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METHOD FOR IMPARTING PERMANENT
DEFORMATION TO TEXTILES

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This invention relates to and has as its primary object the provision of novel methods for producing textile materials having permanent creases, folds, pleats, or other deformations as may be selected by the operator.

It is of course well known that garments of all types are provided with some sort of fabric arrangement, for example, pleats, creases, etc. Such deformations are conventionally produced by ironing the garment while constrained in the desired pattern. However, these arrangements are but temporary and when the garment is subjected to wear or is exposed to humid conditions the creases, pleats, etc. disappear leaving the garment in a shapeless condition. Materials such as starch are widely employed to obtain more durable creases. However, impregnation of the textile with starch and similar materials has the drawback that the textile is rendered stiff and harsh so that it does not have the original characteristics of the untreated fabric.

A principal feature of the process of the invention is that the textile material is provided with permanent deformations without materially affecting the hand of the textile. Thus the textile material treated in accordance with the invention exhibits essentially the same flexibility, drape, and other qualities contributing to its hand as the original fabric. Moreover, tensile strength, porosity, resiliency, and other valuable properties of the textile are retained.

Briefly described, the process of the invention involves the steps of impregnating the textile material with a polyamide and a polyepoxide, arranging the impregnated textile in the desired pattern of deformation and then while constraining it in such pattern applying heat to cure the polyamide-polyepoxide deposit on the fibers. Application of heat causes the polyamide and polyepoxide to react producing a resinous reaction product on the fibers which is insoluble in water, conventional aqueous laundering media, and dry cleaning solvents. The curing is most conveniently accomplished by pressing the textile while applying direct steam thereto as for example with a conventional tailor's steam press.

Creases, pleats, and similar fabric patterns produced in accordance with the invention are permanent as this expression is understood in the textile industry. That is, the fabrics may be subjected to immersion in water, laundering in aqueous medium, dry cleaning, or long wear as garments without substantial loss of the fabric arrangement. At the same time the fabric retains its original hand so that the treated material is not harsh or boardy but is suitable for all garment usage. It is also to be noted that the process in accordance with the invention is not to be confused with procedures wherein lapping cloth sections are cemented or otherwise adhered together. Such action does not take place with the pro-

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cedure described herein; the fabric pattern is maintained by a permanent deformation of fabric areas. There is no adherence between fabric areas.

An additional advantage of the invention is that properties of the treated fabric are generally upgraded by the treatment. A notable point is that the treated fabric is much more resistant to shrinkage when subjected to laundering than is the untreated fabric. Also, the treated fabric is highly resistant to wrinkling and creasing as compared with the original material.

The textile material is most conveniently impregnated with the polyepoxide and polyamide by applying these compounds in the form of a dispersion in a volatile, inert, liquid carrier. The expression "dispersion" is used herein in a generic sense as including true solutions, colloidal solutions, suspensions, emulsions, and the like. The two agents, polyepoxide and polyamide, may both be in a single dispersion or separate dispersions of each may be applied serially to the textile. The dispersion or dispersions may be applied to the textile in any of the usual ways as by immersion, brushing, spraying, etc. To assist in wetting the textile and removing excess liquid, it may be run through padding rolls, wringer rolls, or the like. The proportion of active material in the dispersion or dispersions and the amount of liquid left in the textile is usually selected so that there is deposited on the textile about 0.5 to 10% of its weight of the mixture of polyepoxide and polyamide. In general, the greater the proportion of these agents, the more permanent will be the fiber deformation and the greater will be the shrinkage protection and wrinkle-resistance imparted to the textile.

In forming the dispersion for impregnating the textile, water may be used as the liquid carrier where the selected polyepoxide and polyamide are soluble in this medium. In many cases organic solvents are more preferably employed, for example, methanol, ethanol, propanol, isopropyl alcohol, any of the isomeric butyl alcohols, acetone, benzene, toluene, monomethyl ether of ethylene glycol, monoethyl ether of ethylene glycol, etc. Mixtures of solvents may be used and particularly advantageous are mixtures of (a) hydrocarbon solvents such as benzene, toluene, petroleum naphthas, xylene, etc., and (b) alcohol, hydroxy-ether, or ketone solvents, such as butanols, acetone, methyl-ethyl ketone, monomethyl ether of ethylene glycol, monoethyl ether of ethylene glycol, etc. The polyepoxide and polyamide may also be dissolved in solvent systems containing water and water-miscible solvents such as methanol, ethanol, propanol, isopropyl alcohol, acetone, etc. As noted above, both polyepoxide and polyamide may be dissolved in a single batch of solvent or separate solutions of the two agents may be prepared for serial application in any order to the textile. Where the solutions are applied serially, the textile may be dried after application of the first solution and prior to treatment with the second.

In accordance with a preferred embodiment of the invention, the polyepoxide and polyamide are deposited on the textile by impregnating it with an aqueous emulsion containing both the polyepoxide and polyamide. Such aqueous emulsions can be prepared by any of the known emulsification techniques. A preferred procedure is to dissolve the polyepoxide and polyamide in separate batches of alcohol and add these alcohol solutions to the water with vigorous agitation. In this manner the contact of the alcohol solutions with the water will precipitate the polyepoxide and polyamide in minute particles which are relatively easy to emulsify. To assist in forming and maintaining the emulsion, one may add a small proportion, on the order of 0.1 to 5%, of a conventional emulsifying agent. For such purpose one may employ agents such as soaps, long chain alkyl sodium

sulphates or sulphonates, long chain alkyl benzene sodium sulphonates, esters of sulphosuccinic acid, etc., typical examples being sodium oleate, sodium lauryl sulphate, sodium dodecane sulphonate, sodium alkyl (C_{10} - C_{20}) benzene sulphonate, sodium dioctylsulphosuccinate, etc. Preferably, agents of the non-ionic type are used, for example, the reaction products of ethylene oxide with fatty acids, polyhydric alcohols, alkyl phenols, and so forth. Typical examples of such agents are a polyoxyethylene stearate containing 20 oxyethylene groups per mol, a polyoxyethylene ether of sorbitan monolaurate containing 16 oxyethylene groups per mol, a distearate of polyoxyethylene ether of sorbitol containing 40 oxyethylene groups per mol, isooctyl ether of polyethylene glycol, and so forth. Cationic agents may also be used for example, long-chain alkyl trimethyl ammonium chlorides, bromides, and methosulphates. Other suspending agents such as gums, gelatin, pectin, soluble starch, dextrans, etc. can of course be employed to keep the active agents in suspension. Although water is ordinarily the primary vehicle in the emulsions, the emulsions may contain, in addition to water, organic water-miscible solvents such as methanol, ethanol, propanol, isopropyl alcohol, acetone, and the like.

The total concentration of polyepoxide and polyamide in the emulsion or other type of dispersion is not critical and may be varied depending on the amount of these agents to be deposited on the textile, on the type of impregnation technique employed, and particularly on the wet pickup of dispersion by the textile. For practical purposes dispersions containing about from 1% to 20%, usually about from 5 to 10%, of polyepoxide and polyamide are generally used.

As noted hereinabove, it is not essential to employ a single emulsion containing both polyepoxide and polyamide. These agents may be separately prepared as aqueous emulsions and these emulsions applied serially in any order to the textile. If desired, the textile may be dried after application of the first emulsion and prior to application of the second emulsion.

Ordinarily the textile material is uniformly impregnated with the polyepoxide and polyamide. However, it may be desirable to apply the said compounds only in the areas where the permanent deformation is desired. Another alternative is to uniformly treat the textile with the polyepoxide and polyamide and apply an extra amount thereof on the areas where the permanent deformations are to be formed. For example, in the treatment of trousers in accordance with the invention, the polyamide and polyepoxide may be uniformly applied to the garment and an extra amount of the compounds applied in stripes along the portions of the legs of the garment where the conventional creases will be made. By such technique, the permanent character of the textile configuration is especially assured.

After the textile has been impregnated with the polyepoxide and polyamide, it is ready for the next steps of deforming and curing. Usually it is preferred to dry the textile in air completely or partly prior to further processing. The textile, impregnated with the polyepoxide and polyamide, is deformed by any suitable means into the desired pattern. The deformation may involve such manipulations as folding over sections of the textile, creasing, pleating, or shaping on suitable mandrels or dies. For example, in applying the invention to garments, these articles are arranged on a flat surface with the areas folded over, creased, or otherwise arranged as desired. Where more complex configurations are involved the textile may be compressed between dies having the desired shape. For example, ribbed effects may be attained by constraining the fabric between dies having corrugated surfaces; pleated effects may be obtained by constraining the fabric between dies having mating V-shaped projections. Yarns may be crimped by compressing between corrugated surfaces or given a perma-

nent twist by twisting the yarn while being wound in a package and curing the package of twisted yarn in toto. Further extensions of these principles will be obvious to those skilled in the textile art. In any event, the textile is constrained in the deformed position while heat is applied to it to form the insoluble polyamide-polyepoxide resin and set the deformation. The cure may be accomplished by directly contacting the textile with steam, hot water, or by the use of heated platens, rollers, dies, or the like. For the establishment of pleats, creases and the like a conventional tailor's steam press is quite satisfactory as the textile can be subjected to direct contact with steam while held in position in the deformed state by the press platens. Generally the textile is heated to about 100 to 200° C. and held at this temperature range until the cure is complete. The time for cure will vary depending on the reactivity of the polyamide and polyepoxide and particularly on the temperature. Thus the time of cure is decreased as the temperature of cure is increased. In any particular case, pilot tests on small samples of textile can be carried out to determine time for optimum cure, that is, the time required to form an insoluble resin and set the deformation. In general, the time for proper cure will vary from about 1 minute to 30 minutes. For example, at 100° C. the cure may require 15 to 30 minutes whereas at 200° C. the cure may require only about 1 to 5 minutes.

During the curing step, the polyepoxide and polyamide react in situ forming an insoluble resinous reaction product on the fibers of the textile. Since the textile is constrained in a deformed shape during the curing operation, the resinous coating on the fibers resiliently maintains the textile in the predetermined configuration after the mechanical forces holding the textile in constraint are removed. The resinous material formed on the fibers effects a permanent deformation because it is essentially insoluble in water, aqueous laundering media, dry cleaning solvents, etc. The mechanism of the interaction between the polyepoxide and polyamide is believed to involve primarily reaction between (a) the free primary amine ($-NH_2$) groups and free secondary amine groups ($-NH-$) of the polyamide with (b) the epoxy groups of the polyepoxide to form direct $-N-C-$ bonds between the polyamide and polyepoxide. Also, reaction may take place between free carboxyl groups in the polyamide and the epoxy groups of the polyepoxide forming ester linkages between the two compounds. In addition, reaction may take place between the epoxy groups of the polyepoxide and amino residues in the wool being treated.

The polyepoxides used in accordance with the invention are organic compounds having at least two epoxy groups per molecule and may be saturated or unsaturated, aliphatic, cycloaliphatic, aromatic or heterocyclic and may be substituted with non-interfering substituents such as hydroxyl groups, ether radicals, and the like. Polyepoxides containing ether groups, generally designated as polyepoxide polyethers, may be prepared as well known in the art by reacting a polyol with a halogen-containing epoxide employing at least 2 moles of the halogen-containing epoxide per mole of polyol. Thus, for example, epichlorohydrin may be reacted with a polyhydric phenol in an alkaline medium. In another technique the halogen-containing epoxide is reacted with a polyhydric alcohol in the presence of an acid-acting catalyst such as hydrofluoric acid or boron trifluoride and the product is then reacted with an alkaline compound to effect a dehydrohalogenation. A preferred example of the halogen-containing epoxide is epichlorohydrin; others are epibromohydrin, epiodohydrin, 3-chloro-1,2-epoxybutane, 3-bromo-1,2-epoxyhexane, and 3-chloro-1,2-epoxycyclohexane. Examples of polyols which may be reacted with the halogen-containing epoxide are glycerol, diglycerol, propylene glycol, ethylene glycol, diethylene glycol, butylene glycol, hexanetriol, sorbitol, mannitol, pentane-

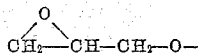
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triol, pentaerythritol, dipentaerythritol, polyglycerol, dulcitol, inositol, carbohydrates, methyltrimethylol propane, 2,6-octanediol, tetrahydroxycyclohexane, 2-ethyl hexanetriol-1, 2, 6, glycerol methyl ether, glycerol allyl ether, polyvinyl alcohol, polyallyl alcohol, resorcinol, catechol, hydroquinone, 4,4'-dihydroxydiphenyl ether, methyl resorcinol, 2,2-bis (parahydroxyphenyl) propane, 2,2-bis (parahydroxyphenyl) butane, 4,4'-dihydroxybenzophenone, bis (parahydroxyphenyl) ethane, 1,5-dihydroxynaphthalene, 1,4-dihydroxycyclohexane, bis (2,2'-dihydroxydinaphthyl) methane, etc. Illustrative examples of polyepoxide polyethers are as follows:

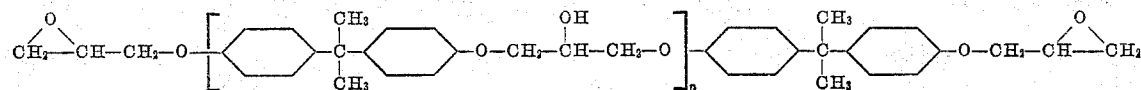
1,4-bis (2,3-epoxypropoxy) benzene; 1,3-bis (2,3-epoxypropoxy) benzene; 4,4'-bis (2,3-epoxypropoxy) diphenyl ether; 1,8-bis (2,3-epoxypropoxy) octane; 1,4-bis (2,3-epoxypropoxy) cyclohexane; 4,4'-bis (2-hydroxy-2,4-epoxybutoxy) diphenyl dimethylmethane; 1,3-bis (4,5-epoxypentoxo)-5-chlorobenzene; 1,4-bis (3,4-epoxybutoxy)-2-chlorocyclohexane; diglycidyl thioether; diglycidyl ether; ethylene glycol diglycidyl ether; propylene glycol diglycidyl ether; diethylene glycol diglycidyl ether; resorcinol diglycidyl ether; 1,2,3,4-tetrakis (2-hydroxy-2,4-epoxybutoxy) butane; 2,2-bis (2,3-epoxypropoxyphenyl) propane; glycerol triglycidyl ether; mannitol tetraglycidyl ether; pentaerythritol tetraglycidyl ether; sorbitol tetraglycidyl ether; glycerol diglycidyl ether; etc. It is evident that the polyepoxide polyethers may or may not contain hydroxy groups, depending primarily on the proportions of halogen-containing epoxide and polyol employed. Polyepoxide polyethers containing polyhydroxyl groups may also be prepared by reacting, in known manner, a polyhydric alcohol or polyhydric phenol with a polyepoxide in alkaline medium. Illustrative examples are the reaction product of glycerol and diglycidyl ether, the reaction product of sorbitol and bis (2,3-epoxy-2-methylpropyl) ether, the reaction product of pentaerythritol and 1,2,4,5-diepoxy pentane, the reaction product of 2,2-bis (parahydroxyphenyl) propane and bis (2,3-epoxy-2-methylpropyl) ether, the reaction product of resorcinol and diglycidyl ether, the reaction product of catechol and diglycidyl ether, and the reaction product of 1,4-dihydroxycyclohexane, and diglycidyl ether.

Polyepoxides which do not contain ether groups may be employed as for example 1,2,5,6-diepoxyhexane; butadiene dioxide (that is, 1,2,3,4-diepoxybutane); isoprene dioxide; limonene dioxide.

For use in accordance with the invention, we prefer the polyepoxides which contain ether groups, that is, polyepoxide polyethers. More particularly we prefer to use the polyepoxide polyethers of the class of glycidyl polyethers of polyhydric alcohols or glycidyl polyethers of polyhydric phenols. These compounds may be considered as being derived from a polyhydric alcohol or polyhydric phenol by etherification with at least two glycidyl groups—



The alcohol or phenol moiety may be completely etherified or may contain residual hydroxy groups. Typical examples of compounds in this category are the glycidyl polyethers of glycerol, glycol, diethylene glycol, 2,2-bis (parahydroxyphenyl) propane, or any of the other polyols listed hereinabove as useful for reaction with halogen-containing epoxides. Many of the specific glycidyl polyethers derived from such polyols are set forth hereinabove. Particularly preferred among the glycidyl polyethers are those derived from 2,2-bis (parahydroxyphenyl) propane and those derived from glycerol. The compounds derived from the first-named of these polyols have the structure—



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wherein n varies between zero and about 10, corresponding to a molecular weight about from 350 to 8,000. Of this class of polyepoxides we prefer to employ those compounds wherein n has a low value, i.e., less than 5, most preferably where n is zero.

It is within the purview of the invention to employ mixtures of different polyepoxides. Indeed, it has been found that especially desirable results are attained by employing mixtures of two commercially-available polyepoxides, one being essentially a diglycidyl ether of glycerol, the other being essentially a diglycidyl ether of 2,2-bis (parahydroxyphenyl) propane. Thus it has been found that a mixture of these compounds produces a very desirable combination of maximum shrinkproofing effect coupled with a minimum alteration of the natural hand of the textile. Particularly preferred to attain such result are mixtures containing more than 1 and less than 10 parts by weight of the glycerol diglycidyl ether per part by weight of the diglycidyl ether of 2,2-bis (parahydroxyphenyl) propane.

The polyamides used in accordance with the invention are those derived from polyamines and polybasic acids. Methods of preparing these polyamides by condensation of polyamines and polycarboxylic acids are well known in the art and need not be described here. One may prepare polyamides containing free amino groups or free carboxylic acid groups or both free amino and free carboxylic acid groups. Generally it is preferred to employ polyamides which contain free amino groups since the active hydrogens on these groups are especially reactive with the epoxy groups of the polyepoxide to form insoluble polyepoxide-polyamide reaction products. The polyamides may be derived from such polyamines as ethylene diamine, diethylene triamine, triethylene tetramine, tetraethylene pentamine, 1,4-diamino butane, 1,3-diaminobutane, hexamethylene diamine, 3-(N-isopropylamino) propylamine, 3,3'-imino-bispropylamine, and the like. Typical polycarboxylic acids which may be condensed with the polyamines to form polyamides are glutaric acid, adipic acid, pimelic acid, suberic acid, azelaic acid, sebacic acid, isophthalic acid, terephthalic acid, beta-methyl adipic acid, 1,2-cyclohexane dicarboxylic acid, malonic acid, polymerized fat acids, and the like. Depending on the amine and acid constituents and the conditions of condensation, the polyamides may have molecular weights varying about from 1,000 to 10,000 and melting points about from 20–200° C. Particularly preferred for the purpose of the invention are the polyamides derived from aliphatic polyamines and polymeric fat acids. Such products are disclosed for example by Cowan et al., Patent No. 2,450,940. Typical of these polyamides are those made by condensing ethylene diamine or diethylene triamine with polymeric fat acids produced from the polymerization of drying or semi-drying oils, or the free acids, or simple aliphatic alcohol esters of such acids. The polymeric fat acids may typically be derived from such oils as soybean, linseed, tung, perilla oil, cottonseed, corn, tall, sunflower, safflower, and the like. As well known in the art, in the polymerization the unsaturated fat acids combine to produce a mixture of dibasic and higher polymeric acids. Usually the mixture contains a preponderant proportion of dimeric acids with lesser amounts of trimeric and higher polymeric acids, and some residual monomeric acid. Particularly preferred are the polyamides of low melting point (about 20–90° C.) which may be produced by heating together an aliphatic polyamine, such as diethylene triamine, triethylene tetramine, 1,4-diaminobutane, 1,3-diaminobutane, and the like with the polymerized fat acids. Typical among these

is a polyamide derived from diethylene triamine and dimerized soybean fatty acids.

As noted above, the total amount of polyepoxide and polyamide deposited on the textile is about from 0.5 to 10% of the weight of the textile. The relative proportions of polyepoxide and polyamide may be varied widely, for example, from 0.1 to 10 parts by weight of polyamide per part by weight of polyepoxide. In many cases mixtures containing 30 to 70% by weight of polyamide and 70 to 30% by weight of polyepoxide give superior results. Since the number of reactive groups in the polyepoxide and polyamide may vary, the proportions for optimum results may be more accurately described by stoichiometric relations. Thus it is preferred that the polyamide be employed in such proportion as to provide about from 0.2 to 1.6 amino groups per epoxy group provided by the polyepoxide.

The process of the invention may be applied to all-wool textiles or textiles containing wool blended with other fibers, for example: animal hair; mohair; silk; synthetic fibers made from proteins such as zein, casein, peanut protein, soybean protein, keratins, etc.; cellulosic fibers such as cotton, linen, rayon, viscose, cellulose acetate, jute, hemp, etc.; nylon; Dynel; Orlon; Dacron; or other organic textile fibers. The expression "wool-containing textile" as used herein is intended to encompass all-wool textiles and blended textiles containing a significant proportion, that is, at least 25% by weight of wool. The process of the invention may be applied to wool-containing textile materials in the form of threads, fibers, yarns, slivers, woven or knitted fabrics, or garments made of woven or knitted fabrics. The textiles may be white or dyed goods. Typical applications of the invention are: to provide garments with pleats, creases or other arrangements customary in tailoring; to provide sheets of fabric with pre-formed pleats so that the product may be used for production of skirts and the like; to produce crimp, twist, or other configuration in yarns, threads and the like. Further applications of the invention will be obvious to those skilled in the art from the above illustrations.

The invention is further demonstrated by the following illustrative examples:

Example I

An aqueous emulsion containing 0.8% of polyamide, 1.2% of polyepoxide, and 2% of emulsifying agent was prepared. The polyamide was a commercial compound being a condensation product of diethylene triamine and heat-dimerized unsaturated fat acids. It was a viscous liquid at ordinary temperatures with a viscosity of A_3-A_1 on the Gardner-Holdt scale at 25° C. and a specific gravity of 0.99 at 25° C. The polyepoxide was a commercial product consisting essentially of 2,2-bis (2,3-epoxypropoxyphenyl) propane prepared by reacting epichlorhydrin with 2,2-bis (parahydroxyphenyl) propane. The emulsifying agent was the iso-octylphenyl ether of polyethylene glycol (Triton-X-100).

A sample of woolen cloth was immersed in the emulsion to wet it thoroughly. The cloth was removed from the emulsion and squeezed to such an extent that the wet cloth was about twice as heavy as the original dry cloth. The wet cloth was laid flat on a towel and allowed to dry in air until its weight was about 1.5 times the original dry weight. The cloth was then placed on the bed of a steam press and arranged in several pleats. The press was then closed onto the pleated cloth and steam at 60-70 lbs. pressure was admitted into the closed press. Subjection to steam was continued for 3 minutes then the press was opened and the cloth allowed to dry for a minute on the bed. The pickup of cured resin was about 2%, based on the weight of the original cloth.

Another sample of the cloth but without the impregna-

tion with polyamide and polyepoxide was pleated and steam pressed as was the first sample.

Both samples of cloth were then laundered with soap and water in a household-type automatic washer. It was observed that the treated sample still retained the pleats sharply whereas in the control sample the pleats had disappeared. It was also observed that the treated cloth shrank about 1% in area whereas the untreated cloth shrank about 40% in area.

Example II

Three aqueous emulsions were prepared each containing 0.8% polyamide, 1.2% polyepoxide, and 2% emulsifying agent (Tween-40). In each case the polyamide was the same as in Example I. Different polyepoxides were employed in the emulsions as follows: Emulsion A—a commercial product consisting essentially of 2,2-bis(2,3-epoxypropoxyphenyl) propane. Emulsion B—a commercial preparation being the reaction product of epichlorhydrin and glycerol and containing on the average a little more than two epoxy groups per mol of glycerol. This material is a liquid at ordinary temperatures, has a viscosity of 0.9 to 1.5 poises at 25° C. and an epoxide equivalent of 140-165. Emulsion C—equal parts by weight of the two polyepoxides described above.

Three pairs of woolen gabardine trousers were each treated with one of the emulsions as follows: The trousers were immersed in the emulsion, squeezed by hand to a weight of about 200% the original weight of the garment, laid flat on towels to partially dry to about 130-150% of original weight. The trousers were then pressed on a tailor's steam press subjecting the garments to about 3 minutes contact with steam at 60-70 lbs. per sq. in. pressure. It was observed that the trousers could be laundered in conventional detergent-water formulations with retention of their creases.

Having thus described the invention, what is claimed is:

1. A method for imparting stable deformations to a textile material which comprises impregnating the textile with a polyepoxide containing at least two epoxy groups per molecule and a polyamide of a polyamine and a polycarboxylic acid, the total amount of polyepoxide and polyamide being about from 0.5 to 10% based on the weight of the textile deforming the impregnated textile in a predetermined pattern and while constraining it in such pattern subjecting it to heat to cure it and set it in the predetermined pattern.

2. A method for imparting stable deformations to a woolen textile material which comprises impregnating the textile with a polyepoxide containing at least two epoxy groups per molecule and a polyamide of a polyamine and a polycarboxylic acid, the total amount of polyepoxide and polyamide being about from 0.5 to 10% based on the weight of the textile deforming the textile in a predetermined pattern and while constraining it in such pattern subjecting it to steam to cure it and set it in the predetermined pattern.

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