EUROPEAN PATENT SPECIFICATION

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Fuser member with epoxy silane cured fluoroelastomer layer, imaging process and image forming apparatus
Schmelzfixierelement mit Epoxysilan vernetzter Fluoroelastomerbeschichtung, Aufzeichnungsprozess und Aufzeichnungselement
ELément de fixage par fusion ayant une couche d’élastomère fluorée durcie avec un époxy silane, procédé de Formation d’image et appareil pour la formation d’images

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Description

[0001] The present invention is directed to crosslinked fluoropolymer materials. More specifically, the present invention is directed to crosslinked fluoropolymer materials suitable for applications such as fuser member coatings for imaging processes. One embodiment of the present invention is directed to a fuser member comprising a substrate and at least one layer thereover which is an outer fusing layer, said layer comprising a crosslinked product of a composition which comprises (a) a fluoroelastomer, and (b) an epoxy silane curative.

[0002] US-A-5,281,506 relates to a fuser member comprising a supporting substrate having an outer layer of a cured fluoroelastomer and having a thin surface of a polyorganosiloxane with a reactive functionality such as an epoxy group.


[0004] Accordingly, while known compositions and processes are suitable for their intended purposes, a need remains for improved crosslinked fluoroelastomer materials. In addition, a need remains for improved methods for curing fluoroelastomer materials. Further, a need remains for methods for curing fluoroelastomers in the absence of particulate basic metal oxides. Additionally, a need remains for methods for curing fluoroelastomers that can employ lower curing temperatures than those presently used. There is also a need for crosslinked fluoroelastomer materials that have desirably high levels of heat stability. In addition, there is a need for crosslinked fluoroelastomer materials that have improved toughness characteristics. Further, there is a need for simplified methods for curing fluoroelastomers. Additionally, there is a need for crosslinked fluoroelastomer materials that need no additional adhesive when they are bound to surfaces such as silicones or other fluoroelastomers. A need also remains for crosslinked fluoroelastomer materials having pigment particles (such as those used to enhance electrical or thermal conductivity) well and uniformly dispersed therein.

[0005] The present invention is directed to a fuser member comprising a substrate and at least one layer thereover which is an outer fusing layer, said outer fusing layer comprising a crosslinked fluoroelastomer, wherein the fluoroelastomer is crosslinked by an epoxy silane curative.

Figure 1 is an illustration of a general electrostatographic apparatus.

Figure 2 illustrates a fusing system in accordance with an embodiment of the present invention.

Figure 3 demonstrates a cross-sectional view of an embodiment of the present invention.

Figure 4 is a plot of stress versus strain illustrating the various physical properties of the films produced in Example I.

[0006] Referring to Figure 1, in a typical electrostatographic reproducing apparatus, a light image of an original to be copied is recorded in the form of an electrostatic latent image on a photosensitive member, and the latent image is subsequently rendered visible by the application of electroscopic thermoplastic resin particles, commonly referred to as toner. Specifically, photoreceptor 10 is charged on its surface by means of a charger 12 to which a voltage has been supplied from power supply 11. The photoreceptor is then imagewise exposed to light from an optical system or an image input apparatus 13, such as a laser and light emitting diode, to form an electrostatic latent image thereon. Generally, the electrostatic latent image is developed by bringing a developer mixture from developer station 14 into contact therewith. Development can be effected by use of a magnetic brush, powder cloud, or other known development process.

[0007] After the toner particles have been deposited on the photoconductive surface in image configuration, they are transferred to a copy sheet 16 by transfer means 15, which can be pressure transfer, electrostatic transfer, or the like. Alternatively, the developed image can be transferred to an intermediate transfer member and subsequently transferred to a copy sheet.

[0008] After transfer of the developed image is completed, copy sheet 16 advances to fusing station 19, depicted in Figure 1 as fusing and pressure rolls, wherein the developed image is fused to copy sheet 16 by passing copy sheet 16 between fusing member 20 and pressure member 21, thereby forming a permanent image. Photoreceptor 10, subsequent to transfer, advances to cleaning station 17, wherein any toner left on photoreceptor 10 is cleaned therefrom by use of a blade 22 (as shown in Figure 1), brush, or other cleaning apparatus.

[0009] Referring to Figure 2, an embodiment of a fusing station 19 is depicted with an embodiment of a fuser roll 20 comprising crosslinked fluoroelastomer surface 5 on a suitable base member or substrate 4, which in this embodiment is a hollow cylinder or core fabricated from any suitable metal, such as aluminum, anodized aluminum, steel, nickel, copper, or the like, having a suitable heating element 6 disposed in the hollow portion thereof which is coextensive with the cylinder. The fuser member 20 optionally can include an adhesive, cushion, or other suitable layer 7 positioned between core 4 and outer layer 5. Backup or pressure roll 21 cooperates with fuser roll 20 to form a nip or contact arc 1 through which a copy paper or other substrate 16 passes such that toner images 24 thereon contact fluoroelastomer surface 5 of fuser roll 20. As shown in Figure 2, an embodiment of a backup roll or pressure roll 21 is depicted as
having a rigid steel core 2 with a polymer or elastomer surface or layer 3 thereon. Optional sump 25 contains optional polymeric release agent 26, which may be a solid or liquid at room temperature, but is a fluid at operating temperatures. The pressure member 21 can also optionally include a heating element (not shown).

[0010] In the embodiment shown in Figure 2 for applying the polymeric release agent 26 to crosslinked fluoroelastomer surface 5, two release agent delivery rolls 27 and 28 rotatably mounted in the direction indicated are provided to transport release agent 26 to crosslinked fluoroelastomer surface 5. Delivery roll 27 is partly immersed in the sump 25 and transports on its surface release agent from the sump to the delivery roll 28. By using a metering blade 29, a layer of polymeric release fluid can be applied initially to delivery roll 27 and subsequently to crosslinked fluoroelastomer 5 in controlled thickness ranging from submicron thickness to thicknesses of several microns of release fluid. Thus, by metering device 29, preferably from about 0.1 to about 2 microns or greater thicknesses of release fluid can be applied to the surface of crosslinked fluoroelastomer 5.

[0011] Figure 3 depicts a cross-sectional view of another embodiment of the invention, wherein fuser member 20 comprises substrate 4, optional intermediate surface layer 7 comprising silicone rubber and optional fillers 30, such as aluminum oxide or the like, dispersed or contained therein, and outer crosslinked fluoroelastomer surface layer 5. Figure 3 also depicts fluid release agent or fusing oil layer 9.

[0012] The term “fuser member” as used herein refers to fuser members including fusing rolls, belts, films, sheets, and the like; donor members, including donor rolls, belts, films, sheets, and the like; and pressure members, including pressure rolls, belts, films, sheets, and the like; and other members useful in the fusing system of an electrostatic or xerographic, including digital, machine. The fuser member of the present invention can be employed in a wide variety of machines, and is not specifically limited in its application to the particular embodiment depicted herein.

[0013] Any suitable substrate can be selected for the fuser member. The fuser member substrate can be a roll, belt, flat surface, sheet, film, or other suitable shape used in the fixing of thermoplastic toner images to a suitable copy substrate. It can take the form of a fuser member, a pressure member, or a release agent donor member, preferably in the form of a cylindrical roll. Typically, the fuser member is made of a hollow cylindrical metal core, such as copper, aluminum, stainless steel, or certain plastic materials chosen to maintain rigidity and structural integrity, as well as being capable of having a polymeric material coated thereon and adhered firmly thereto. It is preferred that the supporting substrate is a cylindrical sleeve, preferably with an outer polymeric layer of from about 1 to about 6 millimeters. In one embodiment, the core, which can be an aluminum or steel cylinder, is degreased with a solvent and cleaned with an abrasive cleaner prior to being primed with a primer, such as Dow Corning® 1200, which can be sprayed, brushed, or dipped, followed by air drying under ambient conditions for thirty minutes and then baked at 150°C for 30 minutes.

[0014] Also suitable are quartz and glass substrates. The use of quartz or glass cores in fuser members allows for a lightweight, low cost fuser system member to be produced. Moreover, the glass and quartz heat allow for quick warm-up, and are therefore energy efficient. In addition, because the core of the fuser member comprises glass or quartz, there is a real possibility that such fuser members can be recycled. Moreover, these cores allow for high thermal efficiency by providing superior insulation.

[0015] When the fuser member is a belt, the substrate can be of any desired or suitable material, including plastics, such as Ultem®, available from General Electric, Ultrapet®, available from BASF, PPS (polyphenylene sulfide) sold under the tradenames Fortron®, available from Hoechst Celanese, Ryton R-4®, available from Phillips Petroleum, and Supec®, available from General Electric; PAI (polyamide imide), sold under the tradename Torlon® 7130, available from Amoco; polyketone (PK), sold under the tradename Kadel® E1230, available from Amoco; PI (polyimide); polyaramide; PEEK (polyether ether ketone), sold under the tradename PEEK 450GL30, available from Victrex; polyphenylene sulfide sold under the tradename Amodel®, available from Amoco; PES (polylethersulfone); PEI (polyetherimide); PAEK (polyaryletherketone); PBA (polyparabanic acid); silicone resin; and fluorinated resin, such as PTFE (polytetrafluoroethylene); PFA (perfluoroalkoxy); FEP (fluorinated ethylene propylene); liquid crystalline resin (Xydar®), available from Amoco; and the like, as well as mixtures thereof. These plastics can be filled with glass or other minerals to enhance their mechanical strength without changing their thermal properties. In preferred embodiments, the plastic comprises a high temperature plastic with superior mechanical strength, such as polyphenylene sulfide, polyamide imide, polyimide, polyketone, polyphenylene sulfide, polyether ketone, polylethersulfone, and polyetherimide. Suitable materials also include silicone rubbers. Examples of belt-configuration fuser members are disclosed in, for example, U.S. Patent 5,487,707, U.S. Patent 5,514,436, and Copending Application U.S. Serial No. 08/297,203, filed August 29, 1994. A method for manufacturing reinforced seamless belts is disclosed in, for example, U.S. Patent 5,409,557.

[0016] The optional intermediate layer can be of any suitable or desired material. For example, the optional intermediate layer can comprise a silicone rubber of a thickness sufficient to form a conformable layer. Suitable silicone rubbers include room temperature vulcanization (RTV) silicone rubbers, high temperature vulcanization (HTV) silicone rubbers, and low temperature vulcanization (LTV) silicone rubbers. These rubbers are known and are readily available commercially such as SILASTIC® 735 black RTV and SILASTIC® 732 RTV, both available from Dow Corning, and 106 RTV Silicone Rubber and 90 RTV Silicone Rubber, both available from General Electric. Other suitable silicone rubbers, and low temperature vulcanization (LTV) silicone rubbers. These rubbers are known and are readily available commercially such as SILASTIC® 735 black RTV and SILASTIC® 732 RTV, both available from Dow Corning, and 106 RTV Silicone Rubber and 90 RTV Silicone Rubber, both available from General Electric. Other suitable silicone rubbers include room temperature vulcanization (RTV) silicone rubbers, high temperature vulcanization (HTV) silicone rubbers, and low temperature vulcanization (LTV) silicone rubbers. These rubbers are known and are readily available commercially such as SILASTIC® 735 black RTV and SILASTIC® 732 RTV, both available from Dow Corning, and 106 RTV Silicone Rubber and 90 RTV Silicone Rubber, both available from General Electric. Other suitable silicone
materials include the silanes, siloxanes (preferably polydimethylsiloxanes), such as fluorosilicones, dimethylsilicones, liquid silicone rubbers, such as vinyl crosslinked heat curable rubbers or silanol room temperature crosslinked materials, and the like. Other materials suitable for the intermediate layer include polyimides and fluoroelastomers, including those commonly used as fuser member outer layers. If desired, the intermediate layer can comprise a crosslinked fluoroelastomer according to the present invention.

[0017] Silicone rubber materials can swell during the fusing process, especially in the presence of a release agent. In the case of fusing color toner, normally a relatively larger amount of release agent is necessary to enhance release because of the need for a larger amount of color toner than is required for black and white copies and prints. Accordingly, the silicone rubber is more susceptible to swell in an apparatus using color toner. Aluminum oxide added in a relatively small amount can reduce the swell and increase the transmissibility of heat. This increase in heat transmissibility is preferred in fusing members useful in fusing color toners, since a higher temperature (for example, from about 155 to about 180°C) is usually needed to fuse color toner, compared to the temperature required for fusing black and white toner (for example, from about 50 to about 180°C).

[0018] Accordingly, optionally dispersed or contained in the intermediate silicone rubber layer is aluminum oxide in a relatively low amount of from about 0.05 to about 5 percent by volume, preferably from about 0.1 to about 5 percent by volume, and more preferably from about 2.2 to about 2.5 percent by total volume of the intermediate layer. In addition to the aluminum oxide, other metal oxides and/or metal hydroxides can be used. Such metal oxides and/or metal hydroxides include tin oxide, zinc oxide, calcium hydroxide, magnesium oxide, lead oxide, chromium oxide, copper oxide, and the like, as well as mixtures thereof. In a preferred embodiment, a metal oxide is present in an amount of from about 10 to about 50 percent by volume, preferably from about 20 to about 40 percent by volume, and more preferably from about 30 to about 35 percent by total volume of the intermediate layer. In a preferred embodiment copper oxide is used in these amounts in addition to the aluminum oxide. In preferred embodiments, the average particle diameter of the metal oxides such as aluminum oxide or copper oxide preferably is from about 1 to about 10 microns, and more preferably from about 3 to about 5 microns, although the average particle diameter can be outside of these ranges.

[0019] The optional intermediate layer typically has a thickness of from about 0.05 to about 10 millimeters, preferably from about 0.1 to about 5 millimeters, and more preferably from about 1 to about 3 millimeters, although the thickness can be outside of these ranges. More specifically, if the intermediate layer is present on a pressure member, it typically has a thickness of from about 0.05 to about 5 millimeters, preferably from about 0.1 to about 3 millimeters, and more preferably from about 0.5 to about 1 millimeter, although the thickness can be outside of these ranges. When present on a fuser member, the intermediate layer typically has a thickness of from about 1 to about 10 millimeters, preferably from about 2 to about 5 millimeters, and more preferably from about 2.5 to about 3 millimeters, although the thickness can be outside of these ranges. In a preferred embodiment, the thickness of the intermediate layer of the fuser member is higher than that of the pressure member, so that the fuser member is more deformable than the pressure member.

[0020] The outer fusing layer of the fuser member of the present invention comprises a crosslinked fluoroelastomer. This layer can be prepared by first preparing a liquid composition containing the uncrosslinked fluoroelastomer and a crosslinking agent according to the present invention as disclosed hereinbelow, as well as any desired optional fillers or other optional components. The liquid composition is applied to the substrate, followed by crosslinking of the fluoroelastomer, typically by heating to a temperature of from about 200 to about 400°F, although the temperature can be outside of this range, and typically for from about 10 to about 24 hours, although the time can be outside of this range, and although other crosslinking methods can also be employed.

[0021] Examples of suitable outer fusing layers of the fuser member include fluoroelastomers. Specifically, suitable fluoroelastomers are those described in, for example, U.S. Patent 5,166,031, U.S. Patent 5,281,506, U.S. Patent 5,366,772, U.S. Patent 5,370,931, U.S. Patent 4,257,699, U.S. Patent 5,017,432, and U.S. Patent 5,061,965. These fluoroelastomers, particularly from the class of copolymers, terpolymers, and tetrapolymers of vinylidene fluoride, hexafluoropropylene, and tetrafluoroethylene and a possible cure site monomer, are known commercially under various designations as VITON® A, VITON® E@, VITON® E60C®, VITON® E430®, VITON® 910®, VITON® GH®, VITON® GF®, VITON® E45®, VITON® A201C®, and VITON® B50®. The VITON® designation is a trademark of E.I. Du Pont de Nemours, Inc. Other commercially available materials include FLUOREL® 2170®, FLUOREL® 2174®, FLUOREL® 2176®, FLUOREL® 2177®, FLUOREL® 2123®, and FLUOREL® LVS 76®, FLUOREL® being a trademark of 3M Company. Additional commercially available materials include AFLAS®, a poly(propylene-tetrafluoroethylene), and FLUOREL® LII® (LII900), a poly(propylene-tetrafluoroethylenevinylidenefluoride) elastomer, both also available from 3M Company, as well as the TECNOFLONS® identified as FOR-60K@, FOR-LHF®, NM®, FOR-THF®, FOR-TFS®, TH®, and TN505®, available from Montedison Specialty Chemical Company. Fluoropolymer, and especially fluoroelastomer, materials such as the VITON® materials, are beneficial when used as fuser roll coatings at normal fusing temperatures (e.g., from about 50 to about 150°C). These materials have the superior properties of high temperature stability, thermal conduction, wear resistance, and release oil swell resistance.

[0022] In another embodiment, the polymer is a fluoroelastomer having a relatively low quantity of vinylidene fluoride,
such as in VITON GF®, available from E.I. DuPont de Nemours, Inc. The VITON GF® has 35 percent by weight of vinylidene fluoride, 34 percent by weight of hexafluoropropylene, and 29 percent by weight of tetrafluoroethylene, with 2 percent by weight cure site monomer. The cure site monomer can be those available from Du Pont, such as 4-bromoperfluorobutene-1, 1,1-dihydro-4-bromoperfluorobutene-1, 3-bromoperfluoropropene-1, 1,1-dihydro-3-bromoperfluoropropene-1, or any other suitable cure site monomer. The fluorine content of the VITON GF® is about 70 percent by weight by total weight of fluoroelastomer.

In yet another embodiment, the polymer is a fluoroelastomer having relatively low fluorine content such as VITON A201®, which is a copolymer of vinylidene fluoride and hexafluoropropylene, having about 65 percent by weight fluorine content. This copolymer is compounded with crosslinkers and phosphonium compounds used as accelerators.

Particularly preferred for the present invention are the fluoroelastomers containing vinylidene fluoride, such as the VITON® materials. Most preferred are the vinylidene fluoride terpolymers such as VITON® GF.

It is preferred that the fluoroelastomer have a relatively high fluorine content of from about 65 to about 71 percent by weight, preferably from about 69 to about 70 percent by weight, and more preferably about 70 percent fluorine by weight of total fluoroelastomer. Less expensive elastomers, such as some containing about 65 percent by weight fluorine, can also be used.

In uncrosslinked polymers without cure site monomers, the relative amount of crosslinking sites such as vinylidene fluoride monomers in the polymer backbone affects the crosslink density and mechanical properties of the crosslinked polymer; accordingly, the relative amount of crosslinking sites in the polymer backbone can be selected depending on the desired physical properties and crosslink density of the crosslinked polymer. The literature available with commercially available fluoroelastomers such as the VITON® series provides extensive information regarding how varying relative amounts of monomers affects the performance objectives of the crosslinked material.

Other suitable fluoropolymers include those such as fluoroelastomer composite materials, which are hybrid polymers comprising at least two distinguishing polymer systems, blocks, or monomer segments, one monomer segment (hereinafter referred to as a "first monomer segment") that possesses a high wear resistance and high toughness, and the other monomer segment (hereinafter referred to as a "second monomer segment") that possesses low surface energy. The composite materials are hybrid or copolymer compositions comprising substantially uniform, integral, interpenetrating networks of a first monomer segment and a second monomer segment, and in some embodiments, optionally a third grafted segment, wherein both the structure and the composition of the segment networks are substantially uniform when viewed through different slices of the fuser member layer. The term "interpenetrating network" refers to the addition polymerization matrix wherein the polymer strands of the first monomer segment and the second monomer segment, as well as those of the optional third grafted segment, are intertwined in one another. A copolymer composition comprises a first monomer segment and a second monomer segment, as well as an optional third grafted segment, wherein the monomer segments are randomly arranged into a long chain molecule. Examples of polymers suitable for use as the first monomer segment or tough monomer segment include, for example, polyamides, polymides, polysulfones, fluoroelastomers, and the like, as well as mixtures thereof. Examples of the low surface energy monomer segment or second monomer segment polymers include polyorganosiloxanes and the like, and also include intermediates that form inorganic networks. An intermediate is a precursor to inorganic oxide networks present in polymers described herein. This precursor goes through hydrolysis and condensation followed by the addition reactions to form desired network configurations of, for example, networks of metal oxides such as titanium dioxide, silicon oxide, zirconium oxide, and the like; networks of metal halides; and networks of metal hydroxides. Examples of intermediates include metal alkoxides, metal halides, metal hydroxides, and polyorganosiloxanes. The preferred intermediates are alkoxides, and particularly preferred are tetraethoxy orthosilicate for silicon oxide networks and titanium isobutoxide for titanium dioxide networks. In some embodiments, a third low surface energy monomer segment is a grafted monomer segment and, in preferred embodiments, is a polyorganosiloxane. In these preferred embodiments, it is particularly preferred that the second monomer segment is an intermediate to a network of metal oxide. Preferred intermediates include tetraethoxy orthosilicate for silicon oxide networks and titanium isobutoxide for titanium dioxide networks.

Also suitable are volume grafted elastomers. Volume grafted elastomers are a special form of hydrofluoroelastomer, and are substantially uniform integral interpenetrating networks of a hybrid composition of a fluoroelastomer and a polyorganosiloxane, the volume graft having been formed by dehydrofluorination of fluoroelastomer by a nucleophilic dehydrofluorinating agent, followed by addition polymerization by the addition of an alkene or alkyne functionally terminated polyorganosiloxane and a polymerization initiator. Examples of specific volume graft elastomers are disclosed in, for example, U.S. Patent 5,166,031, U.S. Patent 5,281,506, U.S. Patent 5,366,772, and U.S. Patent 5,370,931.

Examples of suitable polymer composites include volume grafted elastomers, titamers, grafted titamers, ceramers, grafted ceramers, and the like. Titamers and grafted titamers are disclosed in, for example, U.S. Patent 5,496,987; ceramers and grafted ceramers are disclosed in, for example, U.S. Patent 5,337,129, and volume grafted fluoroelastomers are disclosed in, for example, U.S. Patent 5,366,772. In addition, these fluoroelastomer composite
The fluoroelastomer is cured or crosslinked with an epoxy silane curative. Examples of suitable epoxy silane curatives include those of the formula

\[
\begin{align*}
\text{O} & \quad \text{CH}_2 \\
\text{CH} & \quad \text{CH}_2 \\
\text{O} & \quad (\text{CH}_2)_n \\
(\text{RO})_y & \quad (\text{CH}_3)_{3-y}
\end{align*}
\]

wherein \( y \) is an integer of 0, 1, 2, or 3 and \( R \) is an alkyl group, typically with from 1 to about 5 carbon atoms, although the number of carbon atoms can be outside of these ranges. One specific example of a suitable epoxy silane curative is glycidoxypropyl trimethoxysilane, of the formula

\[
\begin{align*}
\text{O} & \quad \text{CH}_2 \\
\text{CH} & \quad \text{CH}_2 \\
\text{O} & \quad (\text{CH}_2)_3 \\
\text{H}_3\text{CO} & \quad \text{S} \quad \text{OCH}_3 \\
\text{O} & \quad \text{CH}_3
\end{align*}
\]

and is commercially available as, for example, DC Z 6040 from Dow Corning Co., Midland, MI, as G6720 from United Chemical Technologies, and as SIG5840.0 from Gelest. Other suitable commercially available curatives include 3-glycidoxypropyl dimethylethoxysilane, of the formula

\[
\begin{align*}
\text{O} & \quad \text{CH}_2 \\
\text{CH} & \quad \text{CH}_2 \\
\text{O} & \quad (\text{CH}_2)_3 \\
\text{H}_3\text{C} & \quad \text{S} \quad \text{OCH}_2\text{H}_5 \\
\text{CH}_3 &
\end{align*}
\]

commercially available as, for example, UCT G6700 from United Chemical Technologies and as SIG5825.0 from Gelest, and (3-glycidoxypropyl) methyldiethoxysilane, of the formula
commercially available as, for example, G6710 from United Chemical Technologies and as SIG5832.0 from Gelest.

[0031] Optionally, the solution containing the curative and the fluoroelastomer can further contain an acid acceptor, which typically is an inorganic base. The acid acceptor dehydrofluorinates vinylidene fluoride in a polymer containing vinylidene fluoride monomer units, creating double bonds that act as reactive sites for crosslinking. Examples of suitable acid acceptors include magnesium oxide, calcium hydroxide, lead oxide, copper (II) oxide, zinc oxide, and the like, as well as mixtures thereof. Organic acid acceptors can also be employed, including amines and their Schiff base derivatives, such as diamines, aliphatic amines, aromatic amines, wherein the aromatic group can be (but is not limited to) benzene, toluene, naphthalene, anthracene, and the like, with specific examples of suitable amines or their Schiff base derivatives include N,N'-dicinnamylidene-1,6-hexanediylamine (typically from about 2.5 to about 5 parts by weight per 100 hundred parts by weight fluoroelastomer), available under the tradename TECNOFLON® TECNOCIN-A®, hexamethylenediamine carbamate (typically from about 1 to about 3 parts by weight per 100 hundred parts by weight fluoroelastomer), available under the tradename TECNOFLON® TECNOCIN-B®, triethylene tetramine or TETA (typically from about 1 to about 3 parts by weight per 100 hundred parts by weight fluoroelastomer), and the like, as well as mixtures thereof. The inorganic acid acceptor typically is added to the fluoroelastomer solution in an amount of from about 2 to about 20 parts by weight per 100 hundred parts by weight fluoroelastomer, and preferably from about 8 to about 15 parts by weight per 100 hundred parts by weight fluoroelastomer, although the relative amounts can be outside of these ranges. The amine as the acid acceptor typically is added to the fluoroelastomer solution in an amount of from about 0.5 to about 5 parts by weight per 100 hundred parts by weight fluoroelastomer, and preferably from about 1 to about 3 parts by weight per 100 hundred parts by weight fluoroelastomer, although the relative amounts can be outside of these ranges.

[0032] The curative is added to the fluoroelastomer prior to coating the fluoroelastomer onto the substrate. The fluoroelastomer is dissolved or dispersed in a solvent, such as methyl ethyl ketone, methyl isobutyl ketone, toluene, butyl acetate, amyl acetate, mixtures thereof, or the like. The curative is added to the fluoroelastomer in any desired or effective amount, typically from about 0.5 to about 15 percent by weight of the fluoroelastomer, preferably from about 2.5 to about 10 percent by weight of the fluoroelastomer, and more preferably from about 4 to about 7 percent by weight of the fluoroelastomer, although the relative amounts can be outside of these ranges. The fluoroelastomer can be dissolved or dispersed in the solvent in any desired or effective amount, typically from about 5 to about 30 percent by weight of the fluoroelastomer/solvent solution, preferably from about 10 to about 25 percent by weight of the fluoroelastomer/solvent solution, and more preferably from about 15 to about 20 percent by weight of the fluoroelastomer/solvent solution, although the amount can be outside of these ranges. While not required, it is preferred to add the curative to the solution last, thereby enabling adequate mixing, grinding (if additional optional fillers are present in the solution), and dissolution of the polymer prior to the crosslinking reaction. Subsequent to coating the fluoroelastomer/curative mixture onto the substrate, the coating is dried by any desired or suitable means, such as simple exposure to ambient air or the like. Thereafter, the fluoroelastomer is cured by exposure to heat, typically at from about 200 to about 400°F, preferably at from about 250 to about 350°F, and more preferably at from about 250 to about 300°F, although the temperature can be outside of these ranges, and typically for from about 10 to about 24 hours, preferably from about 12 to about 20 hours, and more preferably from about 14 to about 16 hours, although the time can be outside of these ranges. While drying is not required, the drying step is preferred to improve film characteristics of the crosslinked polymer. In addition, while not preferred, drying can occur simultaneously with curing, and curing can be by other methods, such as exposure to ultraviolet, infrared, or e-beam radiation, or the like.

[0033] Optionally, conductive fillers can be dispersed in the outer fusing layer of the fuser member, particularly in embodiments wherein a functional fuser oil is used. Preferred fillers are capable of interacting with the functional groups of the release agent to form a thermally stable film which releases the thermoplastic resin toner and prevents the toner from contacting the filler surface material itself. This bonding enables a reduction in the amount of oil needed to promote
release. Further, preferred fillers promote bonding with the oil without causing problems such as scumming or gelling. In addition, it is preferred that the fillers be substantially non-reactive with the outer polymer material so that no adverse reaction occurs between the polymer material and the filler which would hinder curing or otherwise negatively affect the strength properties of the outer surface material. Fillers in the outer fusing layer can also increase thermal conductivity.

[0034] Other optional adjuvants and fillers can be incorporated in the crosslinked fluoroelastomer of the outer fusing layer according to the present invention, provided that they do not affect the integrity of the crosslinked fluoroelastomer. Such fillers normally encountered in the compounding of elastomers include coloring agents, reinforcing fillers, processing aids, accelerators, and the like. Oxides, such as magnesium oxide, and hydroxides, such as calcium hydroxide, are sometimes added to fluoroelastomers. Proton acids, such as stearic acid, are suitable additives in EPDM and BR polymer formulations to improve release by improving bonding of amino oils to the elastomer composition. Other metal oxides, such as cupric oxide and/or zinc oxide, can also be used to improve release. Metal oxides, such as copper oxide, aluminum oxide, magnesium oxide, tin oxide, titanium dioxide, iron oxide, zinc oxide, manganese oxide, molybdenum oxide, and the like, carbon black, graphite, metal fibers and metal powder particles such as silver, nickel, aluminum, and the like, as well as mixtures thereof, can promote thermal conductivity. The addition of silicone particles to a fluopolymer outer fusing layer can increase release of toner from the fuser member during and following the fusing process. Processability of a fluopolymer outer fusing layer can be increased by increasing absorption of silicone oils, in particular by adding fillers such as fused silica or clays such as organo-montmorillonites. Inorganic particulate fillers can increase the abrasion resistance of the polymeric outer fusing layer. Examples of such fillers include metal-containing fillers, such as a metal, metal alloy, metal oxide, metal salt, or other metal compound; the general classes of suitable metals include those metals of Groups 1b, 2a, 2b, 3a, 3b, 4a, 4b, 5a, 5b, 6b, 7b, 8, and the rare earth elements of the Periodic Table. Specific examples of such fillers are oxides of aluminum, copper, tin, zinc, lead, iron, platinum, gold, silver, antimony, bismuth, zinc, iridium, ruthenium, tungsten, manganese, cadmium, mercury, vanadium, chromium, magnesium, nickel, and alloys thereof. Also suitable are reinforcing calcined alumina and non-reinforcing tabular alumina.

[0035] The polymer layers of the fuser member can be coated on the fuser member substrate by any desired or suitable means, including injection molding, normal spraying, dipping, and tumble spraying techniques. A flow coating apparatus as described in Copending Application U.S. Serial No. 08/672,493 filed June 26, 1996, entitled "Flow Coating Process for Manufacture of Polymeric Printer Roll and Belt Components," can also be used to flow coat a series of fuser rolls. It is preferred that the polymers be diluted with a solvent, and particularly an environmentally friendly solvent, prior to application to the fuser substrate. Alternative methods, however, can be used for coating layers, including methods described in Copending Application U.S. Serial No. 09/069,476, filed April 29, 1998, entitled "Method of Coating Fuser Members".

[0036] The cured fluoroelastomer layer is situated on the substrate (or any underlying intermediate layers) in any desired or suitable thickness, typically from about 5 to about 30 microns, preferably from about 5 to about 25 microns, and more preferably from about 10 to about 20 microns, although the thickness can be outside of these ranges.

[0037] Other optional layers, such as adhesive layers or other suitable cushion layers or conductive layers, can also be incorporated between the outer elastomer layer and the substrate. Optional intermediate adhesive layers and/or polymer layers can be applied to achieve desired properties and performance objectives. An adhesive intermediate layer can be selected from, for example, epoxy resins and polysiloxanes. Preferred adhesives include materials such as Union Carbide A-1100, Dow TACTIX 740, Dow TACTIX 741, Dow TACTIX 742, Dow Coming PS200, Dow Coming SS-2260, Union Carbide A-1100, and United Chemical Technologies A0728. A particularly preferred curative for the aforementioned adhesives is Dow H41. Preferred adhesive(s) for siliconic adhesion are A4040 silane, available from Dow Corning Corp., Midland, Michigan. 48686, D.C. 1200, also available from Dow Coming, and S-11 silane, available from Grace Specialty Polymers, Lexington, MA. Adhesion of fluoroelastomers can be accomplished with Chemlok® 5150, available from Lord Corp., Coating and Lamination Division, Erie, PA.

[0038] Polymeric fluid release agents can optionally be used in combination with the polymer outer layer to form a layer of fluid release agent which results in an interfacial barrier at the surface of the fuser member while leaving a non-reacted low surface energy release fluid as an outer release film. Suitable release agents include both functional and non-functional fluid release agents. The term "nonfunctional oil" as used herein refers to oils which do not react chemically with the fillers on the surface of the fuser member. The term "functional oil" as used herein refers to a release agent having functional groups which react chemically with the fillers present on the surface of the fuser member so as to reduce the surface energy of the fillers and thereby provide better release of toner particles from the surface of the fuser member. Non-functional release agents include known polydimethylsiloxane release agents. Functional release agents such as amino functional, mercapto functional, hydride functional, and others, can also be used. Specific examples of suitable amino functional release agents include T-Type amino functional silicone release agents, as disclosed in, for example U.S. Patent 5,516,361, monoamino functional silicone release agents, as described in, for example U.S. Patent 5,531,813, and amino functional siloxane release agents, as disclosed in, for example, U.S.

Preferred polymeric fluid release agents to be used in combination with the polymeric layer are those comprising molecules having functional groups which interact with any filler particles in the fuser member and also interact with the polymer itself in such a manner as to form a layer of fluid release agent that results in an interfacial barrier at the surface of the fuser member while leaving a non-reacted low surface energy release fluid as an outer release film. Suitable release agents include polydimethylsiloxane fusing oils having amino, mercapto, and other functionality for fluoroelastomer compositions. For silicone based compositions, a nonfunctional oil can also be used. The release agent can further comprise nonfunctional oil as a diluent.

Specific embodiments of the invention will now be described in detail. These examples are intended to be illustrative, and the invention is not limited to the materials, conditions, or process parameters set forth in these embodiments. All parts and percentages are by weight unless otherwise indicated.

**EXAMPLE I**

Solutions containing methyl ethyl ketone, a fluoroelastomer (VITON® GF, obtained from E.I. DuPont de Nemours & Co., Wilmington, DE) and the additional ingredients listed in the table below (ingredients listed in units of parts by weight) were prepared by stirring the ingredients (except for the curatives) overnight to dissolve the polymer. The oxides were pre-ground and dispersed in methyl isobutyl ketone prior to being added to the solutions. About 10 to 30 minutes prior to coating, the curatives (VC-50, a bisphenol A based curing system with an accelerator, available from E.I. DuPont de Nemours & Co., Wilmington, DE, or DC 6040, an epoxy silane curative, available from Dow Corning Co., Midland, MI) were added to the solutions (with the VC-50 being predissolved in methyl ethyl ketone). The fluoroelastomer solutions were spray coated onto a TEFLON® sheet wrapped around a substrate. The films thus prepared were air dried, followed by heat curing for 22 hours at 400°F and peeling away of the cured polymers from the TEFLON® as free standing films. In each instance wherein the solutions contained oxides, the oxides were a mixture of magnesium oxide, present in an amount of 2 parts by weight per 100 parts by weight of the polymer, and calcium hydroxide, present in an amount of about 1 part by weight per 100 parts by weight of the polymer.

<table>
<thead>
<tr>
<th>Solution</th>
<th>VITON® GF</th>
<th>ME K</th>
<th>Oxides</th>
<th>VC-50</th>
<th>DC6040</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>10</td>
<td>90</td>
<td>no</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>B</td>
<td>10</td>
<td>90</td>
<td>yes</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>C</td>
<td>10</td>
<td>90</td>
<td>yes</td>
<td>5</td>
<td>-</td>
</tr>
<tr>
<td>D</td>
<td>10</td>
<td>90</td>
<td>yes</td>
<td>-</td>
<td>2</td>
</tr>
<tr>
<td>E</td>
<td>10</td>
<td>90</td>
<td>yes</td>
<td>-</td>
<td>5</td>
</tr>
<tr>
<td>F</td>
<td>10</td>
<td>90</td>
<td>yes</td>
<td>-</td>
<td>10</td>
</tr>
<tr>
<td>G</td>
<td>10</td>
<td>90</td>
<td>no</td>
<td>-</td>
<td>10</td>
</tr>
</tbody>
</table>

Thereafter, various physical properties of the films thus prepared were tested by Instron testing as per ASTM F219. Tensile strength was measured in inch pounds per cubic inch (in-#/in³), toughness was measured in pounds per cubic inch, initial modulus was measured in pounds per square inch, and percent strain at maximum load is a percentage figure. In a plot of stress (force divided by area; psi) versus strain (measured in terms of length change: ΔL/L_initial), the maximum load (measured in pounds) is the load at which the film breaks, the tensile strength is the point on the curve at which the material breaks (stress obtained at the highest applied force), the initial modulus is the slope of the initial stress-strain curve, the toughness is the area under the stress-strain curve, and the percent strain at maximum load is the percent change in length of the test sample at the breaking point (Fig 4).
As the data indicate, the films cured with the epoxy silane curative exhibited equivalent heat stability and improved toughness compared to the films cured with the bisphenol A based curative. A comparison of films "A" and "C" to film "G" indicates that the addition of the DC Z-6040 improved the mechanical properties of the film. Film "C" is typical of known standard materials with the known curative and particulate oxide acid acceptor additives, whereas film "G" is illustrative of the instant invention, using only solution or liquid based additives. The curing process of the present invention is simpler than known processes, in that no particulate fillers are required and no grinding is required. The polymer was also more easily dissolved in the solvent with the solutions of the present invention. As a comparison of films "D", "E", "F", and "G" indicates, increasing amounts of crosslinking curative increase the crosslink density of the material, thereby increasing brittleness of the cured polymer. In addition, it is believed that since the compositions of the present invention are more reactive than known compositions, less energy is required to drive the crosslinking reaction. The adhesive properties of the curative also render the use of additional adhesives between the cured fluoroelastomer layer and the substrate optional.

[0042] Other embodiments and modifications of the present invention may occur to those of ordinary skill in the art subsequent to a review of the information presented herein; these embodiments and modifications, as well as equivalents thereof, are also included within the scope of this invention.

Claims

1. A fuser member comprising a substrate and at least one layer thereover which is an outer fusing layer, said outer fusing layer comprising a crosslinked fluoroelastomer, wherein the fluoroelastomer is crosslinked by an epoxy silane curative.

2. The fuser member of claim 1 wherein the fluoroelastomer is a copolymer of vinylidenefluoride and hexafluoropro-
pylene, a terpolymer of vinylidene fluoride, hexafluoropropylene and tetrafluoroethylene, a tetrapolymer of vinylidene fluoride, hexafluoropropylene, tetrafluoroethylene and a cure site monomer, or a mixture thereof.

3. The fuser member of claim 1 wherein the epoxy silane curative is of the formula

\[
\text{\begin{center}
\begin{tikzpicture}
\begin{scope}[scale=0.5]
\node (a) at (0,0) {O};
\node (b) at (1,0) {CH2};
\node (c) at (2,0) {CH2};
\node (d) at (3,0) {O};
\node (e) at (4,0) {\text{(CH2)n}};
\node (f) at (5,0) {\text{(CH3)3-y}};
\node (g) at (6,0) {\text{(RO)y}};
\draw (a) -- (b);
\draw (b) -- (c);
\draw (c) -- (d);
\draw (d) -- (e);
\draw (e) -- (f);
\draw (f) -- (g);
\end{scope}
\end{tikzpicture}
\end{center}
\]

wherein \( y \) is an integer of 0, 1, 2, or 3 and \( R \) is an alkyl group.

4. The fuser member of claim 1 wherein the epoxy silane curative is glycidoxypropyl trimethoxysilane, glycidoxypropyl dimethylethoxysilane, or (3-glycidoxypropyl) methyldiethoxysilane.

5. The fuser member of claim 1 wherein the epoxy silane curative and the fluoroelastomer are present in relative amounts, prior to curing, of from about 0.5 parts by weight curative per 100 parts by weight fluoroelastomer to about 15 parts by weight curative per 100 parts by weight fluoroelastomer.

6. The fuser member of claim 1 wherein the layer comprises a crosslinked fluoroelastomer which comprises (a) a fluoroelastomer, (b) an epoxy silane curative, and (c) an acid acceptor.

7. The fuser member of claim 6 wherein the acid acceptor is magnesium oxide, calcium hydroxide, lead oxide, copper (III) oxide, zinc oxide, or mixtures thereof.

8. The fuser member of claim 6 wherein the acid acceptor and the fluoroelastomer are present in relative amounts, prior to curing, of from about 2 parts by weight acid acceptor per 100 parts by weight fluoroelastomer to about 20 parts by weight acid acceptor per 100 parts by weight fluoroelastomer.

9. A process which comprises (i) generating an electrostatic latent image on an imaging member; (ii) developing the latent image by contacting the imaging member with a developer; (iii) transferring the developed image to a copy substrate; and (iv) affixing the developed image to the copy substrate by contacting the developed image with the fuser member of claim 1.

10. An image forming apparatus for forming images on a recording medium which comprises: (i) a charge-retentive surface capable of receiving an electrostatic latent image thereon; (ii) a development assembly to apply toner to the charge-retentive surface, thereby developing the electrostatic latent image to form a developed image on the charge retentive surface; (iii) a transfer assembly to transfer the developed image from the charge retentive surface to a copy substrate; and (iv) a fixing assembly to fuse toner images to a surface of the copy substrate, wherein the fixing assembly includes the fuser member of claim 1.

**Patentansprüche**

1. Schmelzfüxielement, umfassend ein Substrat und mindestens eine Schicht darüber, welche eine äußere Schmelzschiicht ist, wobei besagte äußere Schmelzschiicht ein vemetztes Fluorelastomer umfasst, wobei das Fluorelastomer mittels eines Epoxysilal Vulkanisiermittels vemetzt ist.
2. Schmelzfixierelement nach Anspruch 1, wobei das Fluorelastomer ein Copolymer aus Vinylidenfluorid und Hexafluorpropylen, ein Terpolymer aus Vinylidenfluorid, Hexafluorpropylen und Tetrafluorethylen, ein Tetrapolymer aus Vinylidenfluorid, Hexafluorpropylen, Tetrafluorethylen und einem Monomer mit Verzweigungsstelle, oder eine Mischung daraus ist.

3. Schmelzfixierelement nach Anspruch 1, wobei das Epoxysilan Vulkanisiermittel die folgende Formel aufweist

![Chemical Structure]

worin \( y \) eine ganze Zahl von 0,1, 2 oder 3 und \( R \) eine Alkylgruppe ist.

4. Schmelzfixierelement nach Anspruch 1, wobei es sich bei dem Epoxysilan Vulkanisiermittel um Glycidoxypropyltrimethoxysilan, Glycidoxypropyldimethylethoxysilan oder (3-Glycidoxypropyl)methyldiethoxysilan handelt.

5. Schmelzfixierelement nach Anspruch 1, wobei das Epoxysilan Vulkanisiermittel und das Fluorelastomer vor dem Vernetzen in relativen Mengen von etwa 0,5 Gewichtsteilen Vulkanisiermittel pro 100 Gewichtsteilen Fluorelastomer bis etwa 15 Gewichtsteilen Vulkanisiermittel pro 100 Gewichtsteilen Fluorelastomer vorliegen.

6. Schmelzfixierelement nach Anspruch 1, wobei die Schicht ein vemetztes Fluorelastomer enthält, welches (a) ein Fluorelastomer, (b) ein Epoxysilan Vulkanisiermittel und (c) einen Säureakzeptor umfasst.

7. Schmelzfixierelement nach Anspruch 6, wobei es sich bei dem Säureakzeptor um Magnesiumoxid, Calciumhydroxid, Bleioxid, Kupfer(III)-oxid, Zinkoxid oder Mischungen daraus handelt.

8. Schmelzfixierelement nach Anspruch 6, wobei der Säureakzeptor und das Fluorelastomer vor dem Vernetzen in relativen Mengen von etwa 2 Gewichtsteilen Säureakzeptor pro 100 Gewichtsteilen Fluorelastomer bis etwa 20 Gewichtsteilen Säureakzeptor pro 100 Gewichtsteilen Fluorelastomer vorliegen.


10. Bild ausbildende Vorrichtung zur Ausbildung von Bildern auf einem Aufnahmemedium, umfassend: (i) eine Ladungs-remanente Oberfläche, welche in der Lage ist, ein elektrostatisches latentes Bild darauf zu empfangen; (ii) eine Entwicklungsbauinheit, um Toner auf die Ladungs-remanente Oberfläche aufzubringen, dabei das elektrostatische latente Bild zu entwickeln, um ein entwickeltes Bild auf der Ladungs-remanenten Oberfläche auszubilden; (iii) eine Übertragungsbaueinheit zur Übertragung des entwickelten Bildes von der Ladungs-remanenten Oberfläche auf ein Kopiersubstrat; und (iv) eine fixierende Bauinheit, um Toner-Bilder auf eine Oberfläche des Kopiersubstrates zu schmelzen, wobei die fixierende Bauinheit das Schmelzfixierelement nach Anspruch 1 einschließt.

Revendications

1. Élément de fixage comprenant un substrat et sur celui-ci au moins unecouche qui est une couche de fixage
extérieure, ladite couche de fixage extérieure comprenant un fluoroélastomère réticulé, dans lequel le fluoroélastomère est réticulé par un agent durcisseur de type époxysilane.

2. Élément de fixage selon la revendication 1, dans lequel le fluoroélastomère est un copolymère de fluorure de vinyldène et d'hexafluoropropylène, un terpolymère de fluorure de vinyldène, d'hexafluoropropylène et de tétrafluoroéthylène, un tétrapolymère de fluorure de vinyldène, d'hexafluoropropylène, de tétrafluoroéthylène et d'un monomère à sites de durcissement, ou un mélange de ceux-ci.

3. Élément de fixage selon la revendication 1, dans lequel l'agent durcisseur de type époxysilane répond à la formule

\[
\begin{align*}
O &-CH_2 \\
\text{H} &\text{CH}_2 \\
\text{O} &\text{(CH}_2\text{n)} \\
\text{Si} &\text{(CH}_3\text{)}_{3-y} \\
\text{y} &\text{ est un nombre entier valant } 0, 1, 2 \text{ ou } 3 \text{ et } R \text{ est un groupe alkylique.}
\end{align*}
\]

dans laquelle y est un nombre entier valant 0, 1, 2 ou 3 et R est un groupe alkylique.

4. Élément de fixage selon la revendication 1, dans lequel l'agent durcisseur de type époxysilane est le glycidoxypropyltriméthoxysilane, le glycidoxypropylidiméthyléthoxysilane ou le (3-glycidoxypropyl)méthylidéthoxysilane.

5. Élément de fixage selon la revendication 1, dans lequel l'agent durcisseur de type époxysilane et le fluoroélastomère sont présents en des quantités relatives, avant durcissement, d'environ 0,5 partie en poids de l'agent durcisseur pour 100 parties en poids du fluoroélastomère à environ 15 parties en poids de l'agent durcisseur pour 100 parties en poids du fluoroélastomère.

6. Élément de fixage selon la revendication 1, dans lequel la couche comprend un fluoroélastomère réticulé qui comprend (a) un fluoroélastomère, (b) un agent durcisseur de type époxysilane et (c) un accepteur d'acide.

7. Élément de fixage selon la revendication 6, dans lequel l'accepteur d'acide est l'oxyde de magnésium, l'hydroxyde de calcium, l'oxyde de cuivre (III), l'oxyde de zinc ou leurs mélanges.

8. Élément de fixage selon la revendication 6, dans lequel l'accepteur d'acide et le fluoroélastomère sont présents en des quantités relatives, avant durcissement, d'environ 2 parties en poids de l'accepteur d'acide pour 100 parties en poids du fluoroélastomère à environ 20 parties en poids de l'accepteur d'acide pour 100 parties en poids du fluoroélastomère.

9. Procédé qui comprend (i) la génération d'une image latente électrostatique sur une élément de formation d'images, (ii) le développement de l'image latente par mise en contact de l'élément de formation d'images avec un développant, (iii) le transfert de l'image développée sur un substrat de copie, et (iv) le fixage de l'image développée sur le substrat de copie par mise en contact de l'image développée avec l'élément de fixage selon la revendication 1.

10. Appareil de formation d'images pour former des images sur un support d'enregistrement qui comprend : (i) une surface retenant les charges pouvant recevoir une image latente électrostatique ; (ii) un assemblage de développement pour appliquer du toner sur la surface retenant les charges, afin de développer l'image latente électrostatique pour former une image développée sur la surface retenant les charges ; (iii) un assemblage de transfert pour transférer l'image développée de la surface retenant les charges à un substrat de copie ; et (iv) un assemblage de fixage pour fixer des images de toner sur une surface du substrat de copie, dans lequel l'assemblage de fixage comprend l'élément de fixage selon la revendication 1.
FIG. 3
FIG. 4