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(54) Title: POLYOLEFIN PIPES

(57) Abstract: Disclosed in one aspect is a pipe comprising a composition of one or more propylene-α-olefm copolymers comprising propylene-derived units and from 5 to 35 wt%, by weight of the propylene-α-olefin copolymer, of ethylene-derived units and/or a C_4 to C_{10} α-olefin-derived unit, and having (i) a density from 0.850 to 0.920 g/cm3; (ii) an Hf of less than 75 J/g; and (iii) a Tm of less than 105°C; and at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof; wherein the pipe composition possesses a Flexural Modulus (23°C) less than 650 MPa, an Izod Impact (23°C) greater than 30 kJ/m2, and a Vicat Softening Temperature greater than 110°C.°

POLYOLEFIN PIPES

FIELD OF THE INVENTION

[0001] The present invention relates in general to pipes made from polyolefin compositions, and more particularly relates to polypropylene-based pipes suitable as a pipe (channel/conduit) for transporting liquids, gases, and/or flowable solid.

BACKGROUND

[0002] It is common to make pipes suitable for transporting liquids from one of several compositions: poly-1-butene, cross-linked polyethylene (peroxide crosslinked, silane crosslinked or radiation crosslinked), non-crosslinked polyethylene, especially high density polyethylene, and certain types of polypropylene. There are several disadvantages to all of these. Poly-1-butene works well, but is a more expensive composition for pipes. Cross-linked polyethylene is also expensive. Polypropylene, especially homopolymers, are less expensive but are not sufficiently resistant to long term heat/pressure environments and are too rigid and fragile. A propylene random copolymer is typically more resistant to heat and pressure environments, but lacks formability, such as to form "U" shaped bends (and other types of forming) suitable for in-floor heating coils and is still too rigid and fragile. What is needed is a pipe composition that is cost effective, strong enough to meet current regulations (e.g. flexural modulus (23°C) less than 650 MPa, an Izod Impact (23°C) greater than 30 kJ/m^2, and a Vicat Softening Temperature greater than 110°C) and allow for formability.

[0003] It is known to blend other propylene-based materials with polypropylene homopolymers and random copolymers, such as in US 6,635,715, in order to modify their properties. The inventors have found that blends of a polypropylene with a certain type of propylene-α-olefin copolymer are suitable for such an improved pipe composition.

SUMMARY

[0004] Disclosed in one aspect is a pipe comprising a composition of one or more propylene-α-olefin copolymers comprising propylene-derived units and from 5 to 35 wt%, by weight of the propylene-α-olefin copolymer, of ethylene-derived units and/or C_4 to C_{10} α-olefin-derived units, and having (i) a density from 0.850 to 0.920 g/cm^3; (ii) an H_f of less than 75 J/g; and (iii) a T_m of less than 105°C; and at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof; wherein the pipe composition possesses a Flexural Modulus (23°C) less than 650 MPa, an Izod Impact (23°C) greater than 30 kJ/m^2, and a Vicat Softening Temperature greater than 110°C.

[0005] In another aspect is a method of forming a pipe comprising melt-blending one or more propylene-α-olefin copolymers comprising propylene-derived units and from 5 to 35 wt%, by weight of the propylene-α-olefin copolymer, of ethylene-derived units and/or C_4 to C_{10} α-olefin-derived units, and having (i) a density from 0.850 to 0.920 g/cm^3; (ii) an H_f of less than 75 J/g; (iii) a T_m of less than 105°C; and at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof; followed by extruding the blend of propylene-α-olefin copolymers and additional component through a pipe-forming die; and isolating a pipe possessing a flexural modulus (23°C) less than 650 MPa, an Izod Impact (23°C) greater than 30 kJ/m^2, and a Vicat Softening Temperature greater than 110°C.

[0006] Insofar as certain numerical ranges of the features of the invention(s) are described, it is understood that any desirable upper limit of that range can be combined with any desirable lower limit of that range, as disclosed herein, to achieve a preferred range.

DETAILED DESCRIPTION

[0007] As used herein, "pipe" refers to a cylindrical channel (or conduit) having a circular, ellipsoidal, square or other suitable cross-section, and having a certain wall thickness, and capable of transporting liquid and/or gas and/or flowable solid. The inside of the wall makes contact with the substance to be conveyed in one embodiment, or is a layer of a multi-layer pipe (thus contacting another layer of pipe material) in another embodiment. The pipe can be of any desirable length and made by any suitable means known in the art.

[0008] As used herein, a "pipe composition" is a combination of two or more components defined herein that is used to form the pipe, or at least a section or layer of the pipe. The pipe compositions include one or more propylene-α-olefin copolymers and at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof. In a particular embodiment, the pipes herein consist essentially of one or more propylene-α-olefin copolymers and at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof. It will be understood that the properties (e.g., Vicat Softening Temperature, HDT, Tensile, Izod, Elongation, etc.) disclosed herein refer to the pipe composition and/or the pipe made exclusively from the pipe composition and/or only the portion of the pipe made from the pipe composition.

[0009] The "propylene-α-olefin copolymers" described herein are copolymers of propylene-derived units and one or more units derived from ethylene and/or a C_4-C_{10} α-olefin and optionally one or more diene-derived units. Preferred α-olefins are ethylene, 1-butene, 1-hexene

and 1-octene. In certain embodiments, the propylene-α-olefin copolymers consist of propylene-derived units and from 5 to 35 wt%, by weight of the propylene-α-olefin copolymer, of ethylene-derived units and/or C_4 to C_{10} α-olefin-derived units.

[0010] Typical comonomer content of the copolymer is from 5 to 35 wt% in one embodiment. In general, the comonomer content is adjusted so that the copolymer preferably has a molecular weight distribution ("MWD", Mw/Mn) of from 1.5 to 20, more preferably from 1.5 to 5, a heat of fusion (H_f) less than or equal to 75 J/g and a melting temperature (T_m) less than or equal to 105°C. In some embodiments, where more than one comonomer is present, the amount of a particular comonomer may be less than 5 wt%, but the combined comonomer content is preferably greater than 5 wt%.

[0011] In such an embodiment, when there is more than one α-olefin-derived unit in the copolymer, the total weight percent of the ethylene or C_4-C_{10} α-olefin-derived units (or "α-olefin") is preferably from 5 to 35 wt%, more preferably from 7 to 32 wt%, more preferably from 8 to 25 wt%, more preferably from 8 to 20 wt%, and more preferably from 8 to 18 wt%. Particular embodiments of copolymers having more than one α-olefin include propylene-ethylene-octene, propylene-ethylene-hexene and propylene-ethylene-butene polymers. These copolymers may further comprise a diene as described below.

[0012] In one embodiment, the propylene-α-olefin copolymer comprises propylene derived units and ethylene derived units. The propylene-ethylene copolymer can comprise from 5 to 25 wt% ethylene-derived units, preferably from 5 to 20 wt%, more preferably from 5 to 16 wt%, and more preferably from 6 to 18 wt% ethylene. In a preferred embodiment, the propylene-ethylene copolymer comprises greater than 11 wt% ethylene, the remainder of the copolymer comprising propylene derived units.

[0013] The propylene-α-olefin copolymer, a propylene-ethylene copolymer in one embodiment, may optionally comprise less than or equal to 10 wt% diene derived units (or "diene"), preferably less than or equal to 5 wt% diene, more preferably less than or equal to 3 wt% diene, preferably from 0.1 to 3 or 4 or 5 or 6 wt%, more preferably from 0.1 to 2 wt%, and more preferably from 0.1 to 1 wt% diene. Suitable dienes useful as co-monomers are, for example: 1,4-hexadiene, 1,6-octadiene, 5-methyl-1,4-hexadiene, 3,7-dimethyl-1,6-octadiene, dicyclopentadiene (DCPD), ethylidiene norbornene (ENB), norbornadiene, 5-vinyl-2-norbornene (VNB), and combinations thereof. The diene, if present, is most preferably ENB.

[0014] The propylene-α-olefin copolymer can have a triad tacticity of three propylene units, as measured by ^{13}C NMR, of 75% or greater, 80% or greater, 82% or greater, 85% or greater, or 90% or greater. Preferred ranges include from 50 to 99%, more preferably from 60 to 99%, more preferably from 75 to 99% and more preferably from 80 to 99%; and in other embodiments from

60 to 97%. Triad tacticity was determined as follows: The tacticity index, expressed herein as "m/r", is determined by ^{13}C nuclear magnetic resonance (NMR). The tacticity index m/r is calculated as defined by *H. N. Cheng* in 17 MACROMOLECULES 1950 (1984). The designation "m" or "r" describes the stereochemistry of pairs of contiguous propylene groups, "m" referring to meso and "r" to racemic. An m/r ratio of 1.0 generally describes a syndiotactic polymer, and an m/r ratio of 2.0 an atactic material. An isotactic material theoretically may have a ratio approaching infinity, and many by-product atactic polymers have sufficient isotactic content to result in ratios of greater than 50. Embodiments of the propylene-α-olefin copolymer have a tacticity index m/r ranging from a lower limit of 4 or 6 to an upper limit of 8 or 10 or 12.

[0015] In certain embodiments the propylene-α-olefin copolymer has an H_f, determined according to the Differential Scanning Calorimetry (DSC) procedure described herein, greater than or equal to 0.5 or 1 or 5 J/g, and is less than or equal to 80 J/g, preferably less than or equal to 70 J/g, more preferably less than or equal to 50 J/g, more preferably less than or equal to 35 J/g. Stated another way, in one or more embodiments the H_f ranges from a lower limit of 1.0, or 1.5, or 3.0, or 4.0, or 6.0, or 7.0 J/g to an upper limit of 30, or 35, or 40, or 50, or 60 or 70, or 80 J/g.

[0016] In certain embodiments, the propylene-α-olefin copolymer, a propylene-ethylene copolymer in one embodiment, has a percent crystallinity of from 0.5 to 40%, preferably 1 to 30%, and more preferably 5 to 25% wherein "percent crystallinity" is determined according to the DSC procedure described herein. The thermal energy for the highest order of polypropylene is estimated at 189 J/g (i.e., 100% crystallinity is equal to 189 J/g). In another embodiment, the propylene-ethylene copolymer of the present disclosure preferably has a crystallinity of less than 40%, preferably from 0.25 to 25%, more preferably from 0.5 to 22%, and most preferably from 0.5 to 20%.

[0017] The procedure for DSC determinations is as follows. About 0.5 grams of polymer was weighed out and pressed to a thickness of about 15-20 mils (about 381-508 microns) at about 140°C-150°C, using a "DSC mold" and Mylar as a backing sheet. The pressed pad was allowed to cool to ambient temperature by hanging in air (the Mylar was not removed). The pressed pad was annealed at room temperature (about 23-25°C) for about 8 days. At the end of this period, an about 15-20 mg disc was removed from the pressed pad using a punch die and was placed in a 10 microliter aluminum sample pan. The sample was placed in a differential scanning calorimeter (Perkin Elmer Pyris 1 Thermal Analysis System) and was cooled to about -100°C. The sample was heated at about 10°C/min to attain a final temperature of about 165°C. The thermal output, recorded as the area under the melting peak of the sample, is a measure of the heat of fusion and can be expressed in Joules per gram (J/g) of polymer and was automatically calculated by the

Perkin Elmer System. Under these conditions, the melting profile shows two (2) maxima, the maxima at the highest temperature was taken as the melting point within the range of melting of the sample relative to a baseline measurement for the increasing heat capacity of the polymer as a function of temperature.

[0018] In addition to this level of crystallinity, the propylene-ethylene copolymer preferably has a single broad melting transition. The "T_m" is defined as the temperature of the greatest heat absorption within the range of melting of the sample. However, the propylene-ethylene copolymer may show secondary melting peaks adjacent to the principal peak, but for purposes herein, such secondary melting peaks are considered together as a single melting point, with the highest of these peaks being considered the T_m of the propylene-ethylene copolymer. The propylene-ethylene copolymer preferably has a T_m of from 25 to 105°C, preferably from 25 to 85°C, more preferably from 25 to 75°C, more preferably from 25 to 65°C, more preferably from 30 to 80 °C, more preferably from 30 to 70°C and more preferably from 30 to 60°C.

[0019] In certain embodiments, the propylene-α-olefin copolymer can have a density of 0.850 to 0.920 g/cm^3, more preferably, 0.870 to 0.900 g/cm^3, more preferably 0.880 to 0.890 g/cm^3 at room temperature as measured per the ASTM D-1505 test method.

[0020] In certain embodiments, the propylene-α-olefin copolymer can have a melt flow rate ("MFR", ASTM D1238, 2.16 kg, 230°C), equal to or greater than 0.2 dg/min. Preferably, the MFR is from 0.5 to 5000 dg/min and more preferably from 1 to 2500 dg/min. In one embodiment, the propylene-α-olefin copolymer has an MFR of 0.5 to 1500 dg/min, from 2 to 1000 dg/min in another embodiment, and from 5 to 500 dg/min in yet another embodiment, and from 10 to 250 dg/min in yet another embodiment, and from 10 to 100 dg/min in yet another embodiment, and from 2 to 40 dg/min in yet another embodiment, and from 2 to 30 dg/min in yet another embodiment, and from 0.5 to 10 dg/min in yet another embodiment.

[0021] In certain embodiments, the propylene-α-olefin copolymers, propylene-ethylene copolymers in one embodiment, may have a Mooney viscosity [ML (1+4) @ 125°C] as determined according to ASTM D1646, of less than 150, more preferably less than 100, even more preferably less than, most preferably less than 60.

[0022] In one embodiment, the propylene-α-olefin copolymer can have a Mw of 40,000 to 5,000,000 g/mole, more preferably a Mw of 50,000 to 1,000,000, and more preferably a Mw of 70,000 to 400,000. In another embodiment, the propylene-α-olefin copolymer can have a Mn of 20,000 to 2,500,000 g/mole, more preferably a Mn of 40,000 to 350,000, and more preferably a Mn of 60,000 to 200,000. In yet another embodiment, the propylene-α-olefin copolymer can have a Mz of 50,000 to 7,000,000 g/mole, more preferably a Mz of 80,000 to 700,000, and more preferably a Mz of 100,000 to 500,000.

[0023] The molecular weight distribution (MWD) of the propylene-ethylene copolymer is from 1.5 to 20 in one embodiment, and from 1.5 to 15 in another embodiment, and more preferably 1.5 to 5, more preferably 1.8 to 5 and most preferably 1.8 to 4 or 3.

[0024] Techniques for determining the molecular weight (Mn, Mz and Mw) and molecular weight distribution (MWD) are as follows, and as in *Verstate et al.* in 21 MACROMOLECULES 3360 (1988). Molecular weight and molecular weight distribution are measured using a Waters 150 gel permeation chromatograph equipped with a Chromatix KMX-6 on-line light scattering photometer. The system was used at 135°C with 1,2,4-trichlorobenzene as the mobile phase. Showdex (Showa-Denko America, Inc.) polystyrene gel columns 802, 803, 804 and 805 are used. This technique is discussed in LIQUID CHROMATOGRAPHY OF POLYMERS AND RELATED MATERIALS III 207 (J. Cazes ed., Marcel Dekker, 1981). No corrections for column spreading were employed; however, data on generally accepted standards, for example, National Bureau of Standards Polyethylene 1484 and anionically produced hydrogenated polyisoprenes (an alternating ethylenepropylene copolymer) demonstrate that such corrections on Mw/Mn or Mz/Mw are less than 0.05 units. Mw/Mn was calculated from an elution time-molecular weight relationship whereas Mz/Mw was evaluated using the light scattering photometer. The numerical analyses can be performed using the commercially available computer software GPC2, MOLWT2 available from LDC/Milton Roy-Riviera Beach, Fla.

[0025] The propylene-α-olefin copolymers can include copolymers prepared according to the procedures in WO 02/36651, US 6,992,158, and/or WO 00/01745. Preferred methods for producing the propylene-α-olefin copolymers are found in US Patent Application Publication 2004/0236042 and US 6,881,800. Preferred propylene-α-olefin copolymers are available commercially under the trade names Vistamaxx™ (ExxonMobil Chemical Company, Houston, TX, USA) and Versify™ (The Dow Chemical Company, Midland, Michigan, USA), certain grades of Tafmer™ XM or Notio™ (Mitsui Company, Japan) or certain grades of Softel™ (Basell Polyolefins of the Netherlands).

[0026] The pipe of the present invention also includes an additional component selected from the group consisting of propylene homopolymers, propylene random copolymers, propylene block copolymers, and combinations thereof. If present, the propylene homopolymer has a MFR (230°C, 2.16 kg) of from 0.05 to 10 dg/min in one embodiment, and from 0.1 to 5 dg/min in another embodiment, and from 0.1 to 2 dg/min in yet another embodiment. In a particular embodiment, the propylene homopolymer is an isotactic propylene homopolymer.

[0027] The "propylene random copolymer" is a polymer comprising propylene-derived units and from less than 5 wt% comonomer-derived units in one embodiment. Suitable comonomers include ethylene-derived units and/or units selected from C_4 to C_{10} α-olefin derived units. In a

particular embodiment, the propylene random copolymer is a propylene-ethylene random copolymer.

[0028] In one embodiment, the propylene random copolymer comprises from 0.1 to 5 wt% of comonomer-derived units, and from 0.2 to 4 wt% in another embodiment, and from 0.3 to 2 wt% in yet another embodiment. In yet another embodiment, the propylene random copolymer has an MFR (230°C, 2.16 kg) of from less than 5 dg/min, and from 0.05 to 5 dg/min in another embodiment, and from 0.1 to 3 dg/min in yet another embodiment. In yet another embodiment, the propylene random copolymer has a melt temperature (T_m) of greater than 135°C, and greater than 140°C in another embodiment, and greater than 150°C in yet another embodiment. In yet another embodiment, the propylene random copolymer has a Flexural Modulus (23°C) of greater than 650 MPa, and greater than 700 MPa in another embodiment, and greater than 750 MPa in yet another embodiment.

[0029] The "propylene block copolymer" is a reactor made blend of at least one propylene homopolymer with at least one propylene-ethylene random copolymer containing less than 50 weight % propylene-ethylene random copolymer.

[0030] In one embodiment, the propylene block copolymer comprises from 1 to 50 wt% of propylene-ethylene random copolymer, and from 0.2 to 30 wt% in another embodiment, and from 0.3 to 20 wt% in yet another embodiment. In yet another embodiment, the propylene block copolymer has an MFR (230°C, 2.16 kg) of from less than 5 dg/min, and from 0.05 to 5 dg/min in another embodiment, and from 0.1 to 3 dg/min in yet another embodiment. In yet another embodiment, the propylene block copolymer has a melt temperature (T_m) of greater than 135°C, and greater than 140°C in another embodiment, and greater than 150°C in yet another embodiment. In yet another embodiment, the propylene block copolymer has a Flexural Modulus (23°C) of greater than 650 MPa, and greater than 700 MPa in another embodiment, and greater than 750 MPa in yet another embodiment.

[0031] The propylene homopolymer, propylene random copolymer, or propylene block copolymer are present in the pipe compositions, individually, from 60 to 95 wt% of the composition in one embodiment, and from 65 to 90 wt% of the composition in another embodiment, and from 68 to 75 wt% of the composition in yet another embodiment. In yet another embodiment, a mixture of two or more of the propylene homopolymer, propylene random copolymer, and propylene block copolymer are present from 60 to 95 wt% of the composition in one embodiment, and from 65 to 90 wt% of the composition in another embodiment, and from 68 to 75 wt% of the composition in yet another embodiment.

[0032] The pipe compositions of the present invention can include one or more plastomers. A "plastomer" comprises ethylene-derived units and at least one of C_3 to C_8 α-olefin derived

units from 1 wt% to 40 wt% of the plastomer in one embodiment, and from 5 to 35 wt% of the plastomer in another embodiment, and from 5 to 30 wt% of the plastomer in yet another embodiment. More particularly, a plastomer is a copolymer of ethylene-derived units and at least one of non-cyclic mono-olefins such as propylene, 1-butene, 1-pentene, 1-hexene, 1-octene and 4-methyl-1-pentene. However, cyclic mono-olefins and both linear and cyclic dienes can also be used in copolymerization with ethylene to form the plastomer. It is desirable in some applications to use ethylene-α-olefin-diene terpolymers.

[0033] In a preferred embodiment, the plastomer is a copolymer of ethylene derived units and 1-hexene or 1-octene derived units, wherein the 1-hexene or 1-octene derived units are present from 5 to 40 wt% of the plastomer in one embodiment, from 5 to 30 wt% of the plastomer in another embodiment, and from 10 to 28 wt% in another embodiment, and from 15 to 27 wt% in yet another embodiment.

[0034] In one embodiment of the invention, the plastomer has a density in the range of 0.856 to 0.915 g/cm^3, and a density of from 0.880 to 0.915 g/cm^3 in another embodiment, and from 0.865 to 0.915 g/cm^3 in one embodiment, and in the range of from 0.870 to 0.910 g/cm^3 in another embodiment, and in the range of 0.880 to 0.908 g/cm^3 in yet another embodiment, and in the range of 0.880 to 0.906 g/cm^3 in yet another embodiment.

[0035] The I_2 (ASTM D-1238, 190°C, 2.16 kg) of the plastomer is in the range of from 0.10 to 40 dg/min in one embodiment, and from 0.5 to 10 dg/min in another embodiment, and from 1.0 to 6.0 dg/min in another embodiment, and from 1.5 to 5.0 dg/min in yet another embodiment.

[0036] Desirable plastomers are sold, for example, under the trademark Exact™ (ExxonMobil Chemical Company, Houston, Texas), such as Exact 0203. The invention can also be practiced using Engage™ polymers (also Affinity™ and Versify™; Dow Chemical Company, Midland, Michigan) and Tafmer™ (Mitsui Petrochemical Co.). The propylene-based polymer Vistamaxx™ (ExxonMobil Chemical Co.) may also be used as the one or more plastomers.

[0037] When present, the plastomer can be present in an amount of from 0.5 to 20 wt% of the pipe composition in one embodiment, and from 1 to 15 wt% in another embodiment, and from 2 to 12 wt% in yet another embodiment.

[0038] In another embodiment, the composition that is used to form the pipe is blended with from 1 to 40 wt% of a filler, and from 2 to 30 wt% in another embodiment, and from 3 to 20 wt% in yet another embodiment. Fillers that can be included in the pipe composition include those reinforcing and non-reinforcing fillers or extenders that are conventionally employed in the compounding of polymeric materials. Useful fillers include carbon black, calcium carbonate, clays, silica, fumed silica, talc, and titanium dioxide. Useful fillers also include nano fillers such as nano clay, nano talc, wollastonite, kaolin and titanium dioxide. The filler can be surface

treated with a silane derivative such as aminosilane, or other, for better dispersion into polymer phase. A functionalized polypropylene like maleic anhydride grafted polypropylene can be used as compatibilizer between mineral filler and polymer phase.

[0039] Other components may be present in the pipe composition up to 4 wt%, including stabilizers, nucleating agents, UV stabilizers, antioxidants, slip agents, pigments, etc. In the embodiments where the pipe composition consists essentially of the propylene-α-copolymer and the propylene homopolymer, propylene random copolymer or propylene block copolymer, the composition can also include these minor components up to 1 or 2 or 3 or 4 wt% based on the weight of the composition.

[0040] The pipe compositions have a MFR (230°C, 2.16 kg) from 0.1 to 10 dg/min in one embodiment, and from 0.2 to 5 dg/min in another embodiment, and from 0.3 to 3 dg/min in yet another embodiment.

[0041] The pipe compositions can have a Flexural Modulus (23°C, ISO 178) from less than 650 MPa in one embodiment, and less than 600 MPa in another embodiment, and less than 550 MPa in yet another embodiment, and greater than 100 MPa in yet another embodiment.

[0042] The pipe compositions can have an Izod Impact (23°C, ISO 180) from greater than 30 kJ/m^2 in one embodiment, and greater than 35 kJ/m^2 in another embodiment, and greater than 40 kJ/m^2 in yet another embodiment, and less than 100 kJ/m^2 in yet another embodiment, and less than 80 kJ/m^2 in yet another embodiment.

[0043] The pipe compositions can have an Charpy Impact (23°C, ISO 179) from greater than 12 kJ/m^2 in one embodiment, and greater than 20 kJ/m^2 in another embodiment, and greater than 30 kJ/m^2 in yet another embodiment, and less than 100 kJ/m^2 in yet another embodiment, and less than 80 kJ/m^2 in yet another embodiment.

[0044] The pipe compositions can have a Vicat Softening Temperature (ISO 306) greater than 110°C in one embodiment, and greater than 115°C in another embodiment, and greater than 120°C in another embodiment, and greater than 130°C in yet another embodiment.

[0045] The pipe compositions can have a heat deflection temperature ("HDT", ISO 75) from 50 to 110°C in one embodiment, and from 70 to 100°C in another embodiment, and from 72 to 95°C in yet another embodiment.

[0046] The pipe composition described herein can be used to make single- or multilayer pipes. Further, the pipe composition can be used to make up one or more layers of a pipe, the other layer(s) being formed from other synthetic, metal or allow materials. Conventional extruders are suitable for producing the single-or multilayer propylene pipe composition. Injection molding machines are suitable. For example, extruders with short compression screws or 3-zone screws with L/D 20 to 40 are suitable for melting the propylene-based polymers and,

when present, plastomer, pursuant to the inventive method. Preferably, 5-zone screws with a feed zone, compression zone, shear zone, decompression zone and homogenizing zone are preferred. Screws with cutting depths of 1:2.5 to 1:3.5 are particularly suitable. Extruders equipped with a grooved barrel section are also suitable. Optionally, a melt pump and/or a static mixer can be used additionally between the extruder and the ring die head. Ring shaped dies with diameters ranging from approximately 16 to 2000 mm and greater are possible. Advantageous die temperatures for discharging the melt are 190 to 240°C. After leaving the ring-shaped die, the polypropylene pipes are taken off over a vacuum calibrating sleeve, and cooled.

[0047] The pipe compositions can be intimately melt-blended prior to formation of the pipe, or the components can be combined and melt-blended in the same apparatus that is used to make the pipe. The pipe compositions can be formed into pipes of any diameter, preferably between 1 cm and 10 cm, with a wall thickness of from 2 to 8 mm. Such pipes are particularly suitable for in-floor heating systems and can be advantageously heat-bent into a "U" shape to make a continuous flow of heated water through the floor in which it is installed.

[0048] Preferred applications of the propylene polymer pipes the conveyance of fluids and pressurized fluids such as natural gas and water and the like at service temperatures at or above room temperature, but also below room temperature and even the freezing point of water when applicable.

[0049] In a particular embodiment, the pipe composition comprises 70-80 wt% of a propylene random copolymer and 10-20 wt% of a propylene-α-olefin, based on the weight of the composition. In another embodiment, the pipe composition comprises 60-70 wt% of a propylene random copolymer, and 20-30 wt% of the propylene-α-olefin. In yet another embodiment, the pipe composition comprises 70-80 wt% of a propylene homopolymer and 10-20 wt% of a propylene-α-olefin. In another embodiment, the pipe composition comprises 60-70 wt% of a propylene homopolymer, and 20-30 wt% of the propylene-α-olefin, by weight of the composition. In yet another embodiment, the pipe composition comprises 60-70 wt% of a propylene random copolymer, 10-20 wt% of a propylene-α-olefin and 5-15 wt% of a plastomer, by weight of the composition. In yet another particular embodiment, the pipe composition consists essentially of 70 to 80 wt% of a propylene random copolymer and 20 to 30 wt% of a propylene-α-olefin, and from 0.1 to 1 wt% of an additional additive such as a nucleator or stabilizer, by weight of the composition.

EXAMPLES

[0050] The data in Table 1 presents some of the basic properties of the propylene-α-olefin copolymers used in the Examples. The MFR was determined according to ASTM D-1238

(230°C, 2.16 kg); the Flexural Modulus is the 1% secant, determined by ASTM D-790; the Tensile at break was determined by ASTM D-412; the Shore A hardness was determined by ASTM D-2240; and the Vicat Softening Point was determined by ASTM D-1525, 200 g.

[0051] The components discussed above and in Table 1 that make up the example compositions of the invention were combined in the following manner: the mixer type was a Brabender Plasti-Corder EU drive equipped with a Brabender twin screw extruder DSE 25/30. The mixer settings were as follows: temperature profile (hopper to die in °C) 160 - 180 - 200 - 210 - 230 – 230, mixer set at 60 rpm, the die was a plate die with 2 round holes (3 mm diameter) - for 2 strands, and the strands were cooled in water bath (± 15°C). Strands were cut in pellets with a Scheer Strand granulator (SGS 50-E).

[0052] The components in the sample compositions 1-11 are as follows: the propylene-α-olefin copolymer is described in Table 1; the stabilizer is Irganox 225 (Ciba Specialty Co.); the nucleating agent was Hyperform HPN 68 (Milliken); the hPP(1) is a propylene homopolymer having an MFR (230°C, 2.16 kg) of 0.3 dg/min (Borealis); the hPP(2) is a propylene homopolymer PP 5341E1, having an MFR (230°C, 2.16 kg) of 0.8 (ExxonMobil Chemical Co.); rPP is Beta-PPR™ R 7050 propylene random copolymer having an MFR (230°C, 2.16 Kg) of 0.3 dg/min, a modulus of Elasticity of 900 MPa (Borealis); and sample 11 is a comparative example of RA130E-1498, a propylene random copolymer (Borealis) having a density of 0.908 g/cm^3 and a MFR of 0.25 dg/min and a Flexural Modulus (2 mm/min) of 800 MPa (Borealis). "Pe/Pa" means perpendicular to flow and parallel to flow, respectively. The plastomer is Exact™ 5062 (ExxonMobil Chemical Co.) with density of 0.860 g/cm^3 and I_2 = 0.5 dg/min (190°C, 2.16 Kg).

[0053] The test methods are as follows:

[0054] Hardness: ISO 868, 15 sec delay, 2 mm/30 mm disk (shore D) (sample size in thickness/diameter).

[0055] Melt flow Rate ("MFR"): ISO 1133, 230°C, 2.16 kg.

[0056] Tensile Strength, ISO 37, 500 mm/min, type 1 dumbbell cut out from injection molded plaque of 2 mm thick, 150 mm long and 100 mm wide (ISO plaque). In Tables 3-4, the first number gives cross to flow and second number gives parallel to flow readings.

[0057] Elongation at Break, ISO 37, 500 mm/min, dumbbell cut out flow from Injection molded plaque of 2 mm thick, 150 mm long and 100 mm wide (ISO plaque). In Tables 3-4, the first number gives cross to flow and second number gives parallel to flow readings.

[0058] Tensile Strength at Yield, ISO 37, 500 mm/min, dumbbell cut out cross to flow from injection molded plaque of 2 mm thick, 150 mm long and 100 mm wide (ISO plaque). In Tables 3-4, the first number gives cross to flow and second number gives parallel to flow readings.

[0059] Elongation at Yield, ISO 37, 500 mm/min, dumbbell cut out cross to flow from Injection molded plaque of 2 mm thick, 150 mm long and 100 mm wide (ISO plaque). In Tables 3-4, the first number gives cross to flow and second number gives parallel to flow readings.

[0060] Flexural Modulus, ISO 178, 1.6 mm/min, 80/10/4 mm sample size.

[0061] Izod Impact Energy, ISO 180, 80/10/4 mm sample size; nature of break, F = fragile, sample broke; D = ductile, sample did not break.

[0062] Charpy Impact Energy, ISO 179, 80/10/4 mm size; nature of break, F = fragile, sample broke; D = ductile, sample did not break.

[0063] Vicat Softening Temperature, ISO 306, 50°C/h, 10N, 1 cm^2/4 mm sample size.

[0064] Heat Deflection Temperature, ISO 75, 120°C/h, 120 mm/10 mm/4 mm sample size, the loading is 455 KPa edgewise.

[0065] Brittleness Temperature, TPE-0089, 2°C temperature increment, 2 mm ISO plaque.

Table 1. Properties of the propylene-α-olefin copolymer

Copolymer	Wt% C_2	MFR (230/2.16)	Density (g/cm^3)	Flexural Modulus (MPa)	Tensile at Break (MPa)	Shore A hardness	Vicat Softening Point (^oC)
A	16	3	0.858	11.9	11.8	64	48
B	11	2	0.873	46.5	> 18.6	80	68

Table 2. Pipe Compositions

Components (wt%)	1	2	3	4	5	6	7	8	9	10
hPP(1)	70	70	0	0	0	0	0	0	70	70
rPP1	0	0	80	80	70	0	0	0	0	0
hPP(2)	0	0	0	0	0	80	70	70	0	0
propylene-α-olefin copolymer A	30	20	20	0	30	20	30	20	30	0
propylene-α-olefin copolymer B	0	0	0	20	0	0	0	0	0	30
plastomer	0	10	0	0	0	0	0	10	0	0
stabilizer	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
nucleating agent	0	0	0	0	0	0	0	0	0.15	0

Table 3. Properties of Pipe Compositions

Property/Sample	1	2	3	4	5
Hardness, Shore D	57	59	60	60	53
MFR (230/2.16), dg/min	0.6	0.6	0.5	0.6	0.6
Tensile Strength, MPa (Pe/Pa)	18.7 / 30.5	18.3 / 31.4	25.0 / 32.0	36.8 / 26.2	27.4 / 20.8
Elongation at Break, % (Pe/Pa)	947 / 111	924 / 99	803 / 107	793 / 97	695 / 47
Tensile Strength at Yield, MPa (Pe/Pa)	18.7 / 30.5	18.3 / 31.3	18.5 / 25.0	20.5 / 26.2	14.5 / 20.8
Elongation at Yield, % (Pe/Pa)	5 / 27	8 / 31	7 / 18	20 / 12	10 / 22
Flexural Modulus, MPa	536	552	404	443	291
Izod Impact Energy (23°C), kJ/m^2	52.2	58.6	52.3	47.0	53.2
Isod Impact, nature of break (23°C)	D	D	D	D	D
Izod Impact Energy (0°C), kJ/m^2	41.9	65.9	37.1	6.9	52.7
Isod Impact, nature of break (0°C)	D	D	D	F	D
Izod Impact Energy (-10°C), kJ/m^2	7.5	31.6	4.3	-	25.6
Isod Impact, nature of break (-10°C)	F	D	F	-	F

Table 3. Properties of Pipe Compositions (Cont'd)

Property/Sample	1	2	3	4	5
Izod Impact Energy (-20°C), kJ/m^2	-	4.7	-	-	-
Isod Impact, nature of break (-20°C)	-	F	-	-	-
Charpy Impact Energy (23°C), kJ/m^2	49.3	49.3	49.3	48.8	49.2
Charpy Impact, nature of break (23°C)	D	D	D	D	D
Charpy Impact Energy (0°C), kJ/m^2	38.5	49.2	23.8	6.3	49.3
Charpy Impact, nature of break (0°C)	D	D	D	F	D
Charpy Impact Energy (-10°C), kJ/m^2	7.2	37.4	4.7	1.9	33.4
Charpy Impact, nature of break (-10°C)	F	F	F	F	F
Charpy Impact Energy (-20°C), kJ/m^2	-	-	-	-	-
Charpy Impact, nature of break (-20°C)	-	-	-	-	-
Vicat Softening Temperature, °C	132	136	120	124	112
Heat Deflection Temperature, °C	75	97	81	83	66
Brittleness Temperature, °C	-17	-21	-14	-8	-18

Table 4. Properties of Pipe Compositions

Property/Sample	6	7	8	9	10	11 (comparative)
Hardness, Shore D	64	60	59	63	58	61
MFR (230/2.16), dg/min	1.1	1.3	1.8	1.8	0.8	0.3
Tensile Strength, MPa (Pe/Pa)	22.6/32.7	19.8/30.5	18.1/29.8	22.3/32.6	24.0 / 23.8	25.0/29.4
Elongation at Break, % (Pe/Pa)	>1000 / 94	814/128	806/99	732/81	98 / 109	717/67
Tensile Strength at Yield, MPa (Pe/Pa)	22.6 / 32.7	19.8/30.5	18.7/29.8	22.3/32.6	24.0 / 23.8	25.0/29.4
Elongation at Yield, % (Pe/Pa)	10 / 20	13 / 32	11 / 28	7 / 20	20/20	11/17
Flexural Modulus, MPa	626.0	449	493	505	359	687
Izod Impact Energy (23°C), kJ/m^2	42.6	48.5	55.2	47.0	52.2	15.2
Isod Impact, nature of break (23°C)	D	D	D	D	D	F
Izod Impact Energy (0°C), kJ/m^2	7.6	14.9	26.1	7.4	8.5	4.3
Isod Impact, nature of break (0°C)	F	D	D	F	F	F
Izod Impact Energy (-10°C), kJ/m^2	-	6.0	6.5	-	-	-
Isod Impact, nature of break (-10°C)	-	F	F	-	-	-

Table 4. Properties of Pipe Compositions (cont'd)

Property/Sample	6	7	8	9	10	11 (comparative)
Izod Impact Energy (-20°C), kJ/m^2	-	-	-	-	-	-
Isod Impact, nature of break (-20°C)	-	-	-	-	-	-
Charpy Impact Energy (23°C), kJ/m^2	29.3	49.2	49.2	49.2	49.2	12.1
Charpy Impact, nature of break (23°C)	F	D	D	D	D	F
Charpy Impact Energy (0°C), kJ/m^2	-	14.5	22.7	6.3	9.3	-
Charpy Impact, nature of break (0°C)	-	D	D	F	F	-
Charpy Impact Energy (-10°C), kJ/m^2	-	5.9	6.5	1.8	2.8	-
Charpy Impact, nature of break (-10°C)	-	F	F	F	F	-
Charpy Impact Energy (-20°C), kJ/m^2	-	-	-	-	-	-
Charpy Impact, nature of break (-20°C)	-	-		-	-	-
Vicat Softening Temperature, °C	118	120	128	139	114	136
Heat Deflection Temperature, °C	55	80	73	84	75	78
Brittleness Temperature, °C	-9	- 15	- 29	- 11	-11	-

1. Having described the invention(s) in its various aspects, described as a first (1) embodiment is a pipe comprising:

(a) one or more propylene-α-olefin copolymers comprising propylene-derived units and from 5 to 35 wt%, by weight of the propylene-α-olefin copolymer, of ethylene-derived units and/or a C_4 to C_{10} α-olefin-derived unit, and having:

(i) a density from 0.850 to 0.920 g/cm^3;

(ii) an H_f of less than 75 J/g;

(iii) a T_m of less than 105°C; and

(b) at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof;

(c) wherein the pipe composition possesses a Flexural Modulus (23°C) less than 650 MPa, an Izod Impact (23°C) greater than 30 kJ/m^2, and a Vicat Softening Temperature greater than 110°C.

2. The pipe of numbered embodiment 1, wherein the composition consists essentially of the propylene-α-olefin copolymer and the propylene random copolymer, propylene block copolymer or propylene homopolymer.

3. The pipe of numbered embodiments 1 and 2, wherein the propylene-α-olefin copolymer possesses a triad tacticity by ^{13}C NMR of 75% or greater.

4. The pipe of any of the preceding numbered embodiments, wherein the propylene-α-olefin copolymer is a propylene-ethylene copolymer comprising from 8 to 20 wt%, based on the weight of the copolymer, of ethylene derived units.

5. The pipe of any of the preceding numbered embodiments, wherein the propylene random copolymer comprises from 0.1 to less than 5 wt%, by weight of the random copolymer, of ethylene-derived units or a C_4 to C_{10} α-olefin-derived unit.

6. The pipe of claim 1, wherein the propylene random copolymer possesses a MFR (230°C, 2.16 kg) of from 0.05 to 5 dg/min.

7. The pipe of any of the preceding numbered embodiments, wherein the propylene random copolymer has a T_m of greater than 135°C.

8. The pipe of any of the preceding numbered embodiments, wherein the random copolymer possesses a flexural modulus (23°C) of greater than 650 MPa.

9. The pipe of any of the preceding numbered embodiments, wherein the propylene-α-olefin copolymer makes up from 10 to 40 wt% of the composition, by weight of the composition.

10. The pipe of any of the preceding numbered embodiments, wherein the propylene-α-olefin copolymer makes up from 15 to 35 wt% of the composition, by weight of the composition.

11. The pipe of any of the preceding numbered embodiments, further comprising a plastomer.

12. The pipe of any of the preceding numbered embodiments, wherein the pipe composition has an Izod Impact (23°C, ISO 180) greater than 30 kJ/m^2.

13. The pipe of any of the preceding numbered embodiments, wherein the pipe composition has a Charpy Impact (23°C, ISO 179) greater than 15 kJ/m^2.

14. A method of forming a pipe of any of the preceding numbered embodiments comprising melt-blending the composition of the propylene-α-olefin copolymer and at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof; extruding the composition through a pipe-forming die; followed by isolating a pipe composition.

Also disclosed is the use of a pipe in transporting liquids, heated water in one embodiment, the pipe comprising (consisting of in a particular embodiment) a composition possessing a Flexural Modulus (23°C) less than 650 MPa, an Izod Impact (23°C) greater than 30 kJ/m^2, and a Vicat Softening Temperature greater than 110°C and comprising: (a) a propylene-α-olefin copolymer comprising propylene-derived units and from 5 to 35 wt%, by weight of the propylene-α-olefin copolymer, of ethylene-derived units or a C_4 to C_{10} α-olefin-derived unit, and having: (i) a density from 0.850 to 0.920 g/cm^3; (ii) an H_f of less than 75 J/g; and (iii) a T_m of less than 105°C; and (b) at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof.

CLAIMS

1. A pipe comprising a composition of:
   (a) one or more propylene-α-olefin copolymers comprising propylene-derived units and from 5 to 35 wt%, by weight of the propylene-α-olefin copolymer, of ethylene-derived units and/or C_4 to C_{10} α-olefin-derived units, and having:
      (i) a density from 0.850 to 0.920 g/cm^3;
      (ii) an H_f of less than 75 J/g;
      (iii) a T_m of less than 105°C; and
   (b) at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof;
   (c) wherein the pipe composition possesses a Flexural Modulus (23°C) less than 650 MPa, an Izod Impact (23°C) greater than 30 kJ/m^2, and a Vicat Softening Temperature greater than 110°C.

2. The pipe of claim 1, wherein the composition consists essentially of at least one propylene-α-olefin copolymer and at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof.

3. The pipe of claim 1, wherein the propylene-α-olefin copolymer possesses a triad tacticity by ^{13}C NMR of 75% or greater.

4. The pipe of claim 1, wherein the propylene-α-olefin copolymer is a propylene-ethylene copolymer comprising from 8 to 20 wt%, based on the weight of the copolymer, of ethylene derived units.

5. The pipe of claim 1, wherein the propylene random copolymer comprises from 0.1 to less than 5 wt%, by weight of the random copolymer, of ethylene-derived units or a C_4 to C_{10} α-olefin-derived unit.

6. The pipe of claim 1, wherein the propylene random copolymer possesses a MFR (230°C, 2.16 kg) of from 0.05 to 5 dg/min.

7. The pipe of claim 1, wherein the propylene random copolymer has a T_m of greater than 135°C.

8. The pipe of claim 1, wherein the random copolymer possesses a flexural modulus (23°C) of greater than 650 MPa.

9. The pipe of claim 1, wherein the propylene-α-olefin copolymer makes up from 10 to 40 wt% of the composition, by weight of the composition.

10. The pipe of claim 1, wherein the propylene-α-olefin copolymer makes up from 15 to 35 wt% of the composition, by weight of the composition.

11. The pipe of claim 1, further comprising a plastomer.

12. The pipe of claim 1, wherein the pipe composition has an Izod Impact (23°C, ISO 180) greater than 30 kJ/m^2.

13. The pipe of claim 1, wherein the pipe composition has a Charpy Impact (23°C, ISO 179) greater than 15 kJ/m^2.

14. A method of forming a pipe comprising melt-blending:
    (a) one or more propylene-α-olefin copolymers comprising propylene-derived units and from 5 to 35 wt%, by weight of the propylene-α-olefin copolymer, of ethylene-derived units and/or C_4 to C_{10} α-olefin-derived units, and having:
        (i) a density from 0.850 to 0.920 g/cm^3;
        (ii) an H_f of less than 75 J/g;
        (iii) a T_m of less than 105°C; and
    (b) at least one additional component selected from the group consisting of propylene random copolymers having a comonomer content of less than 5 wt%, a propylene block copolymer, propylene homopolymer and combinations thereof;
    (c) extruding the composition of (a) and (b) through a pipe-forming die; and
    (d) isolating a pipe possessing a flexural modulus (23°C) less than 650 MPa, an Izod Impact (23°C) greater than 30 kJ/m^2, and a Vicat Softening Temperature greater than 110°C.

15. The method of claim 14, wherein the composition consists essentially of the propylene-α-olefin copolymer and the propylene random copolymer, propylene block copolymer or propylene homopolymer.

16. The method of claim 14, wherein the propylene-α-olefin copolymer possesses a triad tacticity by ^{13}C NMR of 75% or greater.

17. The method of claim 14, wherein the propylene-α-olefin copolymer is a propylene-ethylene copolymer comprising from 8 to 20 wt%, based on the weight of the copolymer, of ethylene derived units.

18. The method of claim 14, wherein the propylene random copolymer comprises from 0.1 to less than 5 wt%, by weight of the random copolymer, of ethylene-derived units or a C_4 to C_{10} α-olefin-derived unit.

19. The method of claim 14, wherein the propylene random copolymer possesses a MFR (230°C, 2.16 kg) of from 0.05 to 5 dg/min.

20. The method of claim 14, wherein the propylene random copolymer has a T_m of greater than 135°C.

21. The method of claim 14, wherein the random copolymer possesses a flexural modulus (23°C) of greater than 650 MPa.

22. The method of claim 14, wherein the propylene-α-olefin copolymer makes up from 10 to 40 wt% of the composition, by weight of the composition.

23. The method of claim 14, wherein the propylene-α-olefin copolymer makes up from 15 to 35 wt% of the composition, by weight of the composition.

24. The method of claim 14, further comprising melt blending a plastomer with components (a) and (b).

25. The method of claim 14, wherein the pipe composition has an Izod Impact (23°C, ISO 180) greater than 30 kJ/m^2.

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2007/087624

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08L23/10

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)
C08L F16L

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 2006/173132 A1 (MEHTA ASPY K [US] ET AL) 3 August 2006 (2006-08-03) the whole document	1-25
X	WO 2006/065664 A (EXXONMOBIL CHEM PATENTS INC [US]; MEHTA ASPY K [US]; LI WEN [US]; DATT) 22 June 2006 (2006-06-22) the whole document	1-25
X	WO 2006/113132 A (EXXONMOBIL CHEM PATENTS INC [US]; DATTA SUDHIN [US]; HU WEIGUO [US]; K) 26 October 2006 (2006-10-26) the whole document	1-25

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

☒ See patent family annex.

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

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
23 September 2008	10/10/2008

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/US2007/087624

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