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(54) Title: HIGHLY ABRASION-RESISTANT IONOMER PIPES

(57) Abstract: A pipe or tube article is disclosed which comprises an innermost layer wherein the innermost layer has a thickness of about 6.3 to about 102 mm and comprises an ionomer composition; the ionomer composition has a melting point of about 80°C or higher. The article provides long lifetime, highly abrasion-resistant pipes for mining and other transportation uses. Methods for preparing the article and transporting abrasive materials through the article are also described.

HIGHLY ABRASION-RESISTANT IONOMER PIPES

The invention relates to highly abrasion-resistant tubular articles (pipes) comprising ionomer layers that provide for the transport of particulates and slurries, methods and compositions to produce the articles, and methods of transporting abrasive materials through them.

BACKGROUND OF THE INVENTION

Mining operations require the transport of highly abrasive particulate or slurry streams. The recovery of bitumen from oil sands is becoming increasingly important within the energy industry. Processing oil sand includes transporting and conditioning the oil sand as an aqueous slurry over kilometer lengths of pipe up to 1 meter in diameter. Processes for recovery of bitumen from oil sands are known (US Patents 4255433, 4414117, 4512956, 4533459, 5039227, 6007708, 6096192, 6110359, 6277269, 6391190, US2006/0016760, US2006/0249431, US2007/0023323, US2007/0025896, WO2006/060917, CA1251146, CA2195604, CA2227667, CA2420034, CA2445645, and CA2520943). Use of caustic to assist in the recovery process of oil from oil sands is also known (US2006/0016760 and US2006/0249431). Other mining operations that include the transport of highly abrasive particulate or slurry streams from the mine to processing refinery include, for example, iron ore, coal and coal dust, and the like, and in further non-mining transport processes, such as grain, sugar and the like.

Often, metal pipes, such as carbon steel or cast iron pipes, are used for the transport of these highly abrasive streams. They are expensive, heavy and only provide a temporary solution since they are eventually destroyed. To increase their lifetimes, the metal pipes may be rotated 90 degrees on their axes on a regular basis to provide a new transport surface. However, because of the pipe weight, this rotation is difficult and ultimately the entire pipe is worn out and must be replaced.

Use of plastic pipes, pipe liners and pipe coatings has been proposed to reduce these shortcomings. Material selection is critical.

Many of the commonly available materials cannot stand up to such highly-abrasive mining streams and are quickly worn out. For example, high density poly(ethylene) pipes are generally used as liners for sanitary sewer and wastewater pipelines but they rapidly degrade under highly abrasive environments. US4042559 discloses abrasive granule-filled, partially-cured coatings for use in abrasion resistant coated pipes for the transport of mining slurries. US4254165 discloses processes to produce abrasion resistant pipes with 0.04-0.05-inch thick coatings of filled (such as sand) polyolefins, such as low and medium density poly(ethylene) and including poly(ethylene-co-acrylic acid). US4339506, WO90/10032, and CA1232553 disclose rubber liners for pipes. US4215178 discloses fluoropolymer-modified rubber pipe liners. US2006/0137757 and US2007/0141285 disclose fluoropolymer pipe liners. Polyurethane pipe coatings are known (US3862921; US4025670, US2005/0194718, US2008/0174110, GB2028461, JP02189379, JP03155937, and JP60197770). US2005/0189028 discloses metal pipe coated with a polyurethane liner to transport tar sand slurry. GB2028461 discloses an abrasion-resistant pipe lining comprising a urethane rubber thermoset embedded with the particles of the material to be transported (coal dust, grain or sugar) through transport of the materials during curing. Abrasion resistant pipes with elastomeric polyurea coatings are disclosed in US6737134. A shortcoming of the polyurethane coatings includes the highly complex processes for applying the coating to the metal pipe.

Use of ionomer compositions as pipes, pipe liners and pipe coatings is known (e.g. JP2000179752, JP2000352480, JP2000352479, JP2002249750 disclosing 1.5 mm (0.05 inch) thick ionomer tubes for use as an anticorrosive lining for metal pipes designed for water service, wastewater; JP08011230 and JP08259704 disclosing heat-shrinkable, crosslinked ionomer tubes for the protection of pipes and cables; EP0586877 disclosing heat-shrinkable, crosslinked ionomer tubes with wall thicknesses of 1.5 mm; JP3700192 disclosing heat-shrinkable, foamed ionomer tubes; and JP2000179752 disclosing use of epoxy primers to adhere ionomer tubes to water service metal pipes).

US2006/0154011 and JP63051135 disclose poly(ethylene) blend pipes with a minor ionomer component. JP2000034415 discloses glass reinforced nylon pipes that include a minor ionomer component. Multilayer coextruded pipes with ionomer layers are known (EP209396;
5 JP2004114389; JP2004098515; JP2001041360; JP59131447; and JP59131448). JP3711305 discloses tubes made from ionomer compositions filled with 10-50 wt% inorganic fine-grain particles for use in lithium secondary batteries.

US3429954, US3534465, US2006/0108016, JP2002248707,
10 JP2002254493, JP2002257264, JP2002257265, JP2002327867, and US2005/0217747 disclose the use of poly(ethylene-co-(meth)acrylic acid) copolymers as adhesive layers to attach poly(ethylene) pipe liners to pipes. JP2002248707, JP2002254493, JP2002257264, JP2002257265, JP2002327867, JP2003294174, and US2005/0257848 disclose ionomers
15 as adhesive layers to attach poly(olefin) pipe liners to steel pipes.

Metal articles coated with ionomers are known (US Patents 3826628, 4049904, 4092452, 4371583, 4438162, 5496652, US2006/0233955; and WO00/10737). Ionomer powder coating compositions are known (US Patents 3959539, 5344883, 6132883,
20 6284311, 6544596 and 6680082). WO00/27892 discloses scratch and abrasion resistant ionomers neutralized with at least 2 metal ions for protective formulations. Acid copolymer powder coating compositions are known (US4237037 and US5981086). Metal articles powder coated with ionomers are known (US Patents 3991235, 4910046, 5036134, 5155162,
25 and 6284311). Metal powder coatings comprising anhydride-grafted polyolefins are disclosed in US4048355. Metal powder coatings comprising acid copolymers are disclosed in US4237037. Corrosion-resistant zinc metal-filled ionomer metal coatings are disclosed in US5562989. Corrosion-resistant zinc metal-filled acid-grafted polyolefin
30 metal coatings are disclosed in US5091260. JP61045514 discloses ionomer coatings for metal pipes. US4407893 discloses powder coating processes to produce abrasion resistant pipes with 0.04-inch thick coatings of sand-filled blends comprising polyethylenes and ionomers. Abrasion resistant ionomer coatings on glass articles are known (US

Patents 3836386, 3909487, 3922450, 3984608 and EP0798053).

Abrasion resistant ionomer coatings are disclosed in US2004/0115399 and US2007/0504331.

5 A shortcoming of prior ionomer pipes, pipe liners and pipe coatings with thicknesses of about 1.5 mm and less is their inability to withstand the desirable transport process temperatures and burst strengths. A further shortcoming of these ionomer pipes, pipe liners and pipe coatings is low abrasion resistance, resulting in short service lifetimes.

SUMMARY OF THE INVENTION

10 The invention is directed to a pipe- or tube-formed article having an innermost layer wherein the innermost layer may have a thickness of about 0.001 to about 102 mm or about 6.3 to about 102 mm comprising, or prepared from, an ionomer composition and the ionomer is made from an acid polymer comprising an α -olefin having 2 to 10 carbons, about 5 to
15 about 25 weight % of an α,β -ethylenically unsaturated carboxylic acid having 3 to 8 carbons, and optionally about 12 to about 60 wt% of an α,β -ethylenically unsaturated carboxylic acid ester, all based on the total weight of the acid polymer; and about 5 to about 90% of the carboxylic acids are neutralized with a metal ion.

20 The invention is also directed to a method comprising pulling or inserting an article into the interior surface of a metal pipe to produce an ionomer-lined metal pipe wherein the article is characterized above.

The invention also provides a method comprising laying up a film or sheet or comprising an ionomer composition into the interior surface of a
25 metal pipe; heating the metal pipe above the softening point of the ionomer composition; and allowing the metal pipe to cool to produce an ionomer-lined metal pipe wherein the ionomer is characterized above.

The invention also provides a method for transporting an abrasive material comprising obtaining a pipe- or tube-formed article as described
30 above; preparing an abrasive material composition suitable for flowing through the article; flowing the abrasive material composition into one end of the pipe- or tube-formed article and receiving the abrasive material composition out of the other end of pipe- or tube-formed article.

DETAILED DESCRIPTION OF THE INVENTION

Trademarks are in upper case.

The terms "comprises", "includes", "characterized by" or any other variation thereof, are intended to cover a non-exclusive inclusion. The phrase "consisting of" excludes any element, step, or ingredient not specified in the claim. The transitional phrase "consisting essentially of" limits the scope of a claim to the specified materials or steps and those that do not materially affect the basic and novel characteristic(s) of the claimed invention.

Preferred α -olefins include ethylene, propylene, 1-butene, 1-pentene, 1-hexene, 1-heptene, 3-methyl-1-butene, 4-methyl-1-pentene, and the like and mixtures thereof.

The α , β -ethylenically unsaturated carboxylic acid comonomers include but are not limited to acrylic acid, methacrylic acid, itaconic acid, maleic acid, maleic anhydride, fumaric acid, monomethyl maleic acid, and mixtures thereof.

The parent acid terpolymer may comprise 15 to 30 or 17 to 25 wt% of copolymerized units of the α , β -ethylenically unsaturated carboxylic acid ester, based on the total weight of the parent acid terpolymer including methyl acrylate, methyl methacrylate, ethyl acrylate, ethyl methacrylate, propyl acrylate, propyl methacrylate, isopropyl acrylate, isopropyl methacrylate, butyl acrylate, butyl methacrylate, isobutyl acrylate, isobutyl methacrylate, tert-butyl acrylate, tert-butyl methacrylate, octyl acrylate, octyl methacrylate, undecyl acrylate, undecyl methacrylate, octadecyl acrylate, octadecyl methacrylate, dodecyl acrylate, dodecyl methacrylate, 2-ethylhexyl acrylate, 2-ethylhexyl methacrylate, isobornyl acrylate, isobornyl methacrylate, lauryl acrylate, lauryl methacrylate, or mixtures thereof.

The parent acid terpolymer comprises about 7 to about 20 wt%, or more preferably about 8 to about 19 wt%, of copolymerized units of the α , β -ethylenically unsaturated carboxylic acid, based on the total weight of the parent acid terpolymer. Preferred α , β -ethylenically unsaturated carboxylic acid comonomers include acrylic acid, methacrylic acid, itaconic

acid, maleic acid, maleic anhydride, fumaric acid, monomethyl maleic acid, and mixtures thereof.

The acid copolymers may optionally further contain other unsaturated comonomers in from about 0.1 weight % to about 50 weight
5 %, or preferably to about 30 weight %, or more preferably to about 20 weight %, based on the total weight of the copolymers.

The parent acid copolymers may be polymerized as disclosed in US3404134, US5028674, US6500888, and US6518365.

The ionomers are neutralized from about 5 to about 90%, or
10 preferably, from about 10 to about 50%, or more preferably, from about 20 to about 40%, with metal ions, based on the total carboxylic acid content of the parent acid copolymers as calculated for the non-neutralized parent acid copolymers.

The metal ions may be monovalent, divalent, trivalent, multivalent,
15 or mixtures thereof including sodium, potassium, lithium, silver, mercury, copper, beryllium, magnesium, calcium, strontium, barium, copper, cadmium, mercury, tin, lead, iron, cobalt, nickel, zinc, aluminum, scandium, iron, yttrium, titanium, zirconium, hafnium, vanadium, tantalum, tungsten, chromium, cerium, iron, and the like, and mixtures thereof. It is
20 noted that when the metallic ion is multivalent, complexing agents such as stearate, oleate, salicylate, and phenolate radicals may be included, as disclosed in US 3404134.

The ionomer may have a melting point of about 80°C or higher, about 90°C or higher, or about 95°C or higher. The ionomer layer
25 provides the high thermal resistance to the pipe required by many demanding uses. The ionomer may have Shore D hardness (ASTM D2240, ISO 868) from about 30 to about 70, about 30 to about 60, about 40 to about 50, or about 60 to about 70.

Suitable ionomers are commercially available from
30 E. I. du Pont de Nemours and Company (DuPont), Wilmington, DE.

The compositions may be used with additives including plasticizers, processing aids, flow enhancing additives, lubricants, flame retardants, impact modifiers, nucleating agents to increase crystallinity, antiblocking agents such as silica, thermal stabilizers, UV absorbers, UV stabilizers,

dispersants, surfactants, chelating agents, coupling agents, adhesives, primers, melt-reducing additive, and the like. Typically the total amount of additives used in an ionomer composition is up to about 5 weight % (based upon the weight of the ionomer composition). Melt flow reducing
5 additives include organic peroxides. Alternative melt flow reducing additives include known peroxide-silanol additives that often include a peroxide, a silane and a catalyst. If desired, initiators, such as dibutyltin dilaurate, may also be present in the terionomer composition at a level of about 0.01 to about 0.05 wt%, based on the total weight of the terionomer
10 composition. Also if desired, inhibitors such as hydroquinone, hydroquinone monomethyl ether, p-benzoquinone, and methylhydroquinone, may be added for the purpose of enhancing control of the reaction and stability. The inhibitors may be added at a level of less than about 5 wt%, based on the total weight of the composition.

15 The ionomer composition may further comprise filler from 0.1 to 80 weight % based on the total weight of the filled composition. Preferably, the filler is abrasion-resistant filler, reinforcing filler, or a non-reinforcing filler. Fillers include high strength fibers prepared from materials selected from the group consisting of fiberglass, continuous
20 glass fiber, polyaramide fiber, KEVLAR (aramid fiber, a product of DuPont, one or more fibers made from one or more aromatic polyamides, wherein at least 85% of the amide (-CONH-) linkages are attached directly to two aromatic rings), graphite, carbon fiber, silica, quartz, ceramic, silicon carbide, boron, alumina, alumina-silica, polyethylene, ultrahigh molecular
25 weight polyethylene, polyimide, liquid crystal polymers, polypropylene, polyester, polyamide and the like. Examples of non-reinforcing fillers include particles of abrasion-resistant minerals, marble, slate, granite, sand, potters sand, silicates, limestone, clay, glass, quartz, metallic powders, aluminum powders, stainless steel powders, zinc metal,
30 refractory metal borides, carbides, nitrides, oxides, silicon carbide, alumina, fused combinations alumina and zirconia, calcium carbonate, barium sulfate, magnesium silicate and the like and combinations thereof.

The size of filler incorporated in the ionomer composition may depend on the thickness and diameter of the ionomer pipe and may be

smaller than the thickness of the ionomer pipe. A mixture of particle sizes may be used to provide a higher density of filler incorporated.

The article in the form of a pipe comprises an innermost layer having a thickness of about 6.3 to about 102 mm (about 0.25 to about 4 inches) comprising a di-ionomer (produced from α -olefin and unsaturated acid) or 0.001 to 102 mm (about 0.00004 to about 4 inches) of the terionomer (produced from α -olefin, unsaturated acid ester, and unsaturated acid) described above. The pipe may have a hollow circular profile and the wall thickness may be uniform around the circumference of the pipe, or the pipe may have any profile and the wall thickness may vary around the circumference of the pipe as desired. The ionomer is positioned as the innermost layer to provide desirably superior abrasion-resistance. The ionomer pipe thickness provides not only a long lifetime under extreme abrasive use conditions, but also provides desirable high burst strength under the high temperature conditions contemplated herein.

The ionomer layer may also have a thickness about of 3.2 to about 102mm, about 9.5 to about 76 mm, or about 13 to about 51 mm.

The ionomer pipe may have any dimensions (including outside diameter, inside diameter and length) required to meet the end use needs. For example but not limitation the ionomer pipe preferably has an outer diameter (OD) of about 2.54 to about 254 cm (about 1 to about 100 inches), more preferably about 25.4 to about 152 cm (about 10 to about 60 inches) and most preferably about 51 to about 102 cm (about 20 to about 40 inches). For example but not limitation the ionomer pipe preferably has a length of about 1.5 to about 12.2 m (about 5 to about 40 feet), more preferably about 3.1 to about 9.1 m (about 10 to about 30 feet) and most preferably about 5.5 to about 6.7 m (about 18 to 22 feet) to provide a convenient length for storage, transport, handling and installation.

The ionomer pipe may be produced by any suitable process. For example, the ionomer pipe may be formed by melt extrusion, melt coextrusion, slush molding, rotomolding, rotational molding or any other procedures known in the art. For example, the ionomer pipe may be

produced by rotational or slush molding processes. The ionomer composition may be in the form of powder, microbeads or pellets for use in rotational molding processes. Methods for rotational molding of pipes are known (e.g., US 4115508).

5 The article may be a multilayer pipe comprising an innermost layer of the ionomer disclosed above and an outside layer.

 Examples of preferred polymeric materials for the outside layer include rubbers, elastomers, thermoplastic elastomers, acid terpolymers, ionomer terpolymers and the like and combinations thereof. Rubbers and
10 elastomers may be categorized as diene elastomers, saturated elastomers, thermoplastic elastomers, or inorganic elastomers. These polymers are well known to one skilled in the art and the description of which is omitted here for the interest of brevity.

 The outer layer may have any thickness such as about 0.1 to about
15 102 mm (about 0.004 to about 4 inches), or about 1 to about 25.4 mm (about 0.04 to about 1 inch) or about 2.5 to about 12.7 mm (about 0.1 to about 0.5 inch).

 An intermediate layer or tielayer may be present between the outer layer and the innermost layer. Materials that may be used in tielayers
20 include anhydride- or acid-grafted materials. The preferred anhydrides and acids are α,β -ethylenically unsaturated carboxylic acid comonomers selected from the group consisting of acrylic acid, methacrylic acid, itaconic acid, maleic acid, maleic anhydride, fumaric acid, monomethyl maleic acid, and mixtures thereof. Most preferred acids and anhydrides
25 are selected from the group consisting of acrylic acid, maleic anhydride and mixtures thereof. Preferably, the materials to be grafted are selected from the preferred polymeric materials recited above.

 The outer layer may comprise fiber reinforcement and optionally a thermoset resin or thermoset resin.

30 The fiber reinforcement may be a filament, warp yarn, tape, unidirectional sheet, mat, cloth, knitted cloth, paper, non-woven fabric or woven fabric, or mixtures thereof. The fiber preferably comprises a high strength fiber such as fiberglass, continuous glass fiber, polyaramide fiber, aramid fiber, graphite, carbon fiber, silica, quartz, ceramic, silicon carbide,

boron, alumina, alumina-silica, polyethylene, ultrahigh molecular weight polyethylene, polyimide, liquid crystal polymers, polypropylene, polyester, polyamide and the like, and is preferably about 3 to about 30 μm thick.

The fiber may be impregnated with a resin ("prepreg"), such as thermoplastic or preferably thermoset resins. Suitable resins for impregnating the fiber layers include polyester, aromatic, aliphatic, cycloaliphatic or anhydride epoxy resins, vinylester, vinyl, acrylic, modified acrylic, urethane, phenolic, polyimide, bismaleimide, polyurea, siloxane-modified resins and the like and combinations thereof.

Fiber-reinforcement of thermoplastic pipe is known (e.g., US4081302, 4521465, 5629062, 5931198, 6737134, and 7018691; US2006/0151042; and WO2004/068016).

An adhesive may be applied to the ionomer pipe and multilayer ionomer pipe prior to the application of the exterior reinforcement layer and/or an adhesive may be applied to the reinforcement layer after its application to the ionomer pipe and multilayer ionomer pipe. The exterior surface of the ionomer pipe and multilayer ionomer pipe may be heated to enhance the adhesion and/or embedding of the reinforcement layer. Suitable adhesives may include the impregnated resins described above or any adhesive known in the art.

The fiber reinforcement may be applied to the ionomer pipe and multilayer ionomer pipe by any known method. For example, the fiber reinforcement may be applied using known filament winding processes through winding the fiber reinforcement onto the ionomer pipe and multilayer ionomer pipe or by wrapping the fiber reinforcement around the ionomer pipe and multilayer ionomer pipe.

The article may be in the form of a multilayer pipe comprising an innermost layer comprising the ionomer and an outer layer comprising a metal, preferably in the form of a metal pipe.

A monolayer or multilayer ionomer (such as in the form of pipe, film, or sheet) may be attached (adhered) to the metal outer layer or not attached. The ionomer or multilayer ionomer may be self-adhered to the metal layer or adhered through the use of an adhesion primer, coating, or layer. As used herein, when the ionomer composition is said to be "self-

adhered" to the metal layer, it is meant that there is no intermediate layer such as a primer or thin adhesive layer between the metal and the ionomer or multilayer ionomer composition. The ionomer compositions described herein have the advantage of forming high adhesion to the metal pipe.

The pipe may comprise an innermost layer comprising the ionomer composition; an intermediate layer comprising a polymer material (such as those polymeric materials described above); and an outer layer comprising metal.

The pipe may comprise an innermost layer comprising the ionomer composition; an intermediate layer comprising a polymer material; and an outer layer comprising metal, wherein the ionomer layer is adhered to the polymer material layer and the polymer material layer is adhered to the metal layer.

The pipe may comprise an innermost layer comprising the ionomer composition; an intermediate layer comprising a polymer material; and an outer layer comprising metal, wherein the ionomer layer is self-adhered to the polymer layer and the polymer layer is self-adhered to the metal layer.

The pipe may further comprise an intermediate layer comprising a fiber reinforcement material comprising a high strength fiber and optionally a thermoset resin as described above.

The metal pipe may comprise carbon steel, steel, stainless steel, cast iron, galvanized steel, aluminum, copper and the like to provide physical properties for the material conveying processes contemplated.

The metal pipe may have any dimensions, including thickness, outer diameter, inner diameter and length suitable for the intended use. The pipe may have a hollow, substantially circular profile and the wall thickness may be generally uniform around the circumference of the pipe, or the pipe may have any profile and the wall thickness may vary around the circumference of the pipe as desired. For example, the metal pipe may have a thickness of about 6.3 to about 51 mm (about 0.25 to about 2 inches, about 9.5 to about 38 mm (about 0.375 to about 1.5 inches) or about 13 to about 25.4 mm (about 0.5 to about 1 inch). The metal pipe may have an outer diameter (OD) of about 5.1 to about 254 cm (about 2 to

about 100 inches), about 25.4 to about 152 cm (about 10 to about 60 inches) or about 51 to about 102 cm (about 20 to about 40 inches). The metal pipe may have a length of about 1.5 to about 12.2 m (about 5 to about 40 feet), about 3.1 to about 9.1 m (about 10 to about 30 feet) or
5 about 5.5 to about 6.7 m (about 18 to 22 feet) to provide a convenient length for storage, transport, handling and installation.

The ionomer-lined metal pipe may be produced by any known method where the ionomer pipe may serve as a liner for a metal pipe. Methods for lining a pipe with a polymeric liner are known (e.g., US
10 Patents 3315348, 3429954, 3534465, 3856905, 3959424, 4207130, 4394202, 4863365, 4985196, 4998871, 5072622, an 6723266; US2006/0093436; US2006/0108016; US2006/0124188; US2006/0151042; and EP0848659).

The inside surface of the metal pipe may be pretreated to provide
15 enhanced adhesion and stability. Such treatments include descaling by sand-, metal grit- or shot-blasting, acid etching, cleaning the metal surface through solvent or chemical washes to remove grease and/or oxide layers, and the application of adhesion primers, coatings, or layers.

An ionomer-lined metal pipe may be prepared by pulling or inserting
20 a preformed ionomer pipe or multilayer ionomer pipe comprising an innermost layer having a thickness of about 6.3 to about 102 mm comprising an ionomer composition as described above into a preformed metal pipe wherein the outer diameter of the ionomer pipe is less than the interior diameter of the metal pipe. This method to produce an ionomer-
25 lined metal pipe includes the following embodiments.

A: (i) pulling or inserting a pre-formed ionomer pipe or multilayer ionomer pipe into the metal pipe; (ii) heating the ionomer-lined metal pipe above the softening point of the ionomer composition; and (iii) allowing the metal pipe to cool.

30 B: (i) heating a metal pipe above the softening point of the ionomer composition; (ii) pulling or inserting a pre-formed ionomer pipe or multilayer ionomer pipe into the heated metal pipe; and (iii) allowing the metal pipe to cool.

C: (i) coating a layer of an adhesive or adhesion primer onto the outside surface of the ionomer pipe or multilayer ionomer pipe; and (ii) pulling or inserting the adhesive-treated ionomer pipe or multilayer ionomer pipe into the metal pipe.

5 D: (i) coating a layer of an adhesive or adhesion primer onto the inside surface of the metal pipe; and (ii) pulling or inserting the ionomer pipe or multilayer ionomer pipe into the adhesive-treated metal pipe.

E: (i) coating a layer of an adhesive or adhesion primer onto the outside surface of the ionomer pipe or multilayer ionomer pipe; (ii) pulling
10 or inserting the adhesive-treated ionomer pipe or multilayer ionomer pipe into the metal pipe; (iii) heating the metal pipe above the softening point of the ionomer composition; and (iv) allowing the metal pipe to cool.

F: (i) coating a layer of an adhesive or adhesion primer onto the inside surface of the metal pipe; (ii) pulling or inserting the ionomer pipe or
15 multilayer ionomer pipe into the adhesive-treated metal pipe; (iii) heating the metal pipe above the softening point of the ionomer composition; and (iv) allowing the metal pipe to cool.

G: (i) coating a layer of an adhesive or adhesion primer onto the outside surface of the ionomer pipe or multilayer ionomer pipe; (ii) heating
20 a metal pipe above the softening point of the ionomer composition; (iii) pulling or inserting the adhesive-treated ionomer pipe or multilayer ionomer pipe into the heated metal pipe; and (iv) allowing the metal pipe to cool.

H: (i) coating a layer of an adhesive or adhesion primer onto the
25 inside surface of the metal pipe; (ii) heating the adhesively-treated metal pipe above the softening point of the ionomer composition; (iii) pulling or inserting the ionomer pipe or multilayer ionomer pipe into the heated metal pipe; and (iv) allowing the metal pipe to cool.

In a specific embodiment, the method for adhering the ionomer pipe
30 or multilayer ionomer pipe to the metal pipe comprises (a) descaling and cleaning the interior surface of the metal pipe; (b) heating the metal pipe to a temperature of about 150 to about 400°C, preferably about 150 to about 300°C and most preferably of about 175 to about 225°C; (c) pulling or inserting the ionomer liner (pipe) or ionomer multilayer liner (pipe) into the

hot metal pipe; and (d) allowing the ionomer-lined metal pipe to cool to ambient conditions.

For example, preparing an ionomer lined metal pipe method with a self-adhered liner (pipe) includes descaling, degreasing and cleaning as described above. The metal pipe is then heated, as in an oven, a furnace, a gas ring burner, electrical resistive heating elements, radiant heaters, induction heating, high frequency electrical heaters and the like, and the heating may be discontinued throughout the remainder of the process or the metal pipe may be continuously heated, as through induction heating, throughout the process. The heating expands the metal pipe. An ionomer liner (pipe) or ionomer multilayer liner (pipe) is pulled or inserted into the hot metal pipe. The ionomer and multilayer ionomer liner preferably has an OD that is no greater than about 0.1 inch (2.5 mm) less than the inside diameter (ID) of the unheated metal pipe, more preferably an OD no greater than about 1.3 mm less than the ID, even more preferably, an OD no greater than about 0.64 mm less than the ID. Most preferably, the ionomer and multilayer ionomer liner OD is about equivalent to the ID of the unheated metal pipe. As the heated metal pipe-ionomer liner structure cools, the metal pipe reduces in diameter and makes intimate contact with the outside surface of the ionomer liner, causing it to soften and self-adhere to the inside surface of the metal pipe. Alternatively, the ionomer liner (pipe) or multilayer ionomer liner (pipe) may be inserted into the metal pipe prior to heating.

If desired, prior to heating the metal pipe and inserting the ionomer and multilayer ionomer liner (pipe), an adhesive primer, coating or layer may be applied to the interior surface of the metal pipe, the exterior surface of the ionomer and multilayer ionomer liner or both, in the form of a solution or solid to provide enhanced interlayer adhesion.

A method to produce an ionomer-lined metal pipe comprises laying up a pre-formed ionomer film or sheet or multilayer ionomer film or sheet into a preformed metal pipe. This method to produce an ionomer-lined metal pipe includes; A: (i) laying up the interior of a metal pipe with ionomer film or sheet or multilayer ionomer film or sheet; (ii) heating a metal pipe above the softening point of the ionomer composition; and

(iii) allowing the metal pipe to cool; B: (i) coating a layer of an adhesive or adhesion primer onto the outside surface of the ionomer film or sheet or multilayer ionomer film or sheet; and (ii) laying up the interior of a metal pipe with ionomer film or sheet or multilayer ionomer film or sheet; C: 5 (i) coating a layer of an adhesive or adhesion primer onto the inside surface of the metal pipe; and (ii) laying up the interior of a metal pipe with ionomer film or sheet or multilayer ionomer film or sheet; or D: (i) coating a layer of an adhesive or adhesion primer onto the outside surface of the ionomer film or sheet or multilayer ionomer film or sheet; (ii) laying up the 10 interior of a metal pipe with ionomer film or sheet or multilayer ionomer film or sheet; (iii) heating a metal pipe above the softening point of the ionomer composition; and (iv) allowing the metal pipe to cool.

The ionomer film or sheet and the multilayer ionomer film or sheet may be produced by any art method such as through melt processes 15 extrusion blown film processes, extrusion film or sheet melt casting processes, sheet profile extrusion processes, calendar processes and the like. The films and sheets may undergo secondary formation processes, such as the plying together of preformed sheets to produce thicker sheets through known calendaring processes.

20 An example ionomer lined metal pipe method with a self-adhered ionomer sheet includes descaling the interior of the metal pipe, followed by degreasing and cleaning. The interior of the metal pipe is then covered with the ionomer sheet, preferably with the sheet overlapping onto itself 0.5 to 4 inches to form a seam. The seam may be heat fused or the 25 excess sheet may be trimmed and the sheet ends may be heat fused, as desired. The metal pipe is then heated, as described above, to the temperature range of 150 to 400°C, 150 to 300°C, or 175 to 225°C. As the heated metal pipe-ionomer sheet structure cools, the metal pipe makes intimate contact with the outside surface of the ionomer sheet, 30 causing it to soften and self-adhere to the inside surface of the metal pipe.

If desired, prior to heating the metal pipe and inserting the ionomer and multilayer ionomer film or sheet, an adhesive primer, coating or layer may be applied to the interior surface of the metal pipe, the exterior

surface of the ionomer and multilayer ionomer film or sheet or both, in the form of a solution or solid to provide enhanced interlayer adhesion.

The ionomer-lined metal pipe may be produced by powder coating processes. Methods for coating the inner or outer surfaces of a pipe with polymeric powder coatings are known (US Patents 3,004,861; 3,016,875; 3,063,860; 3,074,808; 3,138,483; 3,186,860; 3,207,618; 3,230,105; 3,245,824; 3,307,996; 3,488,206; 3,532,531; 3,974,306; 3,982,050; 4,007,298; 4,481,239; and EP778088).

The ionomer composition may be produced in the form of a powder by any known method (e.g., US Patents 3933954, 3959539, 4056653, 4237037, 5344883, 6107412, 6132883, 6284311, 6544596, 6680082, and EP1169390). Preferably, the ionomer composition is cryogenically (for example, with liquid nitrogen as the cooling medium) ground into a powder. Physically grinding the ionomer composition creates irregularly shaped particles of size and shape suitable for achieving constant flow through the application equipment. Preferably, the ionomer composition powder may have a particle size or average particle size of about 20 to about 500 μm . The grinding step may include a sieving or classification step to eliminate large- and fine-sized particles. For fluid bed coating processes, the preferred particle size is of about 75 to about 350 μm .

A method to produce a ionomer-lined metal pipe comprises (i) heating a metal pipe above the softening point of an ionomer composition; (ii) fluidizing the ionomer composition in the form of a powder; (iii) supplying the fluidized ionomer composition powder to the inside of the heated metal pipe until the desired ionomer thickness is achieved; and (iv) allowing the metal pipe to cool.

The heated metal pipe may be in a vertical orientation during step (iii); or the heated metal pipe may be in a horizontal orientation during step (iii). In another embodiment, the heated metal pipe may be rotated during step (iii). For example, the heated metal pipe may be rotated at a rate to force the ionomer composition powder to the inside diameter of the metal pipe during step (iii).

The powder coating process may comprise heating the metal pipe to a temperature above the softening point of the ionomer composition and supplying a fluidized powder of the ionomer composition into the heated pipe for a time sufficient to provide the desired ionomer coating thickness.

- 5 The metal pipe is preferably heated to the temperature range of 150 to 400°C, 200 to 350°C or 250 to 300°C. The metal pipe may be heated as described above and the heating may be discontinued throughout the remainder of the process or the metal pipe may be continuously heated throughout the process. In addition, portions of the pipe may be heated.
- 10 For example, in a fluidized bed method (see below) the metal pipe may be incrementally heated from the top to the bottom to cause the coating to form sequentially from the top to the bottom. Conversely, the metal pipe may be heated from the bottom to the top.

- 15 The ionomer coating may be self-adhered to the metal pipe or the interior surface of the metal pipe may be treated with adhesion primers, coatings and layers. The use of adhesion promoting primers and coupling agents for pipe powder coatings is known (US Patents 3016875, 4048355; and 4481239).

- 20 Pipe powder coating methods may include descaling, degreasing and cleaning as described above. The portions of the pipe which are not desired to be coated, for example the metal pipe ends which are meant to be joined together to form the pipeline, may be masked. If desired, prior to feeding the powder, an adhesive primer, coating or layer may be applied to the interior surface of the metal pipe in the form of a solution or solid
- 25 (powder) to provide enhanced interlayer adhesion. The metal pipe is then heated as described above. Preferably, the heated metal pipe may be rotated about its cylindrical axis at a rate of about 1 to about 300 rpm, more preferably about 10 to about 80 rpm. The metal pipe may be rotated slowly to provide good, even coverage of the powder coating or may be
- 30 rotated fast enough to force the powder to the interior surface of the pipe. The metal pipe may be in a vertical orientation or preferably in a horizontal orientation. If a multilayer coating is desired, different polymeric composition powders may be fed sequentially to provide the different coating layers at the thickness desired. At any stage of the process,

abrasion-resistant particles, such as described above as fillers, may be fed into the interior of the metal pipe, either individually or in combination with the powder. For example, the abrasion-resistant particles may be overcoated onto the hot coating while it is still soft and tacky so that the particles adhere to the interior surface of the coating. The coated metal pipe is then allowed to cool to ambient temperatures. If desired, any coating surface roughness may be smoothed through a post-coating operation, such as by hot gas, flame or oven post-treatments.

In a fluidized bed method, the powder is fed with pressurized gas, such as compressed air, nitrogen or argon, from a fluidized bed of the powder into the interior of the hot metal pipe. Alternatively, the hot metal pipe may be placed above the fluidized bed and the fluidized bed allowed to expand into the interior of the hot metal pipe to be coated. As the powder contacts the heated interior surface of the metal pipe, the material coalesces and flows to form a continuous, fused coating. The powder is fed from the fluidized bed until a continuous, uniform coating of the desired thickness is achieved.

In a spray coating method, a spray nozzle, preferably with a deflector disc to force the powder radially out onto the metal pipe interior surface, supported on an extensible boom, is inserted down the centerline of the metal pipe interior. The powder may be fed with pressurized gas, such as compressed air, nitrogen or argon, from a fluidized bed of the powder. Alternatively, the powder may be delivered from a bin to a vibrating feeder into a hopper and then conveyed to the spray nozzle with a pressurized gas. During the coating operation, the spray nozzle, the metal pipe or both may be moved to ensure uniform coating over the interior surface of the pipe. Multiple coats may be applied to provide the desired coating thicknesses.

The ionomer composition powder may be applied to the inside metal pipe surface through electrostatic spraying processes. For electrostatic spraying applications, the preferred particle size is about 20 to about 120 μm . Preferably, the metal pipe is preheated above the softening point of the ionomer composition as described above. In

electrostatic spraying processes, the ionomer powder is fed out of a reservoir, such as a fluidized bed, to a spray gun by air pressure.

The ionomer composition coating may be applied to the metal pipe by thermal spraying processes, such as flame (combustion) spraying, two wire arc spraying, plasma spraying, cold spraying and high velocity oxy-fuel spraying. Preferably, the thermal spraying process is a flame spraying process. The ionomer composition may be in the form of a wire or a rod to serve as a feedstock for flame spraying processes, or it is a powder with a preferred particle size of about 1 to about 50 μm . The ionomer powder may be fed to the flame spraying gun in a stream of an inert gas (such as argon or nitrogen) and fed into a flame of a fuel gas (such as acetylene or propane) and oxygen. The ionomer powder is melted in the flame and with the help of a second outer annular gas nozzle of compressed air is sprayed onto the cleaned inside surface of the preheated metal pipe to form the ionomer coating.

The ionomer compositions may be too soft for the formation of suitable powder to support powder-based processes. Powder-based processes to produce the pipe are therefore not preferred.

The ionomer-lined metal pipe may be produced by processes similar to the above by rotational or slush molding processes. The ionomer composition may be in the form of powder, microbeads or pellets. The coating process comprises heating the metal pipe to a temperature above the softening point of the ionomer composition, horizontally rotating the pipe and supplying the ionomer composition into the heated pipe for a time sufficient to provide the desired ionomer coating thickness. The metal pipe may be preheated (such as in an oven), may be constantly heated during the process or both. The ionomer composition may be fed all at once, batchwise or continuously to the rotating heated metal pipe. After an even coating of the desired thickness of the ionomer composition is applied to the inner diameter of the metal pipe, the pipe is cooled.

The pipes may provide high abrasion-resistance and corrosion resistance for the conveyance of solids and slurries such as found in the agriculture, food and mining industries. The ionomer layer in the pipes provides very long lifetime, desirable for those industries that require long

Melt Index (MI) was measured by ASTM D1238 at 190°C using a 2160 g mass, unless indicated otherwise. A similar ISO test is ISO 1133. Shore D hardness was measured according to ASTM D2240, ISO 868.

MATERIALS USED

- 5 ION 1: a poly(ethylene-co-methacrylic acid) with 15 wt % methacrylic acid, partially neutralized with about 27 % zinc ions, with MI of about 2 g/10 min.
- ION 2: a poly(ethylene-co-methacrylic acid) with 19 wt % methacrylic acid, partially neutralized with about 37 % zinc ions, with MI of 1 g/10 min.
- ION 3: a poly(ethylene-co-methacrylic acid) with 15 wt % methacrylic acid,
10 partially neutralized with zinc ions, with MI of 5 g/10 min.
- ION 4: a poly(ethylene-co-methacrylic acid) with 10 wt % methacrylic acid, partially neutralized with about 30 % of a mixture of zinc ions and sodium ions in a 75:25 molar ratio, with MI of about 1 g/10 min.
- ION 4: a poly(ethylene-co-methacrylic acid) with 15 wt % methacrylic acid
15 neutralized with approximately 58 % zinc ions; MI of approximately 0.7 g/10 min (2160 g, 190°C, ISO 1133, ASTM D1238); and Shore D hardness of 64 (ASTM D2240, ISO 868).
- ION 5: a poly(ethylene-co-methacrylic acid) with 15 wt % methacrylic acid, partially neutralized with about 35 % of a mixture of zinc ions and sodium
20 ions in a 50:50 molar ratio, with MI of about 5 g/10 min.
- ION 6: a poly(ethylene-co-methacrylic acid) with 19 wt % methacrylic acid, partially neutralized with about 37 % of a mixture of zinc ions and sodium ions in a 75:25 molar ratio, with MI of 2 g/10 min.
- ION 7: a filled composition of 50 wt % ION 1 and 50 wt % sand based on
25 the total weight of the composition.
- ION 8: a filled composition of 25 wt % ION 4 and 75 wt % silica based on the total weight of the composition.
- ION 9: a filled composition of 75 wt % ION 5 and 25 wt % marble dust based on the total weight of the composition.
- 30 ION 10: an ionomer powder comprising a poly(ethylene-co-methacrylic acid) copolymer with 10 wt % methacrylic acid neutralized with about 20 % zinc ions and MI of about 50 g/10 min with an average particle size of about 250 μm .

ION 11: an ionomer powder comprising a poly(ethylene-co-methacrylic acid) copolymer with 15 wt % methacrylic acid neutralized with about 30 % zinc ions and MI of about 35 g/10 min with an average particle size of about 200 microns.

- 5 ION 12: an ionomer powder comprising a poly(ethylene-co-acrylic acid) copolymer with 15 wt % acrylic acid neutralized with about 40 % zinc ions and MI of about 15 g/10 min with an average particle size of about 225 microns.

- 10 ION 13: an ionomer powder comprising a poly(ethylene-co-methacrylic acid) copolymer with 14 wt % methacrylic acid, neutralized with about 25 % of a mixture of zinc ions and sodium ions in 75:25 molar ratio and MI of about 25 g/10 min with an average particle size of about 250 microns.

- 15 ION 14: an ionomer powder comprising a poly(ethylene-co-methacrylic acid) copolymer with 15 wt % methacrylic acid neutralized with about 30 % of a mixture of zinc ions and sodium ions in 50:50 molar ratio and MI of about 35 g/10 min with an average particle size of about 200 microns.

- 20 ION 15: an ionomer powder comprising a poly(ethylene-co-methacrylic acid) copolymer with 18 wt % methacrylic acid neutralized with about 40 % of a mixture of zinc ions and sodium ions in 25:75 molar ratio and MI of about 10 g/10 min with average particle size of about 225 microns.

ION 16: a filled composition of 50 wt % ION 11 and 50 wt % sand based on the total weight of the composition.

ION 17: a filled composition of 25 wt % ION 13 and 75 wt % silica based on the total weight of the composition.

- 25 ION 18: a filled composition of 75 wt % ION 14 and 25 wt % marble dust based on the total weight of the composition.

ION 19: a poly(ethylene-co-methacrylic acid) with 15 wt % methacrylic acid, partially neutralized with about 58 % zinc ions with MI of about 0.7 g/10 min and Shore D hardness of 64.

- 30 ION 21: a poly(ethylene-co-isobutylacrylate-co-methacrylic acid) containing 10 weight% isobutylacrylate and 10 weight % methacrylic acid based on the total weight of the parent acid terpolymer, partially neutralized with about 36 % sodium ions, with an MI of about 1 g/10 min and Shore D hardness of 56.

ION 22: a poly(ethylene-co-n-butylacrylate-co-methacrylic acid) containing 17 weight% n-butylacrylate and 10 weight % methacrylic acid based on the total weight of the parent acid terpolymer, partially neutralized with about 49 % sodium ions with an MI of about 1 g/10 min and Shore D
5 hardness of 39.

ION 23: a poly(ethylene-co-n-butylacrylate-co-methacrylic acid) containing 23.5 weight% n-butylacrylate and 9 weight % methacrylic acid based on the total weight of the parent acid terpolymer, partially neutralized with about 52 % sodium ions with an MI of about 1 g/10 min and a Shore D
10 hardness of 36.

ION 25: a poly(ethylene-co-isobutylacrylate-co-methacrylic acid) containing 10 weight% isobutylacrylate and 10 weight % methacrylic acid based on the total weight of the parent acid terpolymer, partially neutralized with about 73 % zinc ions with an MI of about 1 g/10 min and a
15 Shore D hardness of 55.

ION 26: a poly(ethylene-co-n-butylacrylate-co-methacrylic acid) containing 23.5 weight% n-butylacrylate and 9 weight % methacrylic acid based on the total weight of the parent acid terpolymer, partially neutralized with about 51 % zinc ions with an MI of about 0.6 g/10 min and a Shore D
20 hardness of 40.

ION 27: a poly(ethylene-co-n-butylacrylate-co-methacrylic acid) having 23.5 weight % n-butylacrylate and 9 weight % methacrylic acid based on the total weight of the parent acid terpolymer, partially neutralized with about 49 % magnesium ions, with MI of about 1 g/10 min and Shore D
25 hardness of 43.

ION 28: a poly(ethylene-co-iso-butylacrylate-co-methacrylic acid) containing 20 weight% iso-butylacrylate and 15 weight % methacrylic acid, partially neutralized with about 27 % zinc ions with an MI of about 2 g/10 min.

30 ION 29: a poly(ethylene-co-methylacrylate-co-acrylic acid) containing 25 weight% methylacrylate and 10 weight % acrylic acid, partially neutralized (about 30 %) with a mixture of zinc ions and sodium ions in a 75:25 molar ratio with an MI of about 1 g/10 min.

ION 30 is a poly(ethylene-co-n-butylacrylate-co-methacrylic acid) containing 23.5 weight% n-butylacrylate and 9 weight % methacrylic acid based on the total weight of the parent acid terpolymer, partially neutralized (about 35 %) with a mixture of zinc ions and sodium ions in a 50:50 molar ratio with an MI of about 5 g/10 min.

ION 31 is a poly(ethylene-co-n-butylacrylate-co-methacrylic acid) containing 17 weight% n-butylacrylate and 19 weight % methacrylic acid, partially neutralized with about 37 % of zinc ions with an MI of 2 g/10 min.

ION 32: a filled composition of 50 wt% ION 6 and 50 wt% sand based on the total weight of the composition.

ION 33: a filled composition of 25 wt% ION 7 and 75 wt% silica based on the total weight of the composition.

ION 34: a filled composition of 75 wt% ION 6 and 25 wt% marble dust based on the total weight of the composition.

ACR: a poly(ethylene-co-n-butylacrylate-co-methacrylic acid) containing 23 wt % n-butylacrylate and 9 wt % methacrylic acid having a MI of 5 g/10 min.

EO: a metallocene-catalyzed ethylene-octene copolymer plastomer, sold as EXACT 5361 by the ExxonMobil Chemical Company (ExxonMobil), Houston, TX.

EP 1: a metallocene-catalyzed ethylene-propylene copolymer, VISTALON EPM 722 (ExxonMobil).

EP 2: a metallocene-catalyzed copolymer VISTAMAXX VM1100, ExxonMobil.

EP 3: EP2 grafted with 2 wt % maleic anhydride.

EPDM: a metallocene-catalyzed ethylene-propylene-diene copolymer, sold as VISTALON 5601 (ExxonMobil).

HDPE 1: a high density poly(ethylene) (HDPE)

HDPE 2: an HDPE grafted with 1.5 wt % maleic anhydride.

S: a styrene block copolymer sold as KRATON G7705-1 by Kraton Polymers (Kraton), Houston, TX.

SBS: a styrene-butadiene-styrene block copolymer with a MI of 3 g/10 min at 200 °C/5 kg, sold as KRATON D1153E (Kraton).

SEBS 1: a styrene-ethylene/styrene block copolymer with a MI of 5 g/10 min at 230 °C/5 kg, sold as KRATON G1652M (Kraton).

SEBS 2: a styrene-ethylene/styrene block copolymer grafted with 1.7 wt % maleic anhydride, sold as KRATON FG1901X (Kraton).

5 SEBS 3: a styrene-ethylene/styrene block copolymer grafted with 1 wt % maleic anhydride and is sold as KRATON FG1924X (Kraton).

SIS: a styrene-isoprene-styrene block copolymer with a MI of 3 g/10 min at 200°C/5 kg, sold as KRATON D111K (Kraton).

10 TI: a poly(ethylene-co-n-butylacrylate-co-methacrylic acid) containing 23 wt % n-butylacrylate and 9 wt % methacrylic acid that is 40% neutralized with zinc ions and having a MI of 2.5 g/10 min.

Thickness and diameter in the following tables, unless specifically indicated, are in inches (1 inch = 2.54 cm).

Examples 1–9

15 The ionomer pipes summarized in Table 1 are made from the materials listed by conventional pipe extrusion and sizing methods with melt extrusion temperatures from about 225°C to about 250°C. The pipes are cut into 20 foot lengths. OD = outer diameter.

Table 1

Example	Material	Outer Diameter	Thickness
1	ION 1	20	0.5
2	ION 2	24	1.0
3	ION 3	28	2.0
4	ION 4	22	0.38
5	ION 5	26	0.75
6	ION 6	32	1.5
7	ION 7	26	0.4
8	ION 8	30	1.0
9	ION 9	34	1.8

20

Examples 10–15

The bilayer ionomer pipes described in Table 2 are made from the materials summarized in Table 2 through conventional multilayer pipe extrusion and sizing methods with melt extrusion temperatures about 225°C to about 250°C. The pipes are cut into 20 foot lengths.

Table 2

Example	Inner Layer		Outer Layer		Pipe Outer Diameter
	Material	Thickness	Material	Thickness	
10	ION 1	0.5	ACR	0.25	20
11	ION 3	1.0	EPDM	0.4	24
12	ION 5	2.0	HDPE 2	0.5	28
13	ION 5	0.38	SEBS 2	0.2	22
14	ION 7	0.75	SEBS 3	0.3	26
15	ION 9	1.5	TI	0.5	32

Examples 16–24

The multilayer ionomer pipes summarized in Table 3 are made from the materials listed in Table 3 by conventional multilayer pipe extrusion and sizing methods with melt extrusion temperatures of 225 °C to about 250 °C. The tielayer is about 1 to 2 mils thick (0.026-0.051 mm) and is positioned between the inner layer and outer layer to provide adhesion. All Examples also have a similar tielayer on the outside surface of the outer layer: the structure of the pipe is tielayer/outer layer/tielayer/inner layer. The pipes are cut into 20 foot lengths.

Table 3

Example	Inner Layer		Tie Layer Material	Outer Layer		Pipe Outer Diameter
	Material	Thickness		Material	Thickness	
16	ION 1	0.5	EP3	EO	0.25	20
17	ION 2	1.0	EP3	EP1	0.4	24
18	ION 3	2.0	EP3	EP2	0.5	28
19	ION 4	0.38	EP3	EPDM	0.2	22
20	ION 5	0.75	HDPE2	HDPE 1	0.3	26
21	ION 6	1.5	SEBS 2	S	0.5	32
22	ION 7	0.45	SEBS 3	SBS	0.2	26
23	ION 8	1.0	SEBS 2	SEBS 1	0.1	30
24	ION 9	1.8	SEBS 2	SIS	0.3	34

Examples 25 –32

The ionomer pipe-lined carbon steel pipes summarized in Table 4 are made by inserting the ionomer pipes listed into 20-foot lengths of carbon steel pipes with 0.75-inch wall thickness with the inner diameter (ID) listed. Prior to lining the pipe, the interior surface carbon steel pipe is sandblasted and degreased.

Table 4

Example	Ionomer Pipe (Example)	Pipe ID	Example	Ionomer Pipe (Example)	Pipe ID
25	1	22	29	15	34
26	5	28	30	19	24
27	8	30	31	20	28
28	11	26	32	21	34

Examples 33–40

The ionomer-lined pipelines summarized in Table 5 are made by thermally fusing the ends (“butt fusion”) of the ionomer pipes listed, using conventional methods and inserting the polymeric pipes into the carbon steel pipes with 0.75-inch wall thickness with the length and the inner diameter (ID) listed. Prior to lining the pipe, the interior surface carbon steel pipe is sandblasted and degreased.

Table 5

Example	Ionomer Pipe (Example)	Carbon Steel Pipe Inner Diameter	Length (km)
33	2	26	1
34	4	24	2
35	9	36	3
36	10	22	0.5
37	12	30	1.5
38	17	26	1
39	20	28	2
40	23	32	3

Examples 41–64

The ionomer pipe-lined carbon steel pipes summarized in Table 6 are made by heating 20 foot lengths of carbon steel pipes with 0.75-inch wall thickness and the inner diameter (ID) listed to 200°C; inserting the ionomer pipes listed into the hot carbon steel pipes; and allowing the lined pipe to cool to ambient temperatures. Prior to lining the pipe, the interior surface carbon steel pipe is sandblasted and degreased.

Table 6: Ionomer-Lined Carbon Steel Pipes

Example	Ionomer Pipe (Example)	Inner diameter	Example	Ionomer Pipe (Example)	Inner diameter
41	1	20	53	13	22
42	2	24	54	14	26
43	3	28	55	15	32
44	4	22	56	16	20
45	5	26	57	17	24
46	6	32	58	18	28
47	7	26	59	19	22
48	8	30	60	20	26
49	9	34	61	21	32
50	10	20	62	22	26
51	11	24	63	23	30
52	12	28	64	24	34

Examples 65-73

Powder-coated carbon steel pipes, summarized in Table 7, are prepared by the following procedure. The interior surface of a 20 foot long length carbon steel pipe with the inner diameter listed is sandblasted and degreased. The pipe is then placed in a vertical orientation and induction

heated to a temperature of about 275°C. The ionomer powder listed is fed from a fluidized bed, fluidized with nitrogen gas, by allowing the fluidized bed to expand into the interior of the heated carbon steel pipe from the bottom and allowing it to flow out the top of the pipe. The fluidized bed of ionomer powder is continuously fed into the hot carbon steel pipe until the uniform coating thickness listed is achieved. The ionomer powder feed is then discontinued and the coated carbon steel pipe is then allowed to cool to ambient temperature.

Table 7: Ionomer-Lined Carbon Steel Pipes

Example	Inner diameter	Ionomer Powder	Coating
65	20	ION 10	0.38
66	26	ION 11	1.0
67	30	ION 12	1.5
68	22	ION 13	0.5
69	28	ION 14	0.75
70	34	ION 15	2.0
71	20	ION 16	0.4
72	24	ION 17	0.8
73	32	ION 18	1.0

10

Examples 74–82

Powder-coated carbon steel pipes, summarized in Table 8, are prepared by the following procedure. The interior surface of a 20 foot long length carbon steel pipe with the inner diameter listed is sandblasted and degreased. The pipe is heated to a temperature of about 350 °C in a gas-fired furnace. The hot pipe is then removed from the furnace and placed on a roller in a horizontal orientation and rolled along its axis at a rate of about 80 rpm. The ionomer powder listed is fed from a fluidized bed, fluidized with nitrogen gas, by allowing the fluidized bed to expand into the interior of the heated carbon steel pipe from one pipe end and allowing it to flow out the other end of the pipe. The fluidized bed of ionomer powder is continuously fed into the hot carbon steel pipe until a uniform coating thickness is achieved. The ionomer powder feed is then discontinued and the coated carbon steel pipe is then allowed to cool to ambient temperature while maintaining rotation of the pipe.

Table 8: Ionomer-Lined Carbon Steel Pipes

Example	Inner diameter	Ionomer Powder	Coating Thickness
74	20	ION 10	0.38
75	26	ION 11	1.0
76	30	ION 12	1.5
77	22	ION 13	0.5
78	28	ION 14	0.75
79	34	ION 15	2.0
80	20	ION 16	0.4
81	24	ION 17	0.8
82	32	ION 18	1.0

Examples 83–91

Powder-coated carbon steel pipes, summarized in Table 9, are prepared by the following procedure. The interior surface of a 20 foot long length carbon steel pipe with the diameter listed is sandblasted and degreased. The carbon steel pipe is heated to a temperature of about 350°C in a gas-fired furnace. The hot pipe is then removed from the furnace and placed on a roller in a horizontal orientation and rolled along its axis at a rate of about 80 rpm. A radially-directed spray nozzle on the end of an extensible boom is inserted down the centerline of the rotating, hot pipe. The ionomer powder listed is fed from a fluidized bed with compressed air. The spray nozzle is continuously moved up and down the length of the hot metal pipe until the uniform coating thickness listed is achieved. The ionomer powder feed is then discontinued. For Examples 85, 89 and 90, a blend of 25 wt % of the same ionomer powder and 75 wt % of a finely divided sand is overcoated onto the ionomer coating as described above until a uniform depth of 0.1 inch is achieved. Throughout the coating operation, the carbon steel pipe is in the temperature range of from about 300°C to about 250°C. The coated carbon steel pipe is then allowed to cool while maintaining the rotation until a temperature of about 100 °C is achieved. Rotation is then discontinued and the coated carbon steel pipe is allowed to cool to ambient temperature.

Table 9: Ionomer-Lined Carbon Steel Pipes

Example	Inner diameter	Ionomer Powder	Coating Thickness
83	20	ION 10	0.38
84	26	ION 11	1.0
85	30	ION 12	1.5
86	22	ION 13	0.5
87	28	ION 14	0.75
88	34	ION 15	2.0
89	20	ION 16	0.4
90	24	ION 17	0.8
91	32	ION 18	1.0

Examples 92–93

Abrasion resistance was assessed according to the following procedure. Wear test coupons were cut from injection molded plaques of ionomer ION 19. The wear test coupons were 50 mm by 50 mm by 6.35 mm thick. The wear test coupons were dried in a vacuum oven (20 inches Hg) at room temperature for at least 15 hours and then weighed. The wear test coupons were then mounted in a test chamber and a 10 wt% aqueous sand (AFS50-70 test sand) slurry at room temperature (20 to 25°C) was impinged on the wear test coupon through a slurry jet nozzle positioned 100 mm from its surface with a diameter of 4 mm at a slurry jet rate of 15–16 meters/second with a slurry jet angle of 90° relative to the surface plane for 2 hours. The wear test coupons were then removed and dried in a vacuum oven (20 inches Hg) at room temperature for at least 15 hours and then reweighed (Example 92). In Example 93 wear test coupons were tested as described for Example 92 except the sand slurry was impinged on the wear test coupon at a slurry jet angle of 25° relative to the surface plane. The results are reported in Table 10.

Table 10

Example	Material	Initial Weight (grams)	Final Weight (g)	Weight Loss (g)	Weight Loss (%)
92	ION 19	9.5565	9.5326	0.0239	0.25
93	ION 19	9.5332	9.5160	0.0172	0.18

Comparative Examples CE101–CE102 and Examples 101–104

Abrasion resistance was assessed according to the following procedure. Wear test coupons were cut from injection molded plaques of the ionomers summarized in Table 11. The wear test coupons were 50 mm by 50 mm by 6.35 mm thick. The wear test coupons were dried in

a vacuum oven (20 inches Hg) at room temperature for at least 15 hours and then weighed. The wear test coupons were then mounted in a test chamber and a 10 wt% aqueous sand (AFS50-70 test sand) slurry at room temperature (20 to 25 °C) was impinged on the wear test coupon through a slurry jet nozzle positioned 100 mm from its surface with a diameter of 4 mm at a slurry jet rate of 15–16 meters/second with a slurry jet angle of 90° relative to the surface plane for 2 hours. The wear test coupons were then removed and dried in a vacuum oven (20 inches Hg) at room temperature for at least 15 hours and then reweighed. The results are reported in Table 11.

Table 11

Example	Material	Initial Weight (grams)	Final Weight (grams)	Weight Loss (grams)	Weight Loss (wt%)
CE101	ION 21	9.1257	9.0919	0.0338	0.37
101	ION 22	9.6866	9.6560	0.0306	0.32
102	ION 23	9.2390	9.2132	0.0258	0.28
CE103	ION 25	9.6631	9.6417	0.0214	0.22
103	ION 26	9.0577	9.0431	0.0146	0.16
104	ION 27	9.4865	9.4881	0.0184	0.19

Comparative Examples CE104–CE106 and Examples 105–108

Wear test coupons were tested as described above for Example 101 except the sand slurry was impinged on the wear test coupon at a slurry jet angle of 25° relative to the surface plane. The results are reported in Table 22.

Table 22

Example	Material	Initial Weight (grams)	Final Weight (grams)	Weight Loss (grams)	Weight Loss (wt%)
CE104	ION 21	9.0930	9.0651	0.0279	0.31
105	ION 22	9.6560	9.6259	0.0301	0.31
106	ION 23	9.2132	9.1881	0.0251	0.27
CE105	ION 24	9.5332	9.5160	0.0172	0.18
CE106	ION 25	9.6417	9.6236	0.0181	0.19
107	ION 26	9.0431	9.0297	0.0134	0.15
108	ION 27	9.4681	9.4498	0.0183	0.19

Comparative Examples CE107 and Examples 109–110

Wear test coupons were tested as described above for Example 101 except that the wear test coupons were dried in a vacuum oven (20 inches Hg) at a temperature of 35°C until the weight loss was less than 1 mg/day prior to treating with the aqueous sand slurry at a temperature of 50°C, with the results summarized in Table 23.

Table 3

Example	Material	Initial Weight (grams)	Final Weight (grams)	Weight Loss (grams)	Weight Loss (wt%)
CE 107	ION 25	8.2988	8.2825	0.0163	0.20
109	ION 26	8.9339	8.9296	0.0043	0.05
110	ION 27	9.2138	9.2077	0.0061	0.07
111	ION 26	109.7108	109.6923	0.0185	0.02

Example 111

A coated carbon steel pipe was produced by a rotational coating process. Pellets of ION 6 (0.45 kilograms) were placed in a 5.08-cm inner diameter (ID) steel pipe with a length of 50.8 cm. The pipe was rotated along its length at 30 revolutions per minute (rpm) and heated to 275°C with an external oven. After reaching 275°C, the rotation rate was increased to 120 rpm for 1.5 hours. The coated pipe was then cooled to provide a steel pipe with an internal coating of ION 6 with a thickness of 6.35 to 8.47 mm. A wear test coupon was cut out of the pipe and tested as described above for Example 9, with the results shown above.

Examples 112–120

The terionomer pipes summarized in Table 24 are made from the materials listed through conventional pipe extrusion and sizing methods with melt extrusion temperatures in the range from about 150°C to about 225°C. The pipes are cut into 20 foot lengths.

Table 24

Example	Material	OD	Thickness	Example	Material	OD	Thickness
112	ION 26	20	0.5	117	ION 31	32	1.5
113	ION 27	24	1.0	118	ION 32	26	0.4
114	ION 28	28	2.0	119	ION 33	30	1.0
115	ION 29	22	0.38	120	ION 34	34	1.8
116	ION 30	26	0.75				

Examples 120–126

Terionomer bilayer pipes summarized in Table 25 are made from the materials in Table 5 through conventional multilayer pipe extrusion and sizing methods with melt extrusion temperatures in the range from about 150°C to about 225°C. The pipes are cut into 20 foot lengths.

Table 25

Example	Inner Layer		Outer Layer		OD
	Material	Thickness	Material	Thickness	
121	ION 26	0.5	ACR	0.25	20
122	ION 27	1.0	EPDM	0.4	24
123	ION 29	2.0	HDPE 1	0.5	28
124	ION 33	0.38	SEBS 2	0.2	22
125	ION 34	0.75	SEBS 3	0.3	26
126	ION 36	1.5	HDPE 2	0.5	32

Examples 127–135

Multilayer terionomer pipes are made from the materials summarized in Table 26 by conventional multilayer pipe extrusion and sizing methods with melt extrusion temperatures of about 150°C to about 225°C. The tielayer is about 1-2 mils thick (0.026–0.051 mm) and is positioned between the inner layer and outer layer to provide adhesion. All Examples also have a tielayer on the outside surface of the outer layer, e.g.; the structure of the pipe is tielayer/outer layer/tielayer/inner layer. The pipes are cut into 20-foot lengths.

Table 26

Example	Inner Layer		Tie Layer Material	Outer Layer		OD
	Material	Thickness		Material	Thickness	
127	ION 26	0.5	EP 3	EO	0.25	20
128	ION 27	1.0	EP 3	EP 1	0.4	24
129	ION 28	2.0	EP 3	EP 2	0.5	28
130	ION 29	0.38	EP 3	EPDM	0.2	22
131	ION 30	0.75	HDPE 2	HDPE 1	0.3	26
132	ION 31	1.5	SEBS 2	S	0.5	32
133	ION 32	0.45	SEBS 3	SBS	0.2	26
134	ION 33	1.0	SEBS 2	SEBS 1	0.1	30
135	ION 44	1.8	SEBS 2	SIS	0.3	34

Examples 136–142

The terionomer pipe-lined carbon steel pipes summarized in Table 27 are made by inserting the terionomer pipes listed into 20-foot lengths of carbon steel pipes with 0.75-inch wall thickness with the inner diameter (ID) listed. Prior to lining the pipe, the interior surface of the carbon steel pipe is sandblasted and degreased.

Table 27

Example	Terionomer pipe (Example)	Carbon steel pipe ID	Example	Terionomer pipe (Example)	Carbon steel pipe ID
136	112	22	140	126	34
137	116	28	141	130	24
138	119	32	142	133	28
139	122	26			

Examples 143–150

The terionomer pipe-lined pipelines summarized in Table 28 are made by thermally fusing the ends (“butt fusion”) of the terionomer pipes listed through conventional methods and inserting the polymeric pipes into the carbon steel pipes with 0.75-inch wall thickness and the length and the inner diameter (ID) listed. Prior to lining the pipe, the interior surface of the carbon steel pipe is sandblasted and degreased.

Table 28

Example	Terionomer Pipe (Example)	Carbon Steel Pipeline ID	Carbon Steel Pipeline Length (km)	Example	Terionomer Pipe (Example)	Carbon Steel Pipeline ID	Carbon Steel Pipeline Length (km)
143	113	26	1	147	123	30	1.5
144	115	24	2	148	128	26	1
145	120	36	3	149	133	28	2
146	121	22	0.5	150	134	32	3

Examples 151–172

The terionomer pipe-lined carbon steel pipes summarized in Table 29 are made by heating 20 foot lengths of carbon steel pipes with 0.75-inch wall thickness and the inner diameter (ID) listed to 200°C; inserting the terionomer pipes listed into the hot carbon steel pipes; and allowing the lined pipe to cool to ambient temperatures. Prior to lining the pipe, the interior surface of the carbon steel pipe is sandblasted and degreased.

Table 29

Example	Terionomer Pipe (Example)	Carbon Steel Pipe ID	Example	Terionomer Pipe (Example)	Carbon Steel Pipe ID
151	112	20	162	125	26
152	113	24	163	126	32
153	114	28	164	127	20
154	115	22	165	128	24
155	116	26	166	129	28
156	117	32	167	130	22
157	118	26	168	131	26
158	119	30	169	132	32
159	120	34	170	133	26
160	121	20	171	134	30
161	124	22	172	135	34

Preparative Examples PE1–PE9

Terionomer sheets with a thickness of 0.125 inch and a width of 9 feet are made from the materials summarized in Table 30 by conventional sheet extrusion methods with melt extrusion temperatures of

about 150°C to about 225°C. The sheets are plied together by conventional calendering processes to provide the described thickness.

Table 30

Preparative Example	Material	Sheet Thickness	Preparative Example	Material	Sheet Thickness
PE1	ION 26	0.5	PE6	ION 31	1.5
PE2	ION 27	1.0	PE7	ION 32	0.5
PE3	ION 28	2.0	PE8	ION 33	1.0
PE4	ION 29	0.25	PE9	ION 34	1.75
PE5	ION 30	0.75			

Examples 173–181

5 The terionomer-lined carbon steel pipes summarized in Table 31 are made by inserting the terionomer sheets listed into 20 foot lengths of carbon steel pipes with 0.75-inch wall thickness with the inner diameter (ID) listed. Prior to lining the pipe, the interior surface of the carbon steel pipe is sandblasted and degreased. The terionomer sheets are cut down
 10 in size to fit the carbon steel pipe and the seam is butt welded by thermally fusing the ends (“butt fusion”). The terionomer-lined carbon steel pipe is heated to 200°C while being rotated in the horizontal axis, and then the lined pipe is cooled to ambient temperatures.

Table 31

Example	Terionomer Sheet (Example)	Carbon Steel Pipe ID	Example	Terionomer Sheet (Example)	Carbon Steel Pipe ID
173	PE1	22	178	PE6	24
174	PE2	28	179	PE7	28
175	PE3	32	180	PE8	20
176	PE4	26	181	PE9	30
177	PE5	34			

CLAIMS

1. A pipe- or tube-shaped article having an innermost layer wherein the innermost layer has a thickness of about 0.001 to about 102 mm and comprises an ionomer comprising repeat units derived from an α -olefin having 2 to 10 carbons, 5 to 25 weight % of an α,β -ethylenically unsaturated carboxylic acid having 3 to 8 carbons, and optionally 12 to 60 wt% of an α,β -ethylenically unsaturated carboxylic acid ester, all based on the total weight of the acid polymer; and
5 to 90% of the carboxylic acids are neutralized with a metal ion.
2. The article of claim 1 wherein the α -olefin consists essentially of ethylene and acrylic acid, methacrylic acid, or mixtures thereof and 10 to 50% of the carboxylic acids are neutralized with sodium ion, lithium ion, magnesium ion, zinc ion, or mixtures of two or more thereof.
3. The article of claim 2 wherein the ionomer further comprises repeat units derived from the carboxylic acid ester including methyl acrylate, ethyl acrylate, isopropyl acrylate, butyl acrylate, or mixtures of two or more thereof.
4. The article of claim 1, 2, or 3 wherein the ionomer composition further comprises from about 0.1 to about 80 weight %, based on the total weight of the ionomer composition, of abrasion-resistant filler.
5. The article of claim 1, 2, 3, or 4 further comprising an outer layer having a thickness of about 0.1 to about 102 mm; the outer layer comprises rubber, elastomer, thermoplastic elastomer, acid terpolymer, ionomer terpolymer, or mixtures of two or more thereof; the outer layer optionally comprises a high strength fiber, a thermoset resin, or both; and the high strength fiber is produced from fiberglass, continuous glass fiber, polyaramide fiber, aramid fiber, graphite, carbon fiber, silica, quartz, ceramic, silicon carbide, boron, alumina, alumina-silica, polyethylene, ultrahigh molecular weight polyethylene, polyimide, liquid crystal polymers, polypropylene, polyester, or polyamide.
6. The article of claim 5 further comprising an intermediate layer, between the innermost layer and the outer layer, which comprises rubber, elastomer, thermoplastic elastomer, acid terpolymer, ionomer terpolymer, or mixtures of

two or more thereof wherein the high strength fiber is filament, warp yarn, unidirectional sheet, mat, cloth, knitted cloth, paper, non-woven fabric, woven fabric, or mixtures of two or more thereof.

7. The article of claim 1, 2, 3, 4, 5, or 6 further comprising an outermost layer in contact with the innermost layer and the outermost layer preferably comprises carbon steel, steel, stainless steel, cast iron, galvanized steel, aluminum, or copper, or alloys of two or more thereof.

8. The article of claim 7 wherein the outermost layer comprises carbon steel.

9. A method comprising laying up a pre-formed film or sheet into a preformed metal or plastic pipe to produce ionomer-lined metal or plastic pipe wherein the film or sheet is monolayer or multilayer film or sheet and is produced from an ionomer composition; and the pre-formed film or sheet is as characterized in claim 1, 2, 3, 4, 5, 6, 7, or 8.

10. The method of claim 9 further comprising heating the metal or plastic pipe above the softening point of the ionomer composition and allowing the metal or plastic pipe to cool to produce the ionomer-lined metal or plastic pipe.

11. A method comprising pulling or inserting an article into the interior surface of a metal pipe to produce an ionomer-lined metal pipe; wherein the pipe is the article characterized in claim 1, 2, 3, 4, 5, 6, 7, or 8.

12. A method comprising laying up a film or sheet or comprising an ionomer composition into the interior surface of a metal pipe; heating the metal pipe above the softening point of the ionomer composition; and allowing the metal pipe to cool to produce an ionomer-lined metal pipe of claim 1, 2, 3, 4, 5, 6, 7, or 8.

14. A method for transporting an abrasive material comprising producing a pipe- or tube-shaped article as characterized in claim 1, 2, 3, 4, 5, 6, 7, or 8; producing an abrasive material composition for flowing through the article; flowing the abrasive material composition into one end of the pipe- or tube-shaped article and receiving the abrasive material composition out of the other end of pipe- or tube-formed article.

15. Use of an article for transporting an abrasive material wherein the article is as characterized in claim 1, 2, 3, 4, 5, 6, 7, or 8.

INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER
INV. B32B1/08 C08L23/08

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)
B32B C08L

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	EP 0 209 396 A (DRG UK LTD [GB]) 21 January 1987 (1987-01-21) claims 1,4 column 4, line 14 column 4, line 23 <div style="text-align: center;">-----</div>	1,5

Further documents are listed in the continuation of Box C.

See patent family annex.

* Special categories of cited documents :

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP 0209396	A	21-01-1987	NONE