymer chain by inserting into the carbon hydrogen bonds of CH, CH_2, or CH_3 groups, both aliphatic and/or aromatic, of a polymer chain. The nitrene nitrogen most preferred for inserting into the carbon hydrogen bonds has two lone pairs of electrons. R may be any atom or atoms that do not adversely interfere with the nitrogen inserting into the above-described carbon-hydrogen bonds.

[0026] "Carbene group" means a compound having a structure R--C--R' in which C is carbon capable of reacting with a polymer chain by inserting into the carbon hydrogen bonds

of CH, CH_2 or CH_3 groups, both aliphatic and/or aromatic, of a polymer chain. The carbon most preferred for inserting into the carbon hydrogen bonds has one lone pair of electrons. R and R' are independently any atom or atoms that do not adversely interfere with the carbon inserting into the above-described carbon hydrogen bonds.

[0027] "Antioxidant" means types or classes of chemical compounds that are capable of being used to minimize the oxidation that can occur during the processing of polymers. The term also includes chemical derivatives of the antioxidants. The term further includes chemical compounds as described later in the description of the antioxidant that, when properly combined with the coupling agent, interact with it to form a complex which exhibits a modified Raman spectra compared to the coupling agent alone.

[0028] "Reactive processing" means a method for compatibilization or chemical coupling of blends of polymers by mixing the polymeric components in such a manner that they react with one another *in situ*. The components of the composition are of sufficient reactivity that the reactions can occur across melt-phase boundaries.

[0029] "Reactive processing conditions" means that the blend of polymers is subjected to (1) sufficient mixing to achieve the desired fineness of morphological texture, and (2) reacting, or coupling, at least some of the polymer molecules with one another to form covalent bonds during the mixing/mastication process. The reactions occur rapidly enough such that they are completed during processing in the extruder or mixer within a reasonable time. Typically the processing conditions include a temperature of 100 to 280, more typically 150 to 250 and even more typically 180 to 250, °C. Pressure is typically a function, at least in part, of the equipment in which the polymers are blended, but typically the pressure ranges from atmospheric to a slightly positive pressure. The reactive processing conditions typically proceed until at least 50, more typically at least 70 and even more typically at least 80, percent of the azide has reacted or, in the case of a silane coupling agent, at least 50, more typically at least 70 and even more typically at least 80, percent of the peroxide has been consumed.

Polyolefin Synthesis:

[0030] The polyolefins used in the practice of this invention, specifically including the propylene polymer and the olefinic interpolymer, can be produced using conventional polyolefin polymerization technology, *e.g.*, Ziegler-Natta, metallocene or constrained geometry catalysis, each adapted, of course, for the polyolefin of interest. Metallocene and constrained geometry catalysts (CGC) include mono- or bis-cyclopentadienyl, indenyl, or fluorenyl transition metal (preferably Group 4) complexes in combination with an activator, e.g., an alumoxane. USP 5,064,802, WO93/19104 and WO95/00526 disclose constrained geometry metal complexes and methods for their preparation. Variously substituted indenyl containing metal complexes are taught in WO95/14024 and WO98/49212.

[0031] In general, polymerization can be accomplished at conditions well known in the art for Ziegler-Natta or Kaminsky-Sinn type polymerization reactions, that is, at temperatures from 0-250°C, preferably 30-200°C, and pressures from atmospheric to 10,000 atmospheres (1013 megaPascal (MPa)). Suspension, solution, slurry, gas phase, solid state powder polymerization or other process conditions may be employed as desired. The catalyst can be supported or unsupported, and the composition of the support can vary widely. Silica, alumina or a polymer (especially poly(tetrafluoroethylene) or a polyolefin) are representative supports, and desirably a support is employed when the catalyst is used in a gas phase polymerization process. The support is preferably employed in an amount sufficient to provide a weight ratio of catalyst (based on metal) to support within a range of from 1:100,000 to 1:10, more preferably from 1:50,000 to 1:20, and most preferably from 1:10,000 to 1:30. In most polymerization reactions, the molar ratio of catalyst to polymerizable compounds employed is from 10^{-12}:1 to 10^{-1}:1, more preferably from 10^{-9}:1 to 10^{-5}:1.

[0032] Inert liquids serve as suitable solvents for polymerization. Examples include straight and branched-chain hydrocarbons such as isobutane, butane, pentane, hexane, heptane, octane, and mixtures thereof; cyclic and alicyclic hydrocarbons such as cyclohexane, cycloheptane, methylcyclohexane, methylcycloheptane, and mixtures of two or more of these materials; perfluorinated hydrocarbons such as perfluorinated C_{4-10} alkanes; and aromatic and alkyl-substituted aromatic compounds such as benzene, toluene, xylene, and ethylbenzene.

Propylene Polymer:

[0033] The propylene polymers used in the practice of this invention (component A in the composition) are not the olefin interpolymers (component B of the composition). The propylene polymer may be a propylene homopolymer, or a copolymer of propylene and one or more other olefins, or a blend of two or more homopolymers or two or more copolymers, or a blend of one or more homopolymer with one or more copolymer. The propylene polymers used in the present compositions can vary widely in form and include, for example, substantially isotactic propylene homopolymer, random propylene copolymers, and graft or block propylene copolymers.

[0034] The propylene copolymers typically comprise 90 or more mole percent units derived from propylene. The remainder of the units in the propylene copolymer is derived from units of at least one α-olefin.

[0035] The α-olefin component of the propylene copolymer is preferably ethylene (considered an α-olefin for purposes of this invention) or a C_{4-20} linear, branched or cyclic α-olefin. Examples of C_{4-20} α-olefins include 1-butene, 4-methyl-1-pentene, 1-hexene, 1-octene, 1-decene, 1-dodecene, 1-tetradecene, 1-hexadecene, and 1-octadecene. The α-olefins also can contain a cyclic structure such as cyclohexane or cyclopentane, resulting in an α-olefin such as 3-cyclohexyl-1-propene (allyl cyclohexane) and vinyl cyclohexane. Although not α-olefins in the classical sense of the term, for purposes of this invention certain cyclic olefins, such as norbornene and related olefins, particularly 5-ethylidene-2-norbornene, are α-olefins and can be used in place of some or all of the α-olefins described above. Similarly, styrene and its related olefins (for example, α-methylstyrene, etc.) are α-olefins for purposes of this invention. Illustrative polypropylene copolymers include but are not limited to propylene/ethylene, propylene/1-butene, propylene/1-hexene, propylene/1-octene, and the like. Illustrative terpolymers include ethylene/propylene/1-octene, ethylene/propylene/1-butene, and ethylene/propylene/diene monomer (EPDM). The copolymers can be random or blocky.

[0036] The following are illustrative but non-limiting propylene polymers that can be used in the compositions of this invention: a propylene impact copolymer including but not

limited to DOW Polypropylene T702-12N; a propylene homopolymer including but not limited to DOW Polypropylene H502-25RZ; and a propylene random copolymer including but not limited to DOW Polypropylene R751-12N. The above-mentioned Dow propylene polymers typically have a density of 0.90 g/cm^3 measured using ASTM D792.

[0037] Furthermore, INSPIRE^{tm} D114, which is a branched impact propylene copolymer with a melt flow index of 0.5 dg/min (230°C/2.16kg) and a melting point of 164°C, can be used (also available from The Dow Chemical Company).

[0038] In addition, PROFAX^{tm} SR-256M, which is a clarified propylene copolymer resin with a density of 0.90 g/cc and a MFR of 2 g/10 min, PROFAX^{tm} 8623, which is an impact propylene copolymer resin with a density of 0.90 g/cc and a MFR of 1.5 g/10 min, and CATALLOY^{tm} in-reactor blends of polypropylene (homo- or copolymer) with one or more of propylene-ethylene or ethylene-propylene copolymer can be used (all available from Basell, Elkton, MD). Other propylene polymers include Solvay's KS 4005 propylene copolymer; and Solvay's KS 300 propylene terpolymer.

Olefinic Interpolymers:

[0039] The olefinic interpolymers used in the practice of this invention (component B of the composition) do not include the propylene polymers described above (component A of the composition). The olefinic interpolymers include but are not limited to polyolefin elastomers, polyolefin flexomers, and polyolefin plastomers. Preferably, the olefinic interpolymers are ethylene interpolymers that comprise at least 10, preferably at least 50 and more preferably at least 80, wt% units derived from ethylene based on the weight of the olefinic interpolymer.

[0040] Examples of olefinic interpolymers useful in the practice of this invention include very low density polyethylene (VLDPE), homogeneously branched, linear ethylene/α-olefin copolymers (*e.g.* TAFMER® by Mitsui Petrochemicals Company Limited and EXACT® by DEXPlastomers), and homogeneously branched, substantially linear ethylene/α-olefin polymers (*e.g.*, AFFINITY® polyolefin plastomers and ENGAGE® polyolefin elastomers

available from The Dow Chemical Company). The substantially linear ethylene copolymers are more fully described in USP 5,272,236, 5,278,272 and 5,986,028.

[0041] Other olefinic interpolymers useful in the present invention include heterogeneously branched ethylene-based interpolymers including, but are not limited to, linear medium density polyethylene (LMDPE), linear low density polyethylene (LLDPE), and ultra low density polyethylene (ULDPE). Commercial polymers include DOWLEX™ polymers, ATTANE™ polymer and FLEXOMER™ polymers (all from The Dow Chemical Company), and ESCORENE™ and EXCEED™ polymers (both from Exxon Mobil Chemical).

[0042] Still other olefinic interpolymers include multi-block or segmented copolymers. These are polymers comprising two or more chemically distinct regions or segments (referred to as "blocks") preferably joined in a linear manner, that is, a polymer comprising chemically differentiated units which are joined end-to-end with respect to polymerized ethylenic functionality, rather than in pendent or grafted fashion. In certain embodiments, the blocks differ in the amount or type of comonomer incorporated therein, the density, the amount of crystallinity, the crystallite size attributable to a polymer of such composition, the type or degree of tacticity (isotactic or syndiotactic), regio-regularity or regio-irregularity, the amount of branching, including long chain branching or hyper-branching, the homogeneity, or any other chemical or physical property. The multi-block copolymers are characterized by unique distributions of polydispersity index (PDI or M_w/M_n), block length distribution, and/or block number distribution due to the unique process making of the copolymers. More specifically, when produced in a continuous process, embodiments of the polymers may possess a PDI ranging from about 1.7 to about 8; from about 1.7 to about 3.5 in other embodiments; from about 1.7 to about 2.5 in other embodiments; and from about 1.8 to about 2.5 or from about 1.8 to about 2.1 in yet other embodiments. When produced in a batch or semi-batch process, embodiments of the polymers may possess a PDI ranging from about 1.0 to about 2.9; from about 1.3 to about 2.5 in other embodiments; from about 1.4 to about 2.0 in other embodiments; and from about 1.4 to about 1.8 in yet other embodiments.

[0043] Ethylene/α-olefin multi-block interpolymers comprise ethylene and one or more co-polymerizable α-olefin comonomers in polymerized form, characterized by multiple (i.e.,

two or more) blocks or segments of two or more polymerized monomer units differing in chemical or physical properties (block interpolymer), preferably a multi-block interpolymer. In some embodiments, the multi-block interpolymer may be represented by the following formula:

$$(AB)_n$$

[0044] where n is at least 1, preferably an integer greater than 1, such as 2, 3, 4, 5, 10, 15, 20, 30, 40, 50, 60, 70, 80, 90, 100, or higher; "A" represents a hard block or segment; and "B" represents a soft block or segment. Preferably, A's and B's are linked in a linear fashion, not in a branched or a star fashion. "Hard" segments refer to blocks of polymerized units in which ethylene is present in an amount greater than 95 weight percent in some embodiments, and in other embodiments greater than 98 weight percent. In other words, the comonomer content in the hard segments is less than 5 weight percent in some embodiments, and in other embodiments, less than 2 weight percent of the total weight of the hard segments. In some embodiments, the hard segments comprise all or substantially all ethylene. "Soft" segments, on the other hand, refer to blocks of polymerized units in which the comonomer content is greater than 5 weight percent of the total weight of the soft segments in some embodiments, greater than 8 weight percent, greater than 10 weight percent, or greater than 15 weight percent in various other embodiments. In some embodiments, the comonomer content in the soft segments may be greater than 20 weight percent, greater than 25 eight percent, greater than 30 weight percent, greater than 35 weight percent, greater than 40 weight percent, greater than 45 weight percent, greater than 50 weight percent, or greater than 60 weight percent in various other embodiments.

[0045] The ethylene multi-block copolymers useful in the practice of this invention, and their preparation and use, are more fully described in WO 2005/090427, US2006/0199931, US2006/0199930, US2006/0199914, US2006/0199912, US2006/0199911, US2006/0199910, US2006/0199908, USP 7,355,089, US2006/0199906, US2006/0199905, USP 7,524,911, US2006/0199896, US2006/0199887, USP 7,514,517, US2006/0199872, US2006/0199744, US2006/0199030, USP 7,504,347 and US2006/0199983. Representative

olefin multi-block interpolymers include olefin block copolymers manufactured and sold by The Dow Chemical Company.

[0046] The ethylene interpolymers useful in the present invention include ethylene/α-olefin interpolymers having a α-olefin content typically of at least 5, more typically of at least 15 and even more typically of at least about 20, wt% based on the weight of the interpolymer. These interpolymers typically have an α-olefin content of less than 90, more typically less than 75 and even more typically less than about 50, wt% based on the weight of the interpolymer. The α-olefin content is measured by ^{13}C nuclear magnetic resonance (NMR) spectroscopy using the procedure described in Randall (*Rev. Macromol. Chem. Phys., C29 (2&3)*).

[0047] The α-olefin is preferably a C_{3-20} linear, branched or cyclic α-olefin. Examples of C_{3-20} α-olefins include propene, 1-butene, 4-methyl-1-pentene, 1-hexene, 1-octene, 1-decene, 1-dodecene, 1-tetradecene, 1-hexadecene, and 1-octadecene. The α-olefins also can contain a cyclic structure such as cyclohexane or cyclopentane, resulting in an α-olefin such as 3-cyclohexyl-1-propene (allyl cyclohexane) and vinyl cyclohexane. Although not α-olefins in the classical sense of the term, for purposes of this invention certain cyclic olefins, such as norbornene and related olefins, particularly 5-ethylidene-2-norbornene, are α-olefins and can be used in place of some or all of the α-olefins described above. Similarly, styrene and its related olefins (for example, α-methylstyrene, etc.) are α-olefins for purposes of this invention. Illustrative polyolefin copolymers include ethylene/propylene, ethylene/butene, ethylene/1-hexene, ethylene/1-octene, ethylene/styrene, and the like. Illustrative terpolymers include ethylene/propylene/1-octene, ethylene/propylene/butene, ethylene/butene/1-octene, ethylene/propylene/diene monomer (EPDM) and ethylene/butene/styrene. The copolymers can be random or blocky.

[0048] Additional olefinic interpolymers useful in the practice of this invention include the VERSIFY® propylene-based polymers available from The Dow Chemical Company, and the VISTAMAXX® propylene polymers available from ExxonMobil Chemical Company, at least those VERSIFY® AND VISTAMAXX® propylene polymers with a content of units derived from propylene of less than 85 mol%. A discussion of various other polypropylene

polymers is contained in *Modern Plastics Encyclopedia/89*, mid October 1988 Issue, Volume 65, Number 11, pp. 6-92.

[0049] If a blend of olefinic interpolymers is used in the practice of this invention, then the blend can be made by any in-reactor or post-reactor process. The in-reactor blending processes are preferred to the post-reactor blending processes, and the processes using multiple reactors connected in series are the preferred in-reactor blending processes. These reactors can be charged with the same catalyst but operated at different conditions, *e.g.*, different reactant concentrations, temperatures, pressures, *etc*, or operated at the same conditions but charged with different catalysts, or operated at different conditions and charged with different catalysts.

Metal Hydrate:

[0050] The metal hydrates useful in the practice of this invention include, but are not particularly limited to, for example, compounds having a hydroxyl group or water of crystallization, such as aluminum hydroxide and magnesium hydroxide. These metal hydrates can be used singly or in combination of two or more.

[0051] Examples of commercially available magnesium hydroxide include MAGNIFIN® manufactured by Matinswerk. Other examples include KISUMA 5, KISUMA 5A, KISUMA 5B, KISUMA 5J, KISUMA 5LH and KISUMA 5PH (all trade names of and manufactured by Kyowa Chemical Industry Co., Ltd.). Examples of commercially available aluminum trihydroxide include MARTINAL® manufactured by Matinswerk and HYDRAL manufactured by Alamatis.

[0052] The metal hydrate may be subjected to surface treatment with a surface treating agent, typically a silane surface treating agent, in advance to blending into the composition, or a metal hydrate whose surface is untreated may be blended into the composition together with the surface treating agent, to carry out surface treatment *in situ*. The surface treating agent is suitably added in an amount that is sufficient to provide the desired surface treatment of the metal hydrate. Typically the preferable amount of surface treating agent to be added is 0.1 to 2.0 wt% based on the weight of the metal hydrate. Any of the surface treating agents

known in the art can be employed without any particular restriction. However, a silane surface treating agent having an organic functional group, such as an amino group, a methacrylic group, a vinyl group, an epoxy group and a mercapto group, is preferable, and in terms of the fire retardancy and the tensile property, a silane surface treating agent having a vinyl group and/or an epoxy group is even more preferable.

Coupling Agent:

[0053] As described above, in the context of this invention a coupling agent is a polyfunctional compound, i.e., a compound comprising two or more functional groups, capable of joining together two or more polymer chains via covalently bound links under appropriate reaction conditions. The poly(azide) coupling agents include the alkyl and aryl azides, acyl azides, azidoformates, phosphoryl azides, phosphinic azides, silyl azides and poly(sulfonyl azides). A poly(sulfonyl azide) is any compound having at least two reactive groups (the sulfonyl azide groups ($-SO_2N_3$)) which are reactive with a polyolefin. Preferably the poly(sulfonyl azide)s have a structure X–R–X in which each X is $-SO_2N_3$ and R represents an unsubstituted or inertly-substituted hydrocarbyl, hydrocarbyl ether or silicon-containing group, preferably having sufficient carbon, oxygen or silicon, preferably carbon, atoms to separate the sulfonyl azide groups sufficiently to permit a facile reaction between the polyolefin and the sulfonyl azide. Examples of atoms or groups that may be inertly substituted into R include, but are not limited to, fluorine, aliphatic or aromatic ether, siloxane as well as sulfonyl azide groups in which more than two polyolefin chains are to be joined. R is suitably aryl, alkyl, alkylaryl, arylalkyl silane, siloxane or heterocyclic, groups and other groups which are inert and separate the sulfonyl azide groups as described. More preferably R includes at least one aryl group between the sulfonyl groups, most preferably at least two aryl groups (such as when R is 4,4' diphenylether or 4,4'-biphenyl). When R is one aryl group, it is preferred that the group have more than one ring, as in the case of naphthylene bis(sulfonyl azides). Poly(sulfonyl)azides include but are not limited to such compounds as 1,5-pentane bis(sulfonyl azide), 1,8-octane bis(sulfonyl azide), 1,10-decane bis(sulfonyl azide), 1,10-octadecane bis(sulfonyl azide), 1-octyl-2,4,6-benzene tris(sulfonyl azide), 4,4'-diphenyl ether bis(sulfonyl azide), 1,6-bis(4'sulfonazidophenyl)hexane, 2,7-naphthalene bis(sulfonyl azide), and mixed sulfonyl azides of chlorinated aliphatic

hydrocarbons containing an average of from 1 to 8 chlorine atoms and from about 2 to 5 sulfonyl azide groups per molecule, and mixtures of two or more such compounds. Preferred poly(sulfonyl azide)s include oxy-bis(4-sulfonylazidobenzene), 2,7-naphthalene bis(sulfonyl azide), 4,4'bis(sulfonyl azido)biphenyl, 4,4'-diphenyl ether bis(sulfonyl azide) and bis(4-sulfonyl azidophenyl)methane, and mixtures of two or more such compounds.

[0054] Examples of a silane coupling agent include vinyl-tris(β-methoxyethoxy)silane, vinyltriethoxysilane (VTES), vinyltrimethoxysilane (VMTS), γ-(methacryloyloxypropyl)-trimethoxysilane, γ-(methacryloyloxypropyl)methyldimethoxysilane, γ-glycidoxypropylmethyl-diethoxysilane, and the like. VTES and VTMS are preferred silane coupling agents.

[0055] The coupling agents are used in a coupling amount, e.g., typically in an amount of 0.1 to 6, more typically in an amount of 0.1 to 5 and even more typically in an amount of 0.2 to 3, wt% based on the combined weight of the composition, i.e., the combined weight of the polypropylene, olefinic interpolymer and metal hydrate.

Additives

[0056] The composition may contain additives including but not limited to antioxidants, curing agents, cross linking co-agents, boosters and retardants, processing aids, fillers, ultraviolet absorbers or stabilizers, antistatic agents, nucleating agents, slip agents, plasticizers, lubricants, viscosity control agents, tackifiers, anti-blocking agents, surfactants, extender oils, acid scavengers, and metal deactivators. Additives can be used in amounts ranging from 0.01 wt% or less to 10 wt% or more based on the weight of the composition.

[0057] Examples of antioxidants are as follows, but are not limited to: hindered phenols such as tetrakis[methylene(3,5-di-tert- butyl-4-hydroxyhydro-cinnamate)] methane; bis[(beta-(3,5-ditert-butyl-4-hydroxybenzyl)-methylcarboxyethyl)]sulphide, 4,4'-thiobis(2-methyl-6-tert-butylphenol), 4,4'-thiobis(2-tert-butyl-5-methylphenol), 2,2'-thiobis(4-methyl-6-tert-butylphenol), and thiodiethylene bis(3,5-di-tert-butyl-4-hydroxy)hydrocinnamate; phosphites and phosphonites such as tris(2,4-di-tert-butylphenyl)phosphite and di-tert-butylphenyl-phosphonite; thio compounds such as dilaurylthiodipropionate, dimyristylthiodipropionate, and distearylthiodipropionate;

various siloxanes; polymerized 2,2,4-trimethyl-1,2-dihydroquinoline, n,n'-bis(1,4-dimethylpentyl-p-phenylenediamine), alkylated diphenylamines, 4,4'-bis(alpha, alpha-dimethylbenzyl)diphenylamine, diphenyl-p-phenylenediamine, mixed di-aryl-p-phenylenediamines, and other hindered amine antidegradants or stabilizers. Antioxidants can be used in amounts of 0.1 to 5 wt% based on the weight of the composition.

[0058] Examples of curing agents are as follows: dicumyl peroxide; bis(alpha-t-butyl peroxyisopropyl)benzene; isopropylcumyl t-butyl peroxide; t-butylcumylperoxide; di-t-butyl peroxide; 2,5-bis(t-butylperoxy)2,5-dimethylhexane; 2,5-bis(t-butylperoxy)2,5-dimethylhexyne-3; 1,1-bis(t-butylperoxy)3,3,5-trimethylcyclohexane; isopropylcumyl cumylperoxide; di(isopropylcumyl) peroxide; or mixtures thereof. Peroxide curing agents can be used in amounts of 0.1 to 5 wt% based on the weight of the composition. Various other known curing co-agents, boosters, and retarders, can be used, such as triallyl isocyanurate; ethyoxylated bisphenol A dimethacrylate; α-methyl styrene dimer; and other co-agents described in USP 5,346,961 and 4,018,852.

[0059] Examples of processing aids include but are not limited to metal salts of carboxylic acids such as zinc stearate or calcium stearate; fatty acids such as stearic acid, oleic acid, or erucic acid; fatty amides such as stearamide, oleamide, erucamide, or N,N'-ethylenebis-stearamide; polyethylene wax; oxidized polyethylene wax; polymers of ethylene oxide; copolymers of ethylene oxide and propylene oxide; vegetable waxes; petroleum waxes; non ionic surfactants; and polysiloxanes. Processing aids can be used in amounts of 0.05 to 5 wt% based on the weight of the composition.

[0060] Examples of fillers include but are not limited to clays, precipitated silica and silicates, fumed silica calcium carbonate, ground minerals, and carbon blacks with arithmetic mean particle sizes larger than 10 nanometers. Fillers can be used in amounts ranging from less than 0.01 to more than 50 wt% based on the weight of the composition.

Composition:

[0061] Reactive processing of the composition components will result in a preferred morphology of the solid, high-temperature polymer. Reactively coupled polypropylene

compositions exhibit heat resistance, resistance to ignition and flame spread, and, preferably, flexibility. The reactive processing produces a preferred morphology that includes, but is not limited to, coupling of the polypropylene and olefinic interpolymer.

[0062] Propylene homopolymer or copolymer can be used in any amount such that the composition exhibits as extruded without subsequent crosslinking heat resistance and resistance to ignition and flame spread. Propylene homopolymer or copolymer can comprise at least 10, preferably at least 15 and more preferably at least 20, wt% based on the weight of the composition. The only limit on the maximum amount of propylene homopolymer or copolymer in the composition is that imposed by economics, practicality (*e.g.*, diminishing returns) and the required minimum amounts of the other components of the composition, but typically a general maximum comprises less than 50, preferably less than 45 and more preferably less than 40, wt% based on the weight of the composition.

[0063] The olefinic interpolymer can be used in any amount such that the composition exhibits as extruded without crosslinking heat resistance and resistance to ignition and flame spread. The olefinic interpolymer can comprise at least 10, preferably at least 15 and more preferably at least 20, wt% based on the weight of the composition. The only limit on the maximum amount of olefinic interpolymer in the composition is that imposed by economics, practicality and the required minimum amounts of the other components of the composition, but typically a general maximum comprises less than 50, preferably less than 45 and more preferably less than 40, wt% based on the weight of the composition.

[0064] The metal hydrate can be used in any amount such that the composition exhibits as extruded without subsequent crosslinking heat resistance, flexibility, and resistance to ignition and flame spread. The metal hydrate can comprise at least 35, preferably at least 40, and more preferably at least 50 wt% based on the weight of the composition. The only limit on the maximum amount of metal hydrate in the composition is that imposed by economics, practicality and the required minimum amounts of the other components of the composition, but typically a general maximum comprises less than 75, preferably less than 70 and more preferably less than 65, wt% of the composition.

[0065] The composition also can comprise a coupling package of bis-sulfonyl azide with an antioxidant including but not limited to IRGANOX® 1010 or IRGANOX® MD 1024. This package can comprise at least 0.05, preferably at least 0.1 and more preferably at least 0.2, wt% of the composition. The only limit on the maximum amount of the package in the composition is that imposed by economics, practicality and the required minimum amounts of the other components of the composition, but typically a general maximum comprises less than 2%, preferably less than 1% and more preferably less than 0.5, wt% of the composition. The package is typically added to the composition as it exists in the form of a molecular melt within an extruder or other mixing device.

[0066] Compounding of a cable insulation material can be effected by standard means known to those skilled in the art. Examples of compounding equipment are internal batch mixers, such as a Banbury™ or Bolling™ internal mixer. Alternatively, continuous single, or twin screw, mixers can be used, such as Farrel™ continuous mixer, a Werner and Pfleiderer™ twin screw mixer, or a Buss™ kneading continuous extruder. The type of mixer utilized, and the operating conditions of the mixer, will affect properties of the composition such as viscosity, volume resistivity and extruded surface smoothness.

[0067] Cable comprising an insulation layer that itself comprises a composition of this invention can be prepared with various types of extruders, e.g., single or twin-screw types. USP 4,857,600 provides a description of a conventional extruder. USP 5,575,965 also provides a description of an extruder and a co-extrusion process. Typically, an extruder has a hopper at its upstream end and a die at its downstream end. The hopper feeds into a barrel, which contains a screw. At the downstream end, between the end of the screw and the die, there is a screen pack and a breaker plate. The screw portion of the extruder comprises three sections, i.e., the feed section, the compression section, and the metering section. It also comprises two zones, i.e., the back heat zone and the front heat zone. The sections and zones run from upstream to downstream. Alternatively, the extruder can comprise more than two heating zones along the axis running from upstream to downstream. If the extruder has more than one barrel, then the barrels are typically connected in series. Typically, the length to diameter ratio of each barrel is in the range of 15:1 to 30:1. In wire coating operations in which the polymeric insulation is crosslinked after extrusion, the cable often passes

immediately into a heated vulcanization zone downstream of the extrusion die. Typically, the heated cure zone is maintained at a temperature in the range of 200 to 350°C, preferably in the range of about 170 to about 250°C. The heated zone can be heated by pressurized steam, or by inductively heated, pressurized nitrogen gas. However, crosslinking after extrusion can be eliminated with the practice of this invention.

[0068] The following examples illustrate various embodiments of this invention. All parts and percentages are by weight unless otherwise indicated.

SPECIFIC EMBODIMENTS

[0069] The compositions of the six samples analyzed are reported in Table 1. Three samples (the comparative example (CEX) and EX. 1 and 3) comprise a polypropylene homopolymer (H502-25R) reactively processed with a polyolefin elastomer (AFFINITY™ EG 8200 which has a density of 0.870 g/cm^3 (ASTM D792) and is available from The Dow Chemical Company), and MAGNIFIN®H5 $Mg(OH_2)$ which is available from Albemarle-Martinswerk. Two samples (EX. 2 and EX. 4) comprise a polypropylene homopolymer (H502-25R) reactively processed with ultra-low density polyethylene (ATTANE SC4107 which is available from The Dow Chemical Company), and MAGNIFIN® H5 $Mg(OH_2)$. Example 5 comprises a propylene impact copolymer (C705-44NAHP) and an olefinic interpolymer (VERSIFY™ 3300, 12 mole percent ethylene and 88 mole percent propylene, a density of 0.866 g/ cm^3 and a MFR of 9.8 g/10 min (230°C/21.6kg)) both of which are available from The Dow Chemical Company.

[0070] In addition to the components described above, the samples also comprise FUSABOND® 494 which is a maleic anhydride grafted polyethylene (density of 0.87 g/cc) available from E. I. du Pont de Nemours and Company. The samples also comprise IRGANOX MD 1024, which is a metal deactivator and antioxidant available from Ciba Specialty Chemicals, IRGANOX PS 802DSDP, which is used as a heat stabilizer in combination with a phenolic antioxidant (also available from Ciba Specialty Chemicals), and CHIMASSORB 944 which is a hindered amine light stabilizer (also available from Ciba Specialty Chemical). EX. 3, 4 and 5 also comprise IRGANOX 1010, which is an antioxidant. EX. 1-2 and 5 and the comparative example also comprises Dow-Corning

MB 50-001 which is a formulation containing 50% of an ultra-high molecular weight siloxane polymer dispensed in polypropylene homopolymer. EX. 1-2 and 5 further comprise an additive package of bis-sulfonyl azide (BSA) and IRGANOX 1010. EX 3 and 4 further comprise XL PEarl Silane which is a mixture of peroxide, a vinyltrialkoxysilane and a silane dehydro-condensation catalyst. The peroxide decomposes during compounding causing the vinyl silane to graft to the polymer chains. Small amounts of moisture released from the metal hydrate during compounding in combination with the dehydro-condensation catalyst cause coupling of the silane-grafted polymer chains.

Table 1

Formulations of the samples tested

	CEX.	EX. 1	EX. 2	EX. 3	EX. 4	EX. 5
PP (HP) H502-25R (%)	15	15	15	14	14	
C705-44NAHP (%)						15
AFFINITY EG8200 (%)	32.3	32		31		
ATTANE SC4107 (%)			32		31	
VERSIFY 3300.00 (%)						32
FUSABOND 494 (%)	2	2	2	2	2	2
MAGNIFIN® H5 MV (%)	50	50	50	50	50	50
IRGANOX MD 1024 (%)	0.2	0.2	0.2	0.2	0.2	0.2
CHIMASSORB HALS 944 (%)	0.2	0.2	0.2	0.2	0.2	0.2
IRGANOX 1010 (%)				0.2	0.2	0.2
IRGANOX PS 802 DSTDP (%)	0.1	0.1	0.1	0.1	0.1	0.1
Dow-Corning MB 50-001 (PP based 50%) (%)	0.2	0.2	0.2			0.2
BSA Molecular Melt with IRGANOX 1010 (%)		0.3	0.3			0.3
XI PEARL SILANE (%)				2.3	2.3	
TOTAL (wt%)	100	100	100	100	100	100

[0071] Multiple samples for testing of flexural modulus, melt flow index, thermal mechanical analysis (TMA), Hot Set or Creep, and tensile properties are prepared as follows: one plaque is prepared by molding (of the material out of a mixing bowl) in a 50 mil (1.27

mm) mold at 160°C for 10 minutes, followed by 6 hours cure in 90°C water bath. Multiple dog-bones are cut and tested to provide an average test value.

[0072] The flexural properties are tested according to ISO 178 and are reported in Table 2. The Flexural test measures the force required to bend a beam under 3 point loading conditions. Flexural modulus is used as an indication of the stiffness of a material when flexed.

Table 2

Flexural Test Results for Samples Tested According to ISO179 (1 mm/min)

	CEX	EX. 1	EX. 2	EX. 3	EX. 4	EX. 5
(MPa)	250.7	204.4	687.5	331.8	303.3	260.4

[0073] Melt flow index is tested according to ISO1133 at 230C and are reported in Table 3. Three specified weights, 2.16 kg, 5 kg, and 21.6 kg, are shown in Table 3.

Table 3

Melt Flow Index for Samples Tested Using ISO1133 at 230°C

	CEX	EX. 1	EX. 2	EX. 3	EX. 4	EX. 5
2.16 kg	3.17					
5 kg		0.52				
21.6 kg			7.62	5.75	0.36	20.68

[0074] Thermal mechanical analysis (TMA) is a probe penetration test in which the sample is heated at a constant rate and the temperature at which penetration starts (softening temperature) is reported as the TMA temperature. TMA temperatures for the six samples are reported in Table 4.

Table 4

Thermal Mechanical Analysis of Samples Tested (1 mm penetration at 5°C/min)

	CEX	EX. 1	EX. 2	EX. 3	EX. 4	EX. 5
Temperature (°C)	109	151.4	151.6	154.6	229.9	144.2

[0075] Table 5 reports Hot Set measured for each of the six samples at 150°C.

Table 5

Hot Set Test for Samples Tested According to EN 60811-2-1 at 150°C/20N/cm^2

	CEX	EX. 1	EX. 2	EX. 3	EX. 4	EX. 5
Hot Elongation (%)	break	115	47	61	7	215
Relax Elongation (%)		63	25	32	1	204

[0076] Table 6 reports the Hot Set measured for each of the six samples at 200°C.

Table 6

Hot Set Test for Samples Tested According to EN 60811-2-1 at 200°C/20N/cm^2

	CEX	EX. 1	EX. 2	EX. 3	EX. 4	EX. 5
Hot Elongation (%)	break	break	>200	>200	15	Not Tested
Relax Elongation (%)					34	

[0077] Table 7 reports the tensile properties for the six samples tested.

Table 7

Tensile Properties for Samples Tested According to EN 60811-1-1 at 50mm/min

	CEX	EX. 1	EX. 2	EX. 3	EX. 4	EX. 5
Ultimate Tensile Strength (Mpa)	4.7	9.1	10.3	6.3	9.0	12.1
Elongation at break (%)	424	520	300	366	406	675

[0078] The compositions described above in which a propylene homopolymer is reactively processed with a polyolefin elastomer (AFFINITY®) or ultra-low density polyethylene (ATTANE®) or even a low ethylene content olefinic interpolymer (VERSIFY^{tm}) exhibit, as extruded without subsequent cross-linking, (1) heat resistance; (2) resistance to ignition and

flame spread, and (3) flexibility. These properties make these compositions suitable for cable applications, such as 125°C rated automotive wire.

[0079] Although the invention has been described in considerable detail by the preceding specification, this detail is for the purpose of illustration and is not to be construed as a limitation upon the following appended claims. All cited reports, references, U.S. patents, allowed U.S. patent applications and U.S. Patent Application Publications are incorporated herein by reference.

What is claimed is:

1. A process for coupling a propylene polymer with an olefinic interpolymer, the process comprising contacting under reactive processing conditions at least:

A. 10 wt% of at least one propylene polymer;

B. 10 wt% of at least one olefinic interpolymer;

C. 35 wt% of at least one metal hydrate;

D. A coupling amount of a coupling agent; and

E. Optionally, one or more additives.

2. The process of Claim 1 in which the coupling agent is at least one of a poly(sulfonyl) azide and a silane having a vinyl group.

3. The process of any of the preceding claims in which the coupling agent is present in an amount of 0.1 to 6 weight percent based on the combined weight of the propylene polymer, olefinic interpolymer and metal hydrate.

4. The process of any of the preceding claims in which the reactive processing conditions include a temperature of 150 to 280°C.

5. The process of any of the preceding claims in which the olefinic interpolymer is at least one of very low density polyethylene; homogeneously branched, linear ethylene/α-olefin copolymer; homogeneously branched, substantially linear ethylene/α-olefin polymer; linear medium density polyethylene, linear low density polyethylene, ultra low density polyethylene and an olefinic multi-block copolymer.

6. The process of any of the preceding claims in which the olefinic interpolymer is a homogeneously branched, substantially linear ethylene/α-olefin polymer with a content of units derived from ethylene content of at least 80 wt% and a density of at least 0.870 g/cm^3.

7. The process of Claim 4 in which the metal hydrate is at least one of: aluminum hydroxide and magnesium hydroxide.

8. The process of any of the preceding claims in which an antioxidant is present in an amount of 0.01 to 10 wt% based on the combined weight of the propylene polymer, olefinic interpolymer and metal hydrate.

9. A composition made by any of the preceding processes.

10. The composition of Claim 9 in the form of a wire or cable insulation sheath.

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2009/044805

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08J3/24 C08K5/00 C08L51/06

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)
C08F C08L C08J C08K

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	US 2001/025720 A1 (BISLERI CESARE [IT] ET AL) 4 October 2001 (2001-10-04) claims 1,4,10,13; table I	1-10
X,P	EP 1 985 659 A (NITTO DENKO CORP [JP]) 29 October 2008 (2008-10-29) paragraphs [0002], [0007], [0043], [0053]; table 1	1-10
X	GB 1 470 464 A (MOORE CO S) 14 April 1977 (1977-04-14) page 7, lines 10-16; example 3	1-10
X	WO 01/13381 A (DOW CHEMICAL CO [US]; BETSO STEPHEN R [DE]; GUEST MARTIN J [US]; REMEN) 22 February 2001 (2001-02-22) claims 1,8,9,14,17,19,23-25	1-10

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[X] Further documents are listed in the continuation of Box C.

[X] 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	Date of mailing of the international search report
5 August 2009	14/08/2009

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Form PCT/ISA/210 (second sheet) (April 2005)

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2009/044805

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2006/199910 A1 (WALTON KIM L [US] ET AL) 7 September 2006 (2006-09-07) the whole document	1-10

Form PCT/ISA/210 (continuation of second sheet) (April 2005)

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2009/044805

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