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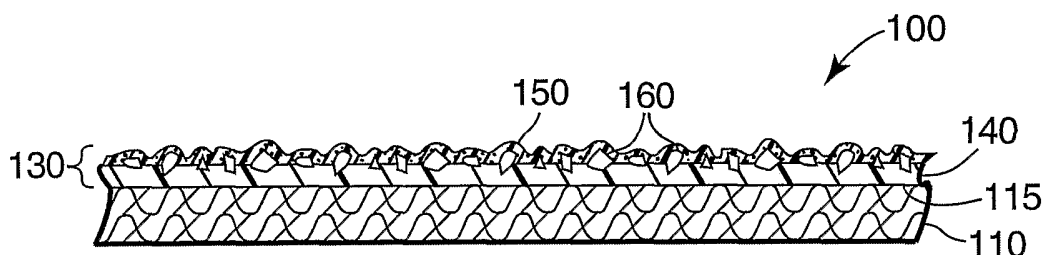
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(54) Title: COMPOSITION, COATED ABRASIVE ARTICLE, AND METHODS OF MAKING THE SAME



(57) Abstract: A composition is made by a method in which a composition comprising a polymerizable compound, a curative, and a hydrolyzable silane coupling agent is combined with water, and the composition is allowed to age. The composition is useful in the manufacture of coated abrasive articles.



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## COMPOSITION, COATED ABRASIVE ARTICLE, AND METHODS OF MAKING THE SAME

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### BACKGROUND

In general, coated abrasive articles have abrasive particles secured to a backing. More typically, coated abrasive articles comprise a backing having two major opposed surfaces and an abrasive layer secured to one of the major surfaces. The abrasive layer typically comprises abrasive particles and a binder, wherein the binder serves to secure the abrasive particles to the backing.

In one common type of coated abrasive article, the abrasive layer comprises a make layer, a size layer, and abrasive particles. In making such a coated abrasive article, a make layer precursor is typically applied to the backing. Abrasive particles are then at least partially embedded into the make layer precursor (for example, by electrostatic coating), and the make layer precursor is polymerized (for example, crosslinked) to provide a make layer having abrasive particles partially embedded therein. A size layer precursor is then applied over the make layer and abrasive particles and is polymerized to provide a size layer.

In some instances, a supersize layer is disposed on the size layer. The supersize layer typically includes, for example, a binder, grinding aids and/or anti-loading materials.

Backings used in coated abrasive articles are optionally treated with one or more applied coatings. Examples of typical backing treatments are a backsize layer (that is, a coating on the major surface of the backing opposite the abrasive layer), a presize layer or a tie layer (that is, a coating on the backing disposed between the abrasive layer and the backing), a saturant that saturates the backing, and/or a subsize. A subsize is similar to a saturant, except that it is applied to a previously treated backing.

Silane coupling agents typically have a reactive silyl group and a second functional group (for example, epoxy, acrylate, or methacrylate). Silane coupling agents have been used as additives to binder precursors to promote adhesion of the resultant binders to certain inorganic materials. For example, silane coupling agents have been used as surface

treatments for abrasive particles that are bonded to and/or included within a size layer precursor, often resulting in improved performance of the resultant coated abrasive article.

#### SUMMARY

5 In one aspect, the present invention provides a method of making a composition, the method comprising:

providing a first polymerizable composition comprising a polyepoxide, a curative, and a hydrolyzable silane coupling agent, wherein the first polymerizable composition has a first level of optical transparency;

10 mixing the first polymerizable composition with water to form a second polymerizable composition, wherein the molar ratio of the water to the hydrolyzable silane coupling agent is less than seven, and wherein the second polymerizable composition has a second level of optical transparency that is less than the first level; and

aging the second polymerizable composition for at least a time sufficient to  
15 provide a third polymerizable composition having a third level of optical transparency that greater than or equal to the first level.

In some embodiments, the third polymerizable composition may be at least partially polymerized.

In another aspect, the present invention provides a method of making a coated  
20 abrasive article, the method comprising:

applying a make layer precursor comprising a first binder precursor onto at least a portion of a major surface of a backing;

at least partially embedding abrasive particles in the make layer precursor; at least partially curing the make layer precursor to provide a make layer;

25 applying a size layer precursor onto at least a portion of the make layer and plurality of abrasive particles; and

at least partially curing the size layer precursor to provide a coated abrasive article, wherein the size layer precursor is prepared by a method comprising:

providing a first polymerizable composition comprising a polyepoxide, a  
30 curative, and a hydrolyzable silane coupling agent, wherein the first polymerizable composition has a first level of optical transparency;

mixing the first polymerizable composition with water to form a second polymerizable composition, wherein the molar ratio of the water to the hydrolyzable silane coupling agent is less than seven, and wherein the second polymerizable composition has a second level of optical transparency that is less than the first level; and

aging the second polymerizable composition for at least a time sufficient to provide a third polymerizable composition having a third level of optical transparency that greater than or equal to the first level.

In yet another aspect, the present invention provides a coated abrasive article comprising:

a backing; and

an abrasive layer secured to at least a portion of the backing, the abrasive layer comprising a make layer, abrasive particles at least partially embedded in the make layer, and a size layer, wherein the size layer is prepared by a method comprising:

providing a first polymerizable composition comprising a polyepoxide, a curative, and a hydrolyzable silane coupling agent, wherein the first polymerizable composition has a first level of optical transparency;

mixing the first polymerizable composition with water to form a second polymerizable composition, wherein the molar ratio of the water to the hydrolyzable silane coupling agent is less than seven, and wherein the second polymerizable composition has a second level of optical transparency that is less than the first level;

aging the second polymerizable composition for at least a time sufficient to provide a third polymerizable composition having a third level of optical transparency that greater than or equal to the first level; and

at least partially polymerizing the third polymerizable composition.

Coated abrasive articles according to the present invention are useful, for example, for abrading a workpiece. Coated abrasive articles prepared according to methods of the present invention typically have one or more improved abrasive performance characteristics.

As used herein,

the term "(meth)acrylate" includes both acrylate and methacrylate; and

the term "optically transparency" as applied to a material refers to that material's ability to transmit visible light through a 0.75-inch (1.9-cm) diameter cylindrical column of the material such that bodies lying beyond are seen clearly by a human eye with 20/20 vision. If a first material has a higher level of optical transparency than a second material, the first material is more optically transparent than the second material.

#### BRIEF DESCRIPTION OF THE DRAWING

The drawing is a cross-sectional view of an exemplary coated abrasive article according to the present invention.

#### DETAILED DESCRIPTION

According to the present invention, an optically transparent first polymerizable composition comprising a polyepoxide, a curative, and a hydrolyzable silane coupling agent is combined with water to form a second polymerizable composition. The second composition is typically an emulsion that lacks optical transparency. The second polymerizable composition is then aged for at least a time sufficient for it to become optical transparent thereby providing a third polymerizable composition, which is useful, for example, as a size layer precursor in the manufacture of coated abrasive articles.

Useful polyepoxides include, for example, alicyclic and aromatic polyepoxides.

Examples of useful alicyclic polyepoxides include monomeric alicyclic polyepoxides, oligomeric alicyclic polyepoxides, and polymeric alicyclic polyepoxides. Exemplary alicyclic polyepoxides monomers useful in practice of the present invention include epoxycyclohexanecarboxylates (for example, 3,4-epoxycyclohexylmethyl 3,4-epoxycyclohexanecarboxylate (available, for example, under the trade designation "ERL-4221" from Dow Chemical Co., Midland, Michigan), 3,4-epoxy-2-methylcyclohexylmethyl 3,4-epoxy-2-methylcyclohexanecarboxylate, bis(3,4-epoxy-6-methylcyclohexylmethyl) adipate, 3,4-epoxy-6-methylcyclohexylmethyl 3,4-epoxy-6-methylcyclohexanecarboxylate (available, for example, under the trade designation "ERL-4201" from Dow Chemical Co.)); vinylcyclohexene dioxide (available, for example, under the trade designation "ERL-4206" from Dow Chemical Co.); bis(2,3-epoxycyclopentyl) ether (available, for example, under the trade designation "ERL-0400" from Dow Chemical Co.), bis(3,4-epoxy-6-methylcyclohexylmethyl) adipate (available, for example,

under the trade designation "ERL-4289" from Dow Chemical Co.), dipenteric dioxide (available, for example, under the trade designation "ERL-4269" from Dow Chemical Co.), 2-(3,4-epoxycyclohexyl-5,1'-spiro-3',4'-epoxycyclohexane-1,3-dioxane, and 2,2-bis(3,4-epoxycyclohexyl)propane.

5           Useful aromatic polyepoxides include, for example, monomeric aromatic polyepoxides, oligomeric aromatic polyepoxides, and polymeric aromatic polyepoxides.

Exemplary aromatic polyepoxides include the polyglycidyl ethers of polyhydric phenols such as bisphenol A-type resins and their derivatives, including polyepoxides having the trade designation "EPON" (for example, "EPON 828" and "EPON 1001F"),  
10           available, for example, from Resolution Performance Products, Houston, Texas; epoxy cresol-novolac resins; Bisphenol-F resins and their derivatives; epoxy phenol-novolac resins; and glycidyl esters of aromatic carboxylic acids (for example, phthalic acid diglycidyl ester, isophthalic acid diglycidyl ester, trimellitic acid triglycidyl ester, and pyromellitic acid tetraglycidyl ester), and mixtures thereof.

15           Commercially available aromatic polyepoxides include, for example, those having the trade designation "ARALDITE" (for example, "ARALDITE MY-720", "ARALDITE 721", "ARALDITE 722", "ARALDITE 0510", "ARALDITE 0500", "ARALDITE PY-306", and "ARALDITE 307"), available, for example, from Ciba Specialty Chemicals, Tarrytown, New York; aromatic polyepoxides having the trade designation "EPON" (for  
20           example, "EPON DPL-862" and "EPON HPT-1079"), available, for example, from Resolution Performance Products; and aromatic polyepoxides having the trade designations "DER", "DEN" (for example, "DEN 438", and DEN 439"), and "QUATREX", available, for example, from Dow Chemical Co.

25           The first polymerizable composition may optionally further comprise a diol such as for example a novolac diol or bisphenol A, or another type of polymerizable material such as, for example, an acrylate monomer, methacrylate monomer, vinyl monomer.

30           The first polymerizable composition may optionally contain one or more curatives for the polymerizable compound(s). The optional curative(s) may be photoactivated and/or thermally activated. If used, the curative(s) is generally present in an amount that is effective (when activated) to at least partially polymerize the third polymerizable  
composition, typically in a range of 0.01 parts to 25 percent by weight, more typically in

the range from 3 to 15 parts by weight, based on the total weight of the third polymerizable composition, although amounts outside of these ranges may also be used.

Useful photoactivated curatives include, for example, photocatalysts and photoinitiators.

5           Examples of useful photoactivated curatives include onium salts and cationic organometallic salts. Useful onium salts include, for example, iodonium complex salts and sulfonium complex salts. Useful aromatic onium complex salts are further described, for example, in U.S. Pat. No. 4,256,828 (Smith). Exemplary aromatic iodonium complex salts include diaryliodonium hexafluorophosphate or a diaryliodonium hexafluoroantimonate.  
10          Exemplary aromatic sulfonium complex salts include as triphenylsulfonium hexafluoroantimonate and p-phenyl(thiophenyl)diphenylsulfonium hexafluoroantimonate.

          Aromatic onium salts, useful in practice of the present invention, are typically photosensitive only in the ultraviolet region of the spectrum. However, they may be sensitized to the near ultraviolet and the visible range of the spectrum by sensitizers for  
15          known photolyzable organic halogen compounds. Exemplary sensitizers include aromatic amines and colored aromatic polycyclic hydrocarbons, as described, for example, in U.S. Pat. No. 4,250,053 (Smith).

          Suitable photoactivated organometallic complex salts useful in the present invention include, for example, those described in U.S. Pat. Nos. 5,059,701 (Keipert),  
20          5,191,101 (Palazzotto et al.), and 5,252,694 (Willett et al.).

          Suitable thermally activated curatives include, for example, aliphatic and aromatic primary and secondary amines such as, for example, di(4-aminophenyl)sulfone, di(4-aminophenyl)ether, and 2,2-bis(4-aminophenyl)propane; aliphatic and aromatic tertiary amines such as, for example, dimethylaminopropylamine; fluorenediamines such as those  
25          described in U.S. Pat. No. 4,684,678; boron trifluoride complexes such as, for example,  $\text{BF}_3 \cdot \text{Et}_2\text{O}$  and  $\text{BF}_3 \cdot \text{H}_2\text{NC}_2\text{H}_4\text{OH}$ ; imidazoles, such as methylimidazole and 2,4-diamino-6-(2'-methylimidazolyl-(1'))-ethyl-s-triazine; hydrazines, such as adipohydrazine; and guanidines, such as tetramethylguanidine and dicyandiamide (cyanoguanidine, also commonly known as DiCy), and mixtures thereof.

30          Suitable hydrolyzable silane coupling agents have hydrolyzable groups bonded to a silicon atom, and additional functionality that is reactive with the polyepoxide by forming a covalent bond, for example, under conditions for polymerization of the polyepoxide.

Examples of hydrolyzable silane coupling agents that may form covalent bonds to epoxy based binders include epoxy-functional silane coupling agents such as, for example, 2-(3,4-epoxycyclohexyl)ethyltriethoxysilane, 2-(3,4-epoxycyclohexyl)ethyltrimethoxysilane, 5,6-epoxyhexyltriethoxysilane, (3-glycidoxypropyl)triethoxysilane, and  
5 (3- glycidoxypropyl)trimethoxysilane); mercapto-functional silane coupling agents such as, for example, 3-mercaptopropyltrimethoxysilane and 3-mercaptopropyltriethoxysilane; amine-functional silane coupling agents such as for example, n-methylaminopropyl-trimethoxysilane, 4-aminobutyltriethoxysilane, N-(2-aminoethyl)-3-aminopropyl-trimethoxysilane, (aminoethylaminomethyl)phenethyltrimethoxysilane, N-(2-aminoethyl)-  
10 3-aminopropyltriethoxysilane. Combinations of more than one hydrolyzable silane coupling agent may also be used.

Hydrolyzable silane coupling agents may be made, for example, by conventional techniques, or they may be purchased from commercial suppliers such as, for example, Gelest, Inc., Morrisville, Pennsylvania; GE Plastics, Pittsfield, Massachusetts; and United  
15 Chemical Technologies, Inc., Horsham, Pennsylvania.

The amount of hydrolyzable silane coupling agent is typically in a range of 0.5 to 3.0 percent by weight, based on the total weight of the first polymerizable composition, although other amounts may also be used. For example, the amount of hydrolyzable silane coupling agent may be in a range of from 0.5 or 1.0 percent by weight up to and  
20 including 2 or even 4 percent by weight, based on the total weight of the first polymerizable composition.

Water is added to the first polymerizable composition in a molar ratio of water to hydrolyzable silane coupling agent up to seven. For example the molar ratio of water to hydrolyzable silane coupling agent may be in a range of from greater than one, two, or  
25 three up to four, five, six, or seven. The water may be added gradually (for example, dropwise) or as a single portion, with or without mixing during addition. Once addition is complete, the combination is typically mixed by any suitable means (for example, mechanical stirring), typically resulting in a hazy or cloudy mixture (that is, second polymerizable composition). Dissipation of the haziness or cloudiness (that is,  
30 corresponding to increased optical transparency) may occur over a period ranging from a minute or less up to hours, days, or even longer. Typically, however, dissipation of haziness or cloudiness occurs within 24 hours.



In some embodiments according to the present invention, the first and/or second polymerizable compositions may be substantially free of volatile organic solvent. In this context, the term "substantially free" means having less than 5 percent by weight, based on the total weight of the first and/or second polymerizable compositions, respectively.

5        Once the haziness or cloudiness has dissipated to a point wherein the second polymerizable composition is at least as optically transparent as the first polymerizable composition, it is referred to as the third polymerizable composition. The third polymerizable composition may be at least partially polymerized. Suitable polymerization techniques typically depend, for example, on the curative(s) and/or polymerizable  
10        compound(s) used. Such techniques are well known in the art and include, for example, exposure to actinic electromagnetic radiation (for example, as generated by a medium pressure mercury vapor lamp), and thermal energy (for example, as generated by a radiant heater or convection oven).

15        Polymerizable compositions according to the present invention are useful as size layer precursors in the manufacture of coated abrasive articles.

      An exemplary coated abrasive article is shown in the drawing, wherein exemplary coated abrasive article 100 according to the present invention has backing 110, and abrasive layer 130 secured to major surface 115 of backing 110. Abrasive layer 130, in turn, includes abrasive particles 160 secured to by make layer 140 and size layer 150.

20        Such coated abrasive articles may be made, for example, by a method in which a make layer precursor is coated onto at least a portion of a major surface of the backing (for example, a treated or untreated backing).

      Suitable make layer precursors are well known in the coated abrasives art. Useful make layer precursors include, for example, free-radically polymerizable compounds,  
25        polyepoxides, phenolic resins, melamine-formaldehyde resins, aminoplast resins, cyanate resins, and combinations thereof, typically in combination with an appropriate curative.

      Typically, the make layer is prepared by coating at least a portion of the backing (treated or untreated) with a make layer precursor. Abrasive particles are then at least partially embedded (for example, by electrostatic coating) in the make layer precursor, and  
30        the make layer precursor is at least partially polymerized. Next, the size layer is prepared by coating at least a portion of the make layer and abrasive particles with a size layer precursor, and at least partially curing the size layer precursor. In one embodiment, the

make layer precursor may be partially polymerized prior to coating with abrasive particles and further polymerized at a later point in the manufacturing process.

Suitable backings include, for example, those known in the art for making coated abrasive articles. Typically, the backing has two opposed major surfaces. The thickness of the backing generally ranges from 0.02 to 5 millimeters, desirably from 0.05 to 2.5 millimeters, and more desirably from 0.1 to 0.4 millimeter, although thicknesses outside of these ranges may also be useful.

The backing may be flexible or rigid, and may be made of any number of various materials including those conventionally used as backings in the manufacture of coated abrasives. Examples include paper, fabric, film, polymeric foam, vulcanized fiber, woven and nonwoven materials, combinations of two or more of these materials. The backing may also be a laminate of two materials (for example, paper/film, cloth/paper, film/cloth).

Exemplary flexible backings include polymeric film (including primed films) such as polyolefin film (for example, polypropylene including biaxially oriented polypropylene, polyester film, polyamide film, cellulose ester film), metal foil, mesh, scrim, foam (for example, natural sponge material or polyurethane foam), cloth (for example, cloth made from fibers or yarns comprising polyester, nylon, silk, cotton, and/or rayon), paper, vulcanized paper, vulcanized fiber, nonwoven materials, and combinations thereof. Cloth backings may be woven or stitch bonded.

The backing may be a fibrous reinforced thermoplastic such as described, for example, as described, for example, in U.S. Pat. No. 5,417,726 (Stout et al.), or an endless spliceless belt, for example, as described, for example, in U.S. Pat. No. 5,573,619 (Benedict et al.). Likewise, the backing may be a polymeric substrate having hooking stems projecting therefrom such as that described, for example, in U.S. Pat. No. 5,505,747 (Chesley et al.). Similarly, the backing may be a loop fabric such as that described, for example, in U.S. Pat. No. 5,565,011 (Follett et al.).

Exemplary rigid backings include metal plates, and ceramic plates. Another example of a suitable rigid backing is described, for example, in U.S. Pat. No. 5,417,726 (Stout et al.).

The backing may be a treated backing having one or more treatments applied thereto such as, for example, a presize, a backsize, a subsize, and/or a saturant. Additional details regarding backing treatments may be found in, for example, U.S. Pat. Nos.

5,108,463 (Buchanan et al.); 5,137,542 (Buchanan et al.); 5,328,716 (Buchanan); and 5,560,753 (Buchanan et al.).

The backing may have one half of an attachment system on its back surface to secure the abrasive article to a support pad or back-up pad. This attachment system half may be, for example, a pressure-sensitive adhesive or tape, a loop fabric for a hook and loop attachment, a hook structure for a hook and loop attachment, or an intermeshing attachment system. Further details concerning such attachment systems may be found, for example, in U.S. Pat. Nos. 5,152,917 (Pieper et al.); 5,454,844 (Hibbard et al.); 5,672,097 (Hoopman); 5,681,217 (Hoopman et al.); and U.S. Pat. Appl. Pub. Nos. 2003/0143938 (Braunschweig et al.) and 2003/0022604 (Annen et al.).

In some instances, it may be desirable to incorporate a pressure-sensitive adhesive onto the back side of the coated abrasive article such that the resulting coated abrasive article may be secured to a back up pad. Exemplary pressure-sensitive adhesives include latex crepe, rosin, acrylic polymers, and copolymers including polyacrylate esters (for example, poly(butyl acrylate)), vinyl ethers (for example, poly(vinyl n-butyl ether)), alkyl adhesives, rubber adhesives (for example, natural rubber, synthetic rubber, chlorinated rubber), and mixtures thereof.

To promote adhesion of the make layer and/or optional backsize layer, it may be necessary to modify the surface to which these layers are applied. Exemplary surface modifications include corona discharge, ultraviolet light exposure, electron beam exposure, flame discharge, and/or scuffing.

Once coated, the make layer precursor may optionally be partially polymerized, for example, to facilitate abrasive particle orientation.

Next, abrasive particles are at least partially embedded in the make layer precursor. Suitable techniques for embedding the abrasive particles include, for example, electrostatic deposition or a mechanical process which maximizes the probability that the individual abrasive particles are positioned with its major axis oriented perpendicular to the backing surface. The make layer precursor is then typically at least partially cured to secure the abrasive particles.

Suitable abrasive particles include, for example, any abrasive particles known in the abrasive art. Exemplary useful abrasive particles include fused aluminum oxide based materials such as aluminum oxide, ceramic aluminum oxide (which may include one or

more metal oxide modifiers and/or seeding or nucleating agents), and heat-treated aluminum oxide, silicon carbide, co-fused alumina-zirconia, diamond, ceria, titanium diboride, cubic boron nitride, boron carbide, garnet, flint, emery, sol-gel derived abrasive particles, and blends thereof. Desirably, the abrasive particles comprise fused aluminum oxide, heat-treated aluminum oxide, ceramic aluminum oxide, silicon carbide, alumina zirconia, garnet, diamond, cubic boron nitride, sol-gel derived abrasive particles, or mixtures thereof. Examples of sol-gel abrasive particles include those described U.S. Pat. No. 4,314,827 (Leitheiser et al.); U.S. Pat. No. 4,518,397 (Leitheiser et al.); U.S. Pat. No. 4,623,364 (Cottringer et al.); U.S. Pat. No. 4,744,802 (Schwabel); U.S. Pat. No. 4,770,671 (Monroe et al.); U.S. Pat. No. 4,881,951 (Wood et al.); U.S. Pat. No. 5,011,508 (Wald et al.); U.S. Pat. No. 5,090,968 (Pellow); U.S. Pat. No. 5,139,978 (Wood); U.S. Pat. No. 5,201,916 (Berg et al.); U.S. Pat. No. 5,227,104 (Bauer); U.S. Pat. No. 5,366,523 (Rowenhorst et al.); U.S. Pat. No. 5,429,647 (Laramie); U.S. Pat. No. 5,498,269 (Larmie); and U.S. Pat. No. 5,551,963 (Larmie). The abrasive particles may be in the form of, for example, individual particles, agglomerates, abrasive composite particles, and mixtures thereof. Exemplary agglomerates are described, for example, in U.S. Pat. No. 4,652,275 (Bloecher et al.) and U.S. Pat. No. 4,799,939 (Bloecher et al.). It is also within the scope of the present invention to use diluent erodible agglomerate grains as described, for example, in U.S. Pat. No. 5,078,753 (Broberg et al.).

Abrasive composite particles comprise abrasive grains in a binder. Exemplary abrasive composite particles are described, for example, in U.S. Pat. No. 5,549,962 (Holmes et al.).

The abrasive particles typically have an average diameter of from 0.1 to 2000 micrometers, more typically from 1 to 1300 micrometers. Coating weights for the abrasive particles may depend, for example, on the binder precursor used, the process for applying the abrasive particles, and the size of the abrasive particles, but typically range from 5 to 1350 grams per square meter.

A size layer precursor comprising the third composition of the present invention is then applied to at least a portion of the make coat precursor and abrasive particles, and at least partially polymerized.

The make and size layer precursors may be coated by any suitable means including, for example, roll coating, dip coating, knife, spray coating, bar coating, powder coating, or hot melt coating.

Suitable curing techniques for the make and size layer precursors are well known in the art and include, for example, e-beam radiation, exposure to actinic electromagnetic radiation (for example, as generated by a medium pressure mercury vapor lamp), and thermal energy (for example, as generated by a radiant heater or convection oven). The choice of specific curing method and conditions will be dictated by the specific make and size layer precursors employed.

In one embodiment, an optional supersize may be applied to at least a portion of the size layer. Useful supersize layers may include, for example, a grinding aid (for example, potassium tetrafluoroborate), metal salts of fatty acids (for example, zinc stearate or calcium stearate), salts of phosphate esters (for example, potassium behenyl phosphate), phosphate esters, urea-formaldehyde resins, mineral oils, crosslinked silanes, crosslinked silicones, and/or fluorochemicals. Further details concerning supersizes may be found, for example, in U. S. Pat. Nos. 3,256,076 (Duwell et al.); 5,520,711 (Helmin); 5,213,589 (Ronning et al.); 5,306,319 (Krishnan); 5,556,437 (Lee et al.); and 6,039,775 (Ho et al.).

Coated abrasive articles according to the present invention may be converted, for example, into belts, tapes, rolls, discs (including perforated discs), and/or sheets. For belt applications, two free ends of the abrasive sheet may be joined together using known methods to form a belt.

Further description of techniques and materials for making coated abrasive articles may be found in, for example, U.S. Pat. Nos. 4,314,827 (Leitheiser et al.); 4,518,397 (Leitheiser et al.); 4,588,419 (Caul et al.); 4,623,364 (Cottringer et al.); 4,652,275 (Bloecher et al.); 4,734,104 (Broberg); 4,737,163 (Larkey); 4,744,802 (Schwabel); 4,751,137 (Tumey et al.); 4,770,671 (Monroe et al.); 4,799,939 (Bloecher et al.); 4,881,951 (Wood et al.); 4,927,431 (Buchanan et al.); 5,498,269 (Larmie); 5,011,508 (Wald et al.); 5,078,753 (Broberg et al.); 5,090,968 (Pellow); 5,108,463 (Buchanan et al.); 5,137,542 (Buchanan et al.); 5,139,978 (Wood); 5,152,917 (Pieper et al.); 5,201,916 (Berg et al.); 5,203,884 (Buchanan et al.); 5,227,104 (Bauer); 5,328,716 (Buchanan); 5,366,523 (Rowenhorst et al.); 5,378,251 (Culler et al.); 5,417,726 (Stout et al.); 5,429,647 (Larmie); 5,436,063 (Follett et al.); 5,490,878 (Peterson et al.); 5,492,550 (Krishnan et al.);

5,496,386 (Broberg et al.); 5,520,711 (Helmin); 5,549,962 (Holmes et al.); 5,551,963 (Larmie); 5,556,437 (Lee et al.); 5,560,753 (Buchanan et al.); 5,573,619 (Benedict et al.); 5,609,706 (Benedict et al.); 5,672,186 (Chesley et al.); 5,700,302 (Stoetzel et al.); 5,942,015 (Culler et al.); 5,954,844 (Law et al.); 5,961,674 (Gagliardi et al.); 5,975,988 (Christianson); 6,059,850 (Lise et al.); and 6,261,682 (Law).

Coated abrasive articles according to the present invention are useful for abrading a workpiece. One such method includes the step of frictionally contacting a coated abrasive article with a surface of the workpiece, and moving at least one of the coated abrasive article or the workpiece relative to the other to abrade at least a portion of the surface.

Examples of workpiece materials include metal, metal alloys, exotic metal alloys, ceramics, glass, wood, wood-like materials, composites, painted surfaces, plastics, reinforced plastics, stone, and/or combinations thereof. The workpiece may be flat or have a shape or contour associated with it. Exemplary workpieces include hardwoods, metal components, plastic components, particleboard, camshafts, crankshafts, furniture, and turbine blades.

Coated abrasive articles according to the present invention may be used by hand and/or used in combination with a machine. At least one or both of the coated abrasive article and the workpiece is moved relative to the other when abrading.

In another aspect, abrading may be conducted under wet or dry conditions. Exemplary liquids for wet abrading include water, water containing conventional rust inhibiting compounds, lubricant, oil, soap, and cutting fluid. The liquid may also contain defoamers, degreasers, and/or the like.

Objects and advantages of this invention are further illustrated by the following non-limiting examples, but the particular materials and amounts thereof recited in these examples, as well as other conditions and, details, should not be construed to unduly limit this invention.

#### EXAMPLES

Unless otherwise noted, all parts, percentages, and ratios reported in the following examples are on a weight basis, and all reagents used in the examples were obtained, or

are available, from general chemical suppliers such as the Sigma-Aldrich Chemical Company, Saint Louis, Missouri, or may be synthesized by conventional techniques.

The following abbreviations are used in the following Examples:

“CAT1”: triarylsulfonium hexafluoroantimonate, 50% in propylene carbonate,  
5 commercially available under the trade designation “CYRACURE UVI-6974” from Dow Chemical Company, Midland, Michigan;

“CAT2”: commercially available under the trade designation “IRGACURE 1173” from Ciba Specialty Chemicals Corporation, Tarrytown, New York;

“FIL1”: anhydrous sodium potassium aluminosilicate available under the trade  
10 designation “MINEX-3” from L. V. Lomas, Ltd., Brampton, Ontario, Canada;

“RES1”: cycloaliphatic epoxide resin commercially available under the trade designation “CYRACURE UVR-6110” from Dow Chemical Company;

“RES2”: trimethylolpropane triacrylate commercially available under the trade designation “TMPTA-N” from UCB Group, Springfield, Massachusetts;

15 “SIL1”: beta-(3,4-epoxycyclohexyl)ethyltriethoxysilane, commercially available under the trade designation “SILQUEST A-186” from General Electric Company, Wilton, Connecticut.; and

“SUP1”: 50 percent by weight aqueous dispersion of zinc stearate/styrene-acrylic acid copolymer (MW >200,000 grams/mole)/sodium dodecylbenzenesulfonate (weight  
20 ratio = 23:3:1).

#### TOTAL CUT TEST

The total cut test is performed as follows:

25 A 5-inch (12.7 cm) test disc is attached to a 5-inch (12.7-cm) foam interface pad, available under the trade designation “HOOKIT II SOFT INTERFACE PAD” from 3M Company, which is then attached to a 5-inch (12.7 cm) by 1.25 inch (3.18 cm) thick vinyl faced foam back up pad available under the trade designation “3M HOOKIT II BACKUP PAD” from 3M Company. The back up pad is mounted on a fine finishing orbital sander available under the trade designation “SANDER #2” from National Detroit, Inc.,  
30 Rockford, Illinois.

The abrasive layer is manually brought into contact with a hard maple panel workpiece, 14 inches x 15 inches (35.6 cm x 38.1 cm). The workpiece is then abraded at 3

inches/second (7.6 cm/second) for 45 seconds at 66 psi (455 kilopascals) and an angle of 10 degrees to the surface of the workpiece. The 45-second abrading cycle is repeated another 4 times, with the amount of material cut after the 1<sup>st</sup>, 2<sup>nd</sup> - 4<sup>th</sup>, and 5<sup>th</sup> cycles recorded, from which the total cut is determined.

5           The above procedure is replicated at least three times and the average total cut is determined from the results.

#### Example 1

10           RES1 (94.8 grams) was mixed with 2 grams SIL1, resulting in a clear mixture (M1). Deionized water (0.2 gram) was mixed with M1, and the resultant cloudy mixture was stirred for 1 hour at 20 °C. Stirring was stopped, and the mixture was covered and allowed to stand for 16 hours. The mixture became clear, and was judged by eye to be at least as optically transparent as mixture M1. CAT1 (3 grams) was added to the mixture, which was then stirred for an additional 30 minutes.

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#### Examples 2-3 and Comparative Examples A-D

          The procedure of Example 1 was repeated using the compositions listed in Table 1 (below), except that it was scaled to 10 g total weight and carried out in a 20-dram (74-mL) cylindrical vial (0.75-inch (1.9-cm) inner diameter), and no CAT1 was added.

20

TABLE 1

	Percent by Weight				Final clarity after mixing and aging
	RES1	SIL1	CAT1	Water	
Example 1	94.8	2.0	3.0	0.2	Clear
Example 2	94.5	2.0	3.0	0.5	Clear
Example 3	94.0	2.0	3.0	1.0	Clear
Comparative Example A	93.0	2.0	3.0	2.0	Cloudy
Comparative Example B	90.0	2.0	3.0	5.0	Cloudy



Comparative Example C	85.0	2.0	3.0	10.0	Cloudy
Comparative Example D	97.0	0	3.0	0	Clear

#### Example 4

RES1 (196 grams) was mixed with 4 grams SIL1, resulting in a clear mixture (M4). Deionized water (0.9 gram) was mixed with M4, and the resultant cloudy mixture was stirred for 1 hour at 20 °C. Stirring was stopped, and the resin mixture was covered and allowed to stand for 16 hours. The mixture became clear, and was judged by eye to be at least as optically transparent as mixture M4. CAT1 (6 grams) was added to the mixture, which was then stirred for an additional 30 minutes. The mixture was brushed onto a 5-inch (12.7 cm) loop-backed grade P80 alumina coated abrasive film disc at a wet coating weight of 70 grams/meter<sup>2</sup>. The abrasive disc was then partially cured by passing once through a UV processor, trade designation "EPIQ 6000", available from Fusion Systems Corp., Rockville, Maryland, with a FUSION D bulb at 0.9 J/cm and 45 feet/minute (13.7 meters/minute). The abrasive disc was then thermally cured for 10 minutes at 150 °C. The abrasive disc was flexed in order to lay flat. SUP1 was applied by brush at 20 °C and approximately 14 grams/meter<sup>2</sup> wet weight, and then dried for 10 minutes at 180 °F (82.2 °C). A total of three abrasive discs were prepared in this fashion.

#### Example 5

The procedure described in Example 4 was repeated, except that the quantity of SIL1 was increased to 8 grams, and the quantity of water was increased to 1.8 grams.

#### Example 6

The procedure described in Example 5 was repeated, except that 101.5 grams FIL1 was added immediately following the addition of the catalyst and the mixture coated onto the abrasive disc at 20 °C and a wet coating weight of 85 grams/meter<sup>2</sup>.

Comparative Example E

The procedure described in Example 4 was repeated, except that the deionized water was omitted.

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Comparative Example F

The procedure described in Comparative Example E was repeated, except that both the water and SIL1 were omitted, and a total of four abrasive discs were prepared.

Comparative Example G

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The procedure described in Comparative Example F was repeated, except that three discs were prepared and thermally cured for 10 minutes at 140 °C.

Comparative Example H

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The procedure described in Example 6 was repeated, except that the 58.8 grams RES1 was replaced with an equal quantity of RES2 and 3 grams of CAT2.

Performance of coated abrasives of Examples 4-7 and Comparative Examples E-G was evaluated using the Total Cut Test. Results are reported in Table 2 (below).

TABLE 2

	Molar Ratio of Added Water to Silane Coupling Agent	CUT, grams				
		1 <sup>ST</sup> Cycle	2 <sup>ND</sup> - 4 <sup>TH</sup> Cycle	5 <sup>TH</sup> Cycle	Total Cut	Average Total Cut
Example 4	1.4	4.47, 4.38, 4.38	9.99, 9.63, 9.58	2.99, 2.81, 2.79	17.45, 16.82, 16.75	17.01
Example 5	1.4	4.51, 4.47, 4.49	10.01, 9.90, 9.82	2.96, 2.87, 2.93	17.48, 17.24, 17.24	17.32
Example 6	1.4	4.46, 4.31, 4.45	10.02, 9.48, 9.89	2.92, 2.74, 2.91	17.40, 16.53, 17.25	17.06
Comparative Example E	Not Applicable	4.20, 4.28, 4.17	9.18, 9.36, 8.98	2.77, 2.70, 2.68	16.15, 16.34, 15.83	16.11
Comparative Example F	Not Applicable	4.23, 4.20, 4.24, 4.32	9.32, 9.18, 9.61, 9.29	2.89, 2.67, 2.85, 2.72	16.44, 16.05, 16.70, 16.33	16.38
Comparative Example G	Not Applicable	4.25, 4.27, 4.27	9.60, 9.36, 9.47	2.78, 2.66, 2.72	16.63, 16.29, 16.46	16.46
Comparative Example H	Not Applicable	4.33, 4.38, 4.35	9.69, 9.82, 9.66	2.83, 2.82, 2.68	16.85, 17.02, 16.69	16.85

Various modifications and alterations of this invention may be made by those skilled in the art without departing from the scope and spirit of this invention, and it should be understood that this invention is not to be unduly limited to the illustrative embodiments set forth herein.

What is claimed is:

1. A method of making a composition, the method comprising:  
providing a first polymerizable composition comprising a polyepoxide, a curative,  
5 and a hydrolyzable silane coupling agent, wherein the first polymerizable composition has  
a first level of optical transparency;  
mixing the first polymerizable composition with water to form a second  
polymerizable composition, wherein the molar ratio of the water to the hydrolyzable silane  
coupling agent is less than seven, and wherein the second polymerizable composition has  
10 a second level of optical transparency that is less than the first level; and  
aging the second polymerizable composition for at least a time sufficient to  
provide a third polymerizable composition having a third level of optical transparency that  
greater than or equal to the first level.
- 15 2. A composition prepared according to the method of claim 1.
3. A method according to claim 1, further comprising at least partially polymerizing  
the third polymerizable composition.
- 20 4. A composition prepared according to the method of claim 3.
5. A method according to claim 1, wherein the curative comprises a photocatalyst.
6. A method according to claim 1, wherein the curative comprises at least one of a  
25 thermal catalyst or thermal crosslinking agent.
7. A method according to claim 1, wherein the hydrolyzable silane coupling agent is  
selected from the group consisting of epoxy-functional silane coupling agents, mercapto-  
functional silane coupling agents, amine-functional silane coupling agents, and hydroxy-  
30 functional silane coupling agents.

8. A method according to claim 1, wherein the first polymerizable composition and second polymerizable compositions are substantially free of volatile organic solvent.
9. A method according to claim 1, wherein the molar ratio of water to hydrolyzable silane coupling agent is at least one.
10. A method according to claim 1, wherein the molar ratio of water to hydrolyzable silane coupling agent is at least two.
11. A method of making a coated abrasive article, the method comprising:  
applying a make layer precursor comprising a first binder precursor onto at least a portion of a major surface of a backing;  
at least partially embedding abrasive particles in the make layer precursor; at least partially curing the make layer precursor to provide a make layer;  
applying a size layer precursor onto at least a portion of the make layer and plurality of abrasive particles; and  
at least partially curing the size layer precursor to provide a coated abrasive article, wherein the size layer precursor is prepared by a method comprising:  
providing a first polymerizable composition comprising a polyepoxide, a curative, and a hydrolyzable silane coupling agent, wherein the first polymerizable composition has a first level of optical transparency;  
mixing the first polymerizable composition with water to form a second polymerizable composition, wherein the molar ratio of the water to the hydrolyzable silane coupling agent is less than seven, and wherein the second polymerizable composition has a second level of optical transparency that is less than the first level; and  
aging the second polymerizable composition for at least a time sufficient to provide a third polymerizable composition having a third level of optical transparency that greater than or equal to the first level.
12. A method according to claim 11, wherein the hydrolyzable silane coupling agent is selected from the group consisting of epoxy-functional silane coupling agents, mercapto-

functional silane coupling agents, amine-functional silane coupling agents, and hydroxy-functional silane coupling agents.

13. A method according to claim 11, wherein the first polymerizable composition and  
5 second polymerizable compositions are substantially free of volatile organic solvent.

14. A method according to claim 11, wherein the curative comprises a photocatalyst.

15. A method according to claim 11, wherein the curative comprises at least one of a  
10 thermal catalyst or thermal crosslinking agent.

16. A method according to claim 11, wherein the molar ratio of water to hydrolyzable  
silane coupling agent is at least one.

17. A method according to claim 11, wherein the molar ratio of water to hydrolyzable  
15 silane coupling agent is at least two.

18. A method according to claim 11, further comprising applying a supersize layer  
precursor to at least a portion of the at least partially cured size layer, and at least partially  
20 curing the supersize layer precursor.

19. A coated abrasive article comprising:  
a backing; and  
an abrasive layer secured to at least a portion of the backing, the abrasive layer  
25 comprising a make layer, abrasive particles at least partially embedded in the make layer,  
and a size layer, wherein the size layer is prepared by a method comprising:  
providing a first polymerizable composition comprising a polyepoxide, a  
curative, and a hydrolyzable silane coupling agent, wherein the first polymerizable  
composition has a first level of optical transparency;  
30 mixing the first polymerizable composition with water to form a second  
polymerizable composition, wherein the molar ratio of the water to the  
hydrolyzable silane coupling agent is less than seven, and wherein the second

polymerizable composition has a second level of optical transparency that is less than the first level;

aging the second polymerizable composition for at least a time sufficient to provide a third polymerizable composition having a third level of optical transparency that greater than or equal to the first level; and  
at least partially polymerizing the third polymerizable composition.

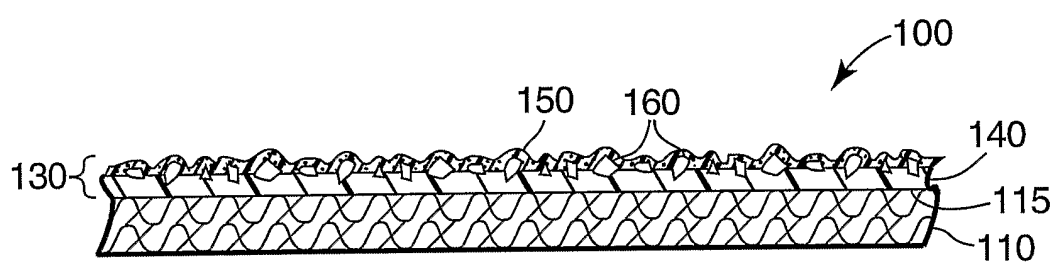
20. A coated abrasive article 19, wherein the hydrolyzable silane coupling agent is selected from the group consisting of epoxy-functional silane coupling agents, mercapto-functional silane coupling agents, amine-functional silane coupling agents, and hydroxy-functional silane coupling agents.

21. A coated abrasive article according to claim 19, wherein the backing comprises a treated backing having at least one treatment selected from the group consisting of a presize, a backsize, a subsize, and a saturant.

22. A coated abrasive article 19, further comprising a supersize layer disposed on at least a portion of the size layer.

23. A method of abrading a workpiece comprising:  
providing a coated abrasive article according to claim 19;  
frictionally contacting at least a portion of the abrasive layer with at least a portion of the surface of the workpiece; and moving at least one of the coated abrasive article or the workpiece relative to the other to abrade at least a portion of the surface.

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## INTERNATIONAL SEARCH REPORT

International Application No  
PCT/US2005/024724

<b>A. CLASSIFICATION OF SUBJECT MATTER</b> B24D3/28      B24D11/00      C08G59/40		
According to International Patent Classification (IPC) or to both national classification and IPC		
<b>B. FIELDS SEARCHED</b> Minimum documentation searched (classification system followed by classification symbols) B24D    C08G		
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		
<b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 4 156 046 A (LIEN, LARRY A ET AL) 22 May 1979 (1979-05-22)	1-10
Y	column 2 - column 7	11-18
X	US 6 077 601 A (DEVOE ET AL) 20 June 2000 (2000-06-20)	19-23
Y	cited in the application column 4, line 20 - column 5, line 35	11-18
A	US 6 132 860 A (BRUXVOORT ET AL) 17 October 2000 (2000-10-17)	1-18
X	the whole document	19-23
<input type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex.		
* Special categories of cited documents : *A* document defining the general state of the art which is not considered to be of particular relevance *E* earlier document but published on or after the international filing date *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) *O* document referring to an oral disclosure, use, exhibition or other means *P* document published prior to the international filing date but later than the priority date claimed *T* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention *X* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone *Y* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art. *&* document member of the same patent family		
Date of the actual completion of the international search  14 November 2005		Date of mailing of the international search report  22/11/2005
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016		Authorized officer  Eschbach, D

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No  
PCT/US2005/024724

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 4156046	A	22-05-1979	BE 865312 A1	25-09-1978
			ZA 7801716 A	28-03-1979
US 6077601	A	20-06-2000	BR 9910108 A	26-12-2000
			CA 2330853 A1	11-11-1999
			CN 1308570 A	15-08-2001
			DE 69922945 D1	03-02-2005
			DE 69922945 T2	19-05-2005
			EP 1075354 A1	14-02-2001
			JP 2002513684 T	14-05-2002
			WO 9956913 A1	11-11-1999
			US 6372336 B1	16-04-2002
			US 6258138 B1	10-07-2001
			US 6359027 B1	19-03-2002
US 6132860	A	17-10-2000	CA 2134334 A1	25-11-1993