

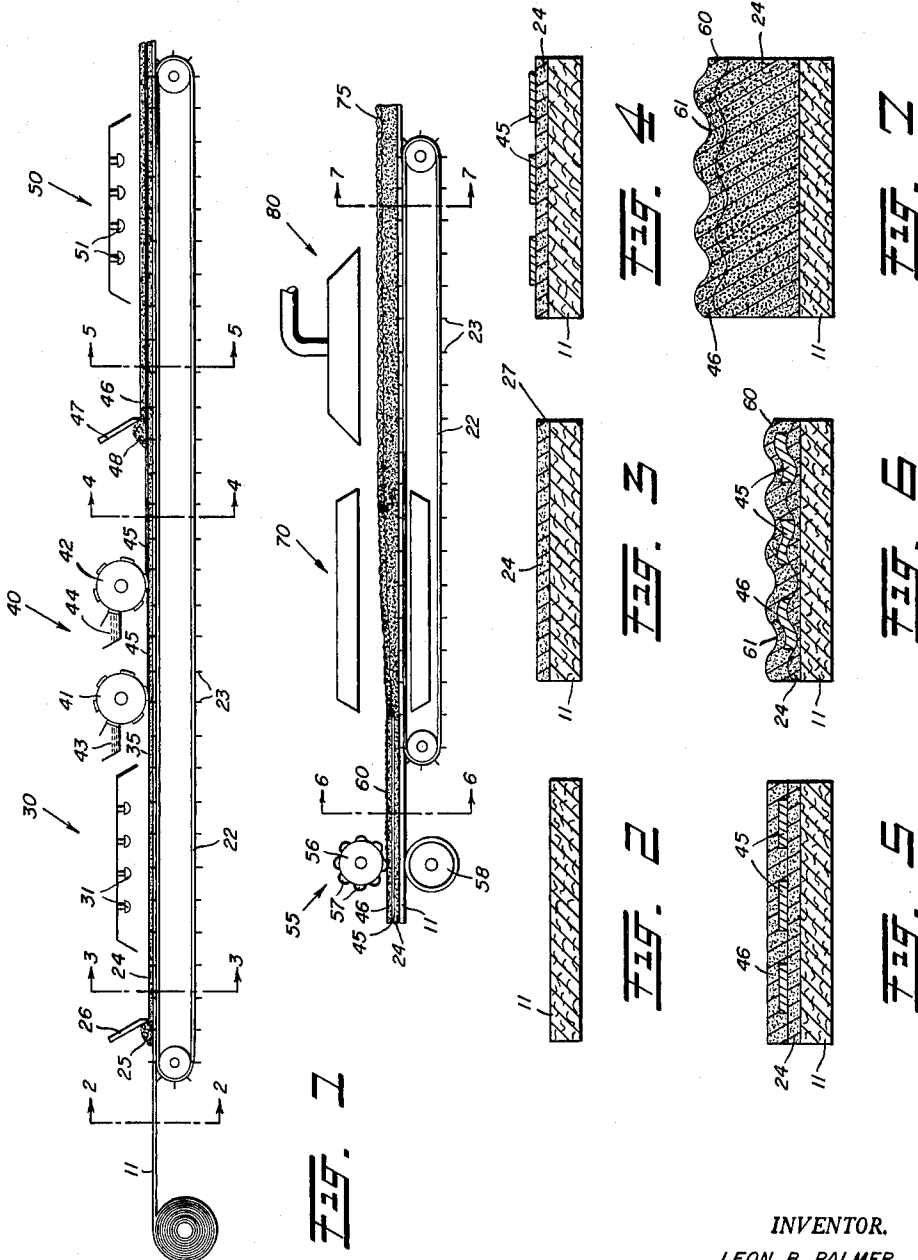
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PROCESS FOR PRODUCING DECORATIVE SURFACE COVERING

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PROCESS FOR PRODUCING DECORATIVE SURFACE COVERING

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This invention relates to flexible decorative surface coverings and particularly to a method for producing a textured foam surface covering.

Printed products adaptable as decorative and protective coverings for floors, walls and the like have been available for many years. The technique of printing an oleoresinous enamel paint decoration upon a flexible backing sheet has been used commercially for at least 40 years to produce products commonly referred to as printed felt base. Such products have the desirable features of being low in cost and they can be readily manufactured in a variety of attractive designs.

Printed felt base has a hard and smooth decorative wearing surface. Although this renders the product easily cleaned, the hard surface tends to result in excessive noise from foot traffic. Also, the hard surface can cause fatigue to those who must stand for long periods of time upon such products. The comfort and noise reduction of conventional printed felt base is somewhat better than floors of wood and stone due to the cushioning characteristics of the felt backing, but since the felt layer is very thin and on the back of the product, the improvement is only slight. Also, the thin product lacks any appreciable resistance to the flow of heat with the result that printed felt-base-covered floors tend to be cold in winter, an effect augmented by the smooth and glossy wearing surface.

Resilient floor coverings are available which are quiet and comfortable under foot by utilizing the resilient properties of a material such as rubber. Rubber floor tile is relatively quiet and comfortable under foot. The product, however, is expensive and also tends to be cool in winter due to its high thermal conductivity.

Efforts have been directed toward improving the resiliency of smooth surface floor coverings. Products with improved resiliency can be made by the application of a thin layer of foam rubber to the back of the surface covering. Although this does improve products such as printed felt base, there are certain disadvantages. The foam rubber layer is subject to deterioration and chemical attack, particularly if the product is installed upon a concrete floor. The resulting breakdown of the cell structure causes the product to lose its resiliency. Also, where products are to be adhesively bonded to a surface during installation, the adhesive can become at least partially absorbed into the foam cell structure with resultant loss of at least a portion of the resiliency.

A major source of competition for smooth surface floor coverings is from woven or tufted soft surface carpeting. Carpeting is not only soft and comfortable under foot but also has a three-dimensional textured appearance which is particularly attractive in certain areas of the home. The three-dimensional texture is due to its thick, readily deformable pile surface, an effect unobtainable in printed felt base floor coverings.

United States Patent 2,943,949, which issued to Robert K. Petry on July 5, 1960, discloses a surface covering product having the advantages of both smooth and soft surface floor coverings. In accordance with this patent, a foam product is produced having a textured or three-dimensional surface. The product is made by embossing or otherwise deforming a base such as felt in a suitable overall design. The surface of the embossed felt is coated

with a resinous composition containing a blowing agent to form a smooth layer. The coated product is thereafter subjected to heat to decompose the blowing agent and convert the resinous layer to a fused and foamed structure. Embossings in the surface of the felt are mirror-imaged in the surface of the product. This result is caused by the greater thickness of foamable composition present in the layer above a depressed area as compared with the thickness of the layer in the undepressed areas. An improvement on this product is disclosed in United States Patent 2,961,332 which issued to R. Frank Nairn on November 22, 1960. In accordance with this latter patent, a foam structure product is produced by utilizing different amounts of blowing agent in various sections of the resinous coating. After decomposition of the blowing agent, the product has a surface with an overall textured appearance caused by the embossing as well as an additional design created by the various heights of foam. A product closely resembling a sculptured carpet can be obtained in this fashion. The products of these patents make excellent surface coverings which have a high degree of resiliency underfoot yet have a hard surface which can be readily cleaned and even scrubbed. These products can be made particularly long-wearing by applying a solid composition coating to their surface. The coating has to be applied so as to prevent the loss of the textured surface of the product. One method of accomplishing this is by spraying the coating on the surface. Such an operation, however, limits the thickness of the coating which can be obtained and, therefore, the service life of the product.

It is an object of the invention to produce an improved decorative surface covering having a textured foam structure. Another object of the invention is to produce such a product characterized by high resistance to wear. A further object of the invention is to provide a process for producing such a surface covering in a wide range of decorative effects. Other objects and the advantages of the invention will appear hereinafter.

In accordance with the invention a foam structure surface covering is produced having a textured surface by coating a base with a resinous composition containing a blowing agent, heating the coating to at least partially gel the composition, printing a design on the surface of the gelled sheet, applying a second transparent resinous composition which does not contain a blowing agent over the surface of the printed design, heating the second coating to gel the composition, passing the composite sheet through an embossing apparatus whereby the surface of the sheet is embossed to a depth extending into the first or bottom coat and thereafter heating the embossed composite structure to decompose the blowing agent and foam the composition of the bottom coat.

The bottom or first coating must be in a physical state which will result in the displacing of the composition by the embossing operation. If the embossing operation merely compacts the composition or the coating is too fluid to maintain the embossing, the desired result will not be obtained. The finished product has the embossed design substantially exaggerated in its surface and covered completely by a solid wear layer formed by the second coat. This procedure permits the utilization of a wide range of printing methods to produce the printed design. In addition, it provides a simple method for producing a foam product having a substantially thick wear resistant surface layer. It is essential to the invention that the coatings be gelled sufficiently to hold the embossed design. The semi-fused coatings must show a percent of elongation of at least about 25 to 150 percent. This state is referred to as the "elastomeric point" as described in *Plastics Technology*, page 43, October 1960.

As an alternate method, the printing step can be omitted and the first, second, or both, coats can be applied by a printing of a number of contrasting colored compositions. It is preferred when utilizing this latter procedure for the printed composition to cover the entire surface of the product so that it has an overall foam or wear layer. The second coat can be omitted if high resistance to wear is unimportant. If a solid color product is desired, the printing step can be omitted and the second coating can be formed from a pigmented composition. Additional decorative effects can be created by applying a contrasting color composition to the embossed areas by a spanning technique and/or by applying small pieces of resin-coated aluminum foil to the surface of the product prior to decomposing the blowing agent.

The invention will be better understood from the following detailed description of one embodiment of the invention when read in connection with the drawings wherein

FIGURE 1 is a schematic representation of one method of producing a surface covering in accordance with the invention; and

FIGURES 2 to 7 are enlarged cross-sectional views of the product in various stages of manufacture as shown in FIGURE 1.

A base, such as felt 11, is placed on a conveyor, as for example, an endless belt 22, provided with pins 23 which project vertically from the belt at spaced points along its edges. The base 11 is engaged by the pins 23 which advance it through the various stages of the process. A first coat 24 of resinous composition 25 is applied to the upper surface of the base 11 by any suitable means such as a reverse roll coater, a doctor blade or similar coating apparatus. If a doctor blade 26 is used, a reservoir of the resinous composition 25 is maintained behind the blade allowing a uniform coating of the composition to be applied to the surface of the felt. The coated base 27 is then passed through a heating unit generally indicated at 30 which can be any conventional heating means such as a bank of infra-red heating lamps 31. The heating unit supplies sufficient heat to at least partially gel the thermoplastic resinous coating. The gelled coating 35 is then passed to a printing unit generally indicated at 40 which can be any of the conventional printing means such as a flat bed printing machine as widely used in the smooth surface flooring industry or a conventional gravure press having cylinders 41 and 42 which are etched to print a design with a suitable ink on the surface of the gelled sheet. The cylinders pick up printing ink composition from ink supplies 43 and 44 on its etched surface and applies the printing composition 45 on the surface of the gelled layer 35. The printing composition is conventionally dried in the printing press. A second coat 46 of resinous composition is then applied over the printed surface by any conventional coating means such as a doctor blade 47 which supplies the composition from a reservoir 48 held back of the doctor blade. The second coat 46 is then passed through a heating unit generally indicated at 50 which can be any type of heating unit such as a bank of infra-red heat lamps 51. The second coating 46 is heated to a temperature sufficient to at least partially gel the composition. The composite product is then passed to an embossing unit generally indicated at 55 which comprises an upper embossed roll 56 bearing a plurality of spaced protuberances 57 which are provided in the pattern to be embossed in the coatings. The back of the composite sheet is contacted by a back-up roll 58 which forces the composite product against the embossed roll. The embossed sheet 60 bearing the depressed portions 61 which extend for a substantial distance into the first coat 24 is passed to a heating unit generally indicated at 70. The heating unit can be any heating apparatus such as a hot air oven. It is preferred to have a heating unit which heats both surfaces of the sheet. The heating unit raises the temperature of the embossed composite sheet 60 suffi-

ciently high to cause the decomposition of the blowing agent contained in the first coat 24 and to completely solvate and fuse both coatings 24 and 46. The foamed and fused product 75 is then cooled by passing through any conventional cooling unit such as a forced cold air unit generally indicated at 80. The cooled product is then withdrawn from the apparatus. The product can be used in sheet form as produced or cut into tiles or other appropriate shapes for use.

As indicated above in one alternate procedure, the first coat 24 of resinous composition is applied by a printing operation such as a conventional block printing machine used in the floor covering industry. The first coat, therefore, is made up of a number of different compositions in an overall design. This procedure, therefore, can combine both the coating and the printing steps in one operation. After the printing of the first coat in the desired design which preferably covers the entire upper surface of the base, the composition is heated to at least partially gel the composition and thereafter the second or top resinous coat can be applied. If the product is going to be used in areas which are not subjected to substantial wear, such as for wall coverings, or for less expensive covering, the second coat can be omitted entirely. The second coat of resinous composition can be transparent if the first coat is in the form of a design or it also can be printed as a design in register with the design in the bottom or first coat.

Suitable backing sheets include those formed of flexible resinous compositions as well as sheets of woven fabric and impregnated felted fibers. Any of the thermoplastic or elastomer resinous compositions which can be calendered or pressed to form a flexible sheet can be used to form backing sheets for use in the invention. Typical of the resins which can be compounded with plasticizers and fillers and sheeted to form a flexible sheet are such resins as butadiene-styrene copolymers, polymerized chloroprene, polyvinyl chloride, polyvinyl acetate, vinyl chloride-vinyl acetate copolymers and the like. In some cases, scrap and degraded resinous compositions can be salvaged by forming them into sheets which can be used as backing sheets in producing products in accordance with the invention.

Suitable backing sheets also include woven fabrics formed of such fibers as cotton, wool, asbestos and various synthetic fibers. Where loosely woven fabrics such as burlap are used, the fabric can be sized to prevent passage of the coating composition through the openings between the fibers by utilizing the conventional sizing composition used in the textile industry.

Felted cellulose or mineral fibrous sheets impregnated with a water-proofing and strengthening saturant are particularly useful in accordance with the invention since they are low in cost and yet are flexible and strong. The sources of cellulose can include cotton or other rags, wood pulp, paper boxes, or mixtures thereof in any proportion. Asbestos is the most commonly used mineral fiber. In addition to the fibers, fillers such as wood flour can be used. A slurry of fibrous material in water is formed into a sheet using any of the techniques conventionally employed in the manufacture of paper. For example, sheet formulation can take place on a Fourdrinier or cylinder sheet forming machine. The fibrous sheet so prepared is then dried. In addition to cellulose and mineral fibers, other fibers can be used including synthetic fibers and those of animal origin.

Felted fibrous sheets as produced by conventional sheet forming techniques are usually unsatisfactory for use as backings for surface covering products without impregnation with a water-proofing and strengthening impregnant, due to poor strength and water resistance.

The particular impregnant chosen must not only be capable of imparting strength and water resistance to the sheet, but must also meet other requirements as to its physical and chemical behavior at high temperatures. The coating compositions applied to the backing in accordance

with the invention must be heated to temperatures as high as 300° F. to 400° F. in order to fuse the resin and/or expand the composition into a foam. Thus, the impregnant chosen must be stable at these temperatures. The impregnant should be substantially free of any components which are volatile at these temperatures and it also must not soften to such an extent as to exude from the sheet. In addition, the impregnant should not be subject to appreciable detrimental chemical changes such as oxidation.

Suitable impregnants include vinyl resins, such as polymers of vinyl chloride and vinyl acetate. Particularly suitable are copolymers of vinyl acetate and vinyl chloride or these monomers copolymerized with other monomers copolymerizable therewith. In addition, polymerized acrylic and methacrylic acids and their polymerized derivatives polyethylene, polystyrene, butadiene-styrene copolymer, butadiene-acrylonitrile copolymer, natural rubber, polymerized chloroprene and the like are suitable. Thermosetting resins which under the influence of heat cure by polymerizing and cross-linking with the cellulose can also be used as impregnants. Such resins as phenolic resins, polyesters, oleoresins such as drying oils and the like, isocyanates and polyurethanes and the like are also useful.

These resins can be incorporated into the felted fibrous sheet by impregnation of the sheet with an emulsion or solution of the resin followed by drying of the sheet to remove the solvent. Alternately, the resin can be added in fine particles to the fiber furnish prior to sheet formation either as solid particles of resin or as an emulsion in water or other emulsifying vehicle.

It is sometimes desirable and particularly when the base is a felt sheet to apply a size coat prior to the application of the first coating. The size coat serves as a barrier coat to prevent migration of the felt impregnant into the coat. In addition, the size coat serves to provide good adhesion between the base sheet and the first coat. The size coat is preferably applied as an aqueous emulsion of a suitable resin.

In accordance with the invention, a coating of formable composition is applied to base and in the preferred embodiment, subsequently a second coat of resinous composition is applied. The resinous binder must be one that is coalesced or fused into a continuous film by the application of heat. The dispersion medium can be water in the case of an aqueous latex, or an organic solvent, but is preferably a fluid plasticizer for the resin used. Such a dispersion of resin in a plasticizer is conventionally termed a plastisol. A plastisol has appreciable fluidity at normal room temperature but is converted by heat into a flexible, tough thermoplastic mass. This ultimate result is brought about by the process of fusion wherein the resin becomes plasticized and completely solvated by the plasticizer. Plastisols are preferred since it is unnecessary to remove the carrier as is necessary with water and organic solvent carriers.

The preferred and most widely used resins for surface coverings are polymers of vinyl chloride. The vinyl chloride polymers can either be simple, unmixed homopolymers of vinyl chloride or copolymers, terpolymers or the like thereof in which the essential polymeric structure of polyvinyl chloride is interspersed at intervals with the residues of other ethylenically unsaturated compounds polymerized therewith. The essential properties of the polymeric structure of polyvinyl chloride will be retained if not more than 40 per cent of the extraneous co-monomer is copolymerized therein. Suitable extraneous co-monomers include, for instance, vinyl esters on the order of vinyl bromide, vinyl fluoride, vinyl acetate, vinyl chloroacetate, vinyl butyrate, other fatty acid vinyl esters, vinyl alkyl sulfonates, trichloroethylene and the like; vinyl ethers such as vinyl ethyl ether, vinyl isopropyl ether, vinyl chloroethyl ether and the like; cyclic unsaturated compounds such as styrene, the mono- and polychlorostyrenes,

coumarone, indene, vinyl naphthalenes, vinyl pyridines, vinyl pyrrole and the like; acrylic acid and its derivatives such as ethyl acrylate, methyl methacrylate, ethyl methacrylate, ethyl chloroacrylate, acrylonitrile, methacrylonitrile, diethyl maleate, diethyl fumarate and the like; vinylidene compounds on the order of vinylidene chloride, vinylidene bromide, vinylidene fluorochloride and the like; unsaturated hydrocarbons such as ethylene, propylene, isobutene and the like; allyl compounds such as allyl acetate, allyl chloride, allyl ethyl ether and the like; and conjugated and cross-conjugated ethylenetically unsaturated compounds such as butadiene, isoprene, chloroprene, 2,3-dimethylbutadiene-1,3-piperylene, divinyl ketone and the like. As a rule, the criterion of a practical co-monomer for use with vinyl chloride to produce copolymers containing 60 percent or more vinyl chloride is that, on a mol percentage basis, an initial charge of 96 percent vinyl chloride, balance comonomer, shall yield on initial copolymer containing (a) at least 90 percent vinyl chloride, and (b) not more than 99 percent vinyl chloride. On this basis, satisfactory comonomers for use with vinyl chloride will be those having Q_2 and e_2 values, as described in J. Polymer Science 2:101, correlated as follows, assuming for vinyl chloride Q vinyl chloride=0.03 and e vinyl chloride=0.3:

$$4.1 < \frac{.029e^{-.3(3-e_2)} + .04}{Q_2} > .37$$

$$1.33Q_2e^{e_2(3-e_2)} + .96$$

Instead of the single unsaturated co-monomers of the types above indicated, mixtures of such co-monomers may enter into the copolymers, it being understood that the total quantity thereof shall be small enough that the essential character of the polyvinyl chloride chain is retained. Although such vinyl chloride resins are preferred, as is apparent, the coating composition can be formed from any resin which can be gelled, embossed and foamed and the invention is not intended to be limited to any particular resin or group since many other types and groups of resins will occur to those skilled in the art.

Resins adaptable for use in formulating vinyl plastisols are commonly referred to as dispersion grade resins. Such resins are available having particle sizes of from 0.02 to about 2 microns in contrast to calendar grade vinyl resins which are available in particles ranging up to 35 microns in size. Dispersion grade resins are usually of higher molecular weight than calendar grade resins and have particle surfaces of a hard, horny nature.

Polymers of vinyl chloride having specific viscosities above about 0.17 and preferably between 0.17 and 0.31 as measured in a solution of 0.2 gram of resin in 100 milliliters of nitrobenzene at 20° C. are particularly effective. In the determination of specific viscosities, the sample of resin in nitrobenzene solution maintained at a temperature of 20° C. is allowed to flow between two calibrated marks in a pipette and time required is recorded. This time is compared with the time required for a control of pure nitrobenzene solvent to pass between the same two marks, also at a temperature of 20° C. The specific viscosity is determined as the sample flow time divided by the control flow time, minus 1. The specific viscosity is an effective measure of relative molecular weight of the polymer, the higher the specific viscosity being the molecular weight. The intrinsic viscosity is another method for determining molecular weight. Resins are preferred which have an intrinsic viscosity of from about 0.75 to about 1.3. The intrinsic viscosity is obtained from viscosity measurements, at 30° C., of cyclohexanone solution of the resin and of cyclohexanone solvent. The intrinsic viscosity $[\eta]$ is defined by the equation

$$[\eta] = C \lim_{C \rightarrow 0} \left(\frac{\ln \eta_{rel}}{C} \right)$$

when η_{rel} is relative viscosity and C is the concentration

of polymer in grams per 100 cc., the concentration being such that η_{rel} has a value of from 1.15 to 1.4.

In the formulation of plastisol coating compositions for use in the invention, the fine particle size resin is uniformly dispersed in a mass of fluid plasticizer. The fluidity of plastisols is influenced in part by the particular resin selected but is also a function of the ratio of plasticizer to resin. Plastisols become less fluid as the ratio of plasticizer to resin is reduced. Plastisol coating compositions for use in the invention contain from about 50 to about 150 parts plasticizer per 100 parts resin with a range of about 60 to about 100 parts plasticizer per 100 parts resin being particularly effective. The viscosity of plastisol compositions can also be reduced by the addition of small amounts of a volatile diluent not exceeding about 100 parts per 100 parts resin. Useful diluents include benzene, toluene, methyl ethyl ketone, petroleum solvents such as V. M. and P. naphtha (Boiling Range of 190–275° F.) and the like. If the compositions are to be applied by a printing step, it is usually necessary to reduce their viscosity. Suitable printing compositions have a viscosity of 25° C. of from about 200 to about 25,000 centipoises as measured with a Brookfield viscometer using a No. 6 spindle at 10 r.p.m. For printing by the flat bed technique, a viscosity range of about 500 to about 5,000 centipoises is desirable with a range of about 1,000 to about 3,500 centipoises being particularly effective.

The selection of the plasticizer is important in determining the strength and flexibility of the coating and also in influencing the viscosity and viscosity stability of the printing fluid and the foaming characteristics of the composition. Esters of straight and branched chain alcohols with aliphatic acids impart low viscosity and good viscosity stability. Typical plasticizers of this type include dibutyl sebacate, dioctyl sebacate, dioctyl adipate, didecyl adipate, dioctyl azelate, triethylene glycol di (2-ethylhexanoate), diethylene glycol dipelargonate, triethylene glycol dicaprylate and the like. Plasticizers of the aromatic type, such as esters of aliphatic alcohols and aromatic acids or aromatic alcohols and aliphatic acids or aromatic alcohols and aromatic acids are desirable in that they impart good foaming characteristics to a plastisol, although the use of highly aromatic plasticizers is limited by their tendency to yield plastisols of high viscosity. Typical plasticizers of this type include dibutyl phthalate, dicapryl phthalate, dioctyl phthalate, dibutoxy ethyl phthalate, dipropylene glycol dibenzoate, butyl benzyl sebacate, butyl benzyl phthalate, dibenzyl sebacate, dibenzyl phthalate and the like. Other types of plasticizers, such as esters of inorganic acids, including tricresyl phosphate, octyl diphenyl phosphate and the like, alkyd derivatives of rosin, chlorinated paraffine, high molecular weight hydrocarbon condensates and the like can also be used. The plasticizers or blend of plasticizers is chosen to yield a composition of the desired viscosity and/or foaming characteristics. In addition, the plasticizer should preferably have a low vapor pressure at the temperatures required to fuse the resin. A vapor pressure of two millimeters of mercury or less at 400° F. is satisfactory.

Minor amounts of stabilizers are usually incorporated in the coating compositions to reduce the effects of degradation by light and heat. Suitable light stabilizers include resorcinol disalicylate, resorcinol dibenzoate, phenyl phthalate, phenyl benzoate, o-tolyl benzoate, eugenol, guaiacol, o-nitrophenol, o-nitraniline, triethylene glycol salicylate, and organic phosphates and other complexes of such metals as barium, cadmium, strontium, lead, tin and the like. Suitable heat stabilizers include sulfides and sulfites of aluminum, silver, calcium, cadmium, magnesium, cerium, sodium, strontium and the like, glycerine, leucine, alanine, o- and p- amino benzoic and sulfanilic acids, hexamethylene tetramine, weak acid radicals including oleates, recinoates, abietates, salicylates and the like. Normally, the compositions contain about 0.5 to about 5 parts stabilizer per 100 parts resin.

The coating compositions can contain pigments in accordance with the particular color desired. Where a multi-colored decorative effect is created in accordance with the invention by printing, separate batches of printing composition for each of the colors desired are needed. Any of the organic and inorganic pigments well known in the art for pigmenting compositions can be used. Normally, from about 0.5 to about 5 parts pigments per 100 parts resin are used.

The foamable compositions contain, in addition, an effective amount of blowing agent. The larger the amount of blowing agent within practical limits used the greater is the expansion of the foam. Foam densities of from 10 percent to 50 percent of the density of the unblown composition can be readily attained. Such results are attainable with from about 1 to about 20 parts blowing agent per 100 parts resin with from 2 to 10 parts blowing agent per 100 parts resin being particularly effective for the production of foams of a density which are most desirable for use in producing surface coverings in accordance with the invention.

Complex organic compounds which when heated decompose to yield an inert gas and have residues which are compatible with the resin used in the compositions are preferred as blowing agents. Such materials have the property of decomposition over a narrow temperature range which is particularly desirable for obtaining a good foam structure. Compounds having the $>N-N<$ and $-N=N-$ linkages decompose at elevated temperatures to yield an inert gas high in nitrogen. Typical compounds include substituted nitroso compounds, substituted hydrazides, substituted azo compounds and the like, such as are tabulated below:

		Decomposition temperature, ° F.
Blowing agent:		
Dinitrosopentamethylenetetramine	-----	355-375
Azodicarbonamide	-----	370-390

Blowing agents for use in the invention must be decomposed an effective amount at a temperature below the decomposition temperature of the resin used but above the elastomeric point of the resin composition. Therefore, in the case of compositions formulated with the preferred vinyl chloride polymers, a blowing agent decomposing between about 300 and about 450° F. should be used. The minimum initial decomposition temperature must be sufficiently high that no premature gas evolution occurs during mixing of the composition, coating operation, and particularly the gelling and embossing step. In the event the coating is to be fused before embossing, then it is necessary to use a blowing agent which decomposes above the fusion temperature of the resin.

When the technique of block printing is used to produce a decorative design which will also serve as the wear layer, a film of decorative composition of appreciable thickness is applied to the backing material. Printed films of 3 to 10 mils in thickness can be applied by block printing. When a film of this thickness is expanded and foamed by decomposition of the blowing agent in the composition, a decorative expanded foamed layer having an average thickness of 10 to 100 mils is produced. This is of sufficient thickness to provide satisfactory resilience and cushion effects when the product is installed as a floor covering. If the product is to be used as a wall covering, lower thicknesses of foam can be used.

After the first coating is applied, the coating is heated to gel the composition. In this specification and claims, the term "gel" includes both the partial (at least the elastomeric point) and complete solvation of the resin or resins with the plasticizer. The heating is limited as to the time and temperature to prevent the decomposition of the blowing agent in the composition. When using the preferred polyvinyl chloride composition, the temperature of the composition is preferably raised to about

240° F. to about 275° F. Generally, the oven temperature would be slightly higher temperature to have the coating reach the desired temperature. It is preferred to avoid fusion of the resin by complete solvation with plasticizer since this simplifies the embossing step as the coating will be softer. After gelling the first coat, the product is cooled in the event it is desired to print a design on the surface of the gelled coating. The design can be printed by any of the conventional printing methods, with the rotogravure printing technique being particularly suitable. The printing composition can be one of the conventional printing inks or similar compositions which will adhere to the gelled coating.

After the printing step, the second coating is applied. The second coating, as indicated above, can be the same or different composition from the first coating. If different compositions are used which are not readily compatible, an adhesive layer can be applied before the second coating. In the event that a decoration has been printed on the surface of the first coating, it is necessary for the second coating to be of transparent or translucent composition so that the printed design can be visible through the second coating. This second coating primarily serves as a wear layer and, therefore, its thickness will depend entirely on how much wear is desired in the final vinyl product. As a general rule, a coating of from about 2 to about 12 mils is sufficient to give the product good wearing qualities. The second coat is then gelled in a manner similar to the manner described above for the first coat. The heat supplied in the gelling operation should be sufficient to soften both coats so that they can be readily deformed when they pass through the embossing mechanism. As an alternate procedure, however, the coated product can be allowed to cool and subsequent reheated prior to embossing.

The embossing is carried out by utilizing the usual apparatus which involves an engraved embossing roll and rubber-covered back roll. The embossing roll can be cold or heated depending on the condition desired. If the embossing roll is heated, it should be to a temperature less than the fusion temperature of the resin since at such temperature, special precautions have to be taken to prevent the composition from sticking to the embossing roll. The embossing must be of sufficient depth to extend into the first or base coat and the first coat must be sufficiently softened so as to cause displacing of the coating rather than mere consolidation. Displacing is essential in order to result in the product having an embossed appearance after the foaming operation. A particularly decorative effect can be obtained by at least partially filling the embossing with a composition of contrasting coloration.

After the application of the coating composition, the coat must be heated to a temperature sufficient to fuse the resin by completely solvating the resin with plasticizer and to decompose the blowing agent. The temperature of the entire mass of composition upon the backing must attain the fusion temperature of the resin in order that a product of satisfactory strength is to be attained. Using the preferred vinyl resin, fusion is attained at a temperature of about 300° to about 375° F. In addition, the entire mass of foamable composition must be heated to a point where the blowing agent is decomposed. When the preferred high temperature blowing agent is used, foaming does not occur until the resinous composition has been completely fused.

If volatile components are used in the top coat, care must be taken that they are essentially completely removed from the film prior to fusion. This can be accomplished by heating the composition at a temperature substantially below the fusion temperature and minimum decomposition temperature of the blowing agent for sufficient time to remove the volatile material. For example, if 5 percent of V. M. & P. Naphtha (boiling range 190-275° F.) is used, heating at 200° F. for 5 minutes

will remove sufficient material so that fusion and blowing at 400° F. can be accomplished with good cell structure and freedom from blisters.

Heating in order to effect fusion and foaming can be brought about in a forced hot air oven or other types of heating can be used. For example, the product can be passed beneath radiant heating elements; alternately, dielectric heating can be used.

The foamed and fused product after leaving the heating oven is permitted to cool. Cooling is particularly important since any premature handling of the product immediately after foaming might cause partial collapse and distortion of the foam structure. Cooling can be brought about by mere exposure of the product to the atmosphere; thus, the speed of motion of the backing along the processing apparatus and the spacing between the fusion oven and the end of the apparatus can be adjusted so that product is given sufficient time to cool. Alternately, cooling can be accelerated by blowing jets of cooled air upon the fused and foamed composition or by means of fine sprays of water upon the fused and foamed composition or by utilizing cooling rolls.

After being cooled, the product is withdrawn from the processing apparatus. It can be used in the form of a sheet as produced or can be cut into tiles or other appropriate shapes depending on the particular use to which the product is to be put. Products produced in accordance with the invention have the characteristics of excellent resilience under foot in view of the foamed layer which is at or extremely near the surface of the product. They are also characterized by having a marked three-dimensional textured appearance conforming to the embossed design. Still further, the products of the invention have good heat insulating properties by virtue of the layer of foamed composition and thus are warmer in winter and cooler in summer than conventional smooth surface coverings.

Table 1 gives the preferred temperature and time relationship using the preferred resin:

TABLE 1

Film Condition ¹	Resin Temperature (° F.)	Oven Temperature (° F.) Circulating Air	Exposure Time (seconds)
Elastomeric Point.....	240-300	250-400	10-300
Embossable.....	250-325	300-400	15-120
Fused.....	340-400	350-450	60-240
Blown.....	340-400	350-450	60-240

¹ 0.014 inch plastisol on .025 inch cellulosic felt base impregnated with 9 percent vinyl acetate and 30 percent petroleum hydrocarbon supported on a wire screen.

The time required to reach the elastomeric point will depend in part on the film thickness and particular base as shown in Table 2:

TABLE 2

Base	Film Thickness (inch)	Time/Temperature (seconds/° F.)
10M ¹	0.008	45/300
10M.....	0.014	60/300
10M.....	0.014	72/310
RX ²	0.014	90/300
RX.....	0.050	240/300

¹ 10M is a cellulosic felt of 0.025 inch thickness impregnated with 9 percent vinyl acetate homopolymer and 30 percent petroleum hydrocarbon resin.

² RX is a cellulosic felt of 0.043 inch thickness containing 5 percent of a cured ureaformaldehyde resin and 25 percent of synthetic rubber.

The following examples are given for purposes of illustration:

Examples I and VI are typical foamable compositions.

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Example I

The following ingredients in the proportions indicated were ground on a three-roll mill:

	Parts
Polyvinyl chloride (dispersion grade)	100
Petroleum hydrocarbon condensate ¹	18
Butyl benzyl phthalate	52
Finely divided filler	3
Stabilizers	4
Azodicarbonamide blowing agent	3.5

¹ Conoco 300—Continental Oil Co. Ponca City, Okla. The plastisol had a viscosity of 4,000 centipoises as measured with a Brookfield viscometer using a No. 6 spindle at 10 r.p.m.

Example II

The following ingredients were ground on a three-roll mill:

	Parts
Polyvinyl chloride (dispersion grade)	100
Petroleum hydrocarbon condensate	18
Butyl benzyl phthalate	52
Finely divided filler	3
Stabilizers	4
Azodicarbonamide blowing agent	1
V. M. and P. naphtha, boiling range 190 to 275° F. ..	5

The plastisol had a viscosity of 2,000 centipoises as measured with a Brookfield viscometer using a No. 6 spindle at 10 r.p.m. It was suitable for printing by the flat bed method.

Example III

	Parts
Polyvinyl chloride (particle size averaging less than 5 microns)	717.50
Tricresyl phosphate	179.41
Paraplex G-62 (epoxidized soya bean oil)	358.75
MPS-500 (chlorinated fatty acid ester)	179.41
Thermalite (thio-organo-tin compound)	8.4
BL-425 (sodium alkyl sulfonate in DOP)	45.15
Azodicarbonamide (70% in mineral oil)	156.15

Example IV

	Parts
Polyvinyl chloride	100
Aromatic hydrocarbon resin	38
Polyester type plasticizer	38
Tricresyl phosphate	24
Tribasic lead sulfate	2.5
Azodicarbonamide	15
Sodium alkyl sulfonate in DOP	7

Example V

	Parts
Polyvinyl chloride	100
Diocetyl phthalate	30
Polyester type plasticizer	45
Dibasic lead sulfate	2
Azodicarbonamide	13
Sodium alkyl sulfonate in DOP	7
Mica	20

Example VI

	Parts
Polyvinyl chloride (dispersion grade)	100
Butyl benzyl phthalate	60
Alkyl aryl hydrocarbon	5
Dibasic lead phosphite	1
Azodicarbonamide	4
Titanium dioxide	2

Examples VII to IX are typical wear layer compositions.

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Example VII

	Parts
Polyvinyl chloride (dispersion grade)	100
Butyl benzyl phthalate	69
Alkyl aryl hydrocarbon	7
Barium-zinc complex	5.6
Titanium dioxide (58% finely divided TiO ₂ in dioctyl phthalate)	4.9
Hydrocarbon diluent	7.0

Stormer viscosity is 55 seconds per 100 rev. at 77° F. under 200 gm. load. Suitable for application using the flat bed printing technique.

Example VIII

	Parts
Polyvinyl chloride (dispersion grade)	100
Diocetyl phthalate	15
Tricresyl phosphate	15
Petroleum mineral spirits	20
Methylethyl ketone	2
Stabilizer	5

Example IX

	Parts
Polyvinyl chloride (dispersion grade)	100
Diocetyl phthalate	10
Tricresyl phosphate	7
Epoxidized soya bean oil	8.5
Barium-Cadmium (stabilizer)	3
Mineral spirits	21.5
Methyl ethyl ketone	2

Example X

35	A size coat is prepared having the following formulation:	
		Parts
	Polyvinyl chloride latex (preplasticized)	53
	Carboxy vinyl polymer (thickener 2% in water) ..	35
40	Water	12
	Ammonia (raise pH to 7-8).	

A foamable plastisol is formulated by grinding the following ingredients on a conventional Cowles mixer:

	Parts
Vinyl chloride-vinyl acetate copolymer (dispersion grade)	100
Diocetyl phthalate	60
Alkyl aryl hydrocarbon	5
Dibasic lead phosphite	1
Finley divided titanium dioxide	2
Azodicarbonamide	4

The plastisol has a viscosity of 2,500 centipoises at 25° C. as measured with a Brookfield viscometer using a No. 6 spindle at 10 r.p.m. The plastisol is applied as a uniform coating of 0.014 inch on the surface of a 0.025 inch thick cellulose felt sheet impregnated with 9 percent vinyl acetate, 30 percent hydrocarbon resin. The felt sheet had previously been coated with the size coating at the rate of 0.025 pound per square inch and dried. The plastisol coating is then heated to a temperature of 310° F. for a period of 72 seconds to obtain a composition temperature of 250° F. thereby gelling the coating into a film having an elongation of 100 percent. The gelled coating is heated to about 325° F. and then embossed to a depth of 0.012 inch in a textured overall design utilizing approximately 500 pounds per lineal inch of pressure. The embossing roll was heated to a temperature of 150° F. The embossed product was then heated for 2¼ minutes at 400° F. to fuse the composition and completely decompose the blowing agent to produce a product having a foamed structure of about 0.060 inch in thickness. The surface of the product reproduced the embossed design with excellent fidelity.

Example XI

The procedure described in Example X is followed except that after the application and gelling of the first coat, a design is printed on the gelled surface utilizing a roto-gravure press and vinyl printing ink of various colors having the following formulation:

	Percent
Polyvinyl chloride -----	12.2
Pigments -----	11.1
Tricresyl phosphate -----	15.7
Methyl ethyl ketone -----	61.0

The ink is dried and then the printed gelled coating is coated to a thickness of 0.004 inch with the following composition:

	Parts
Polyvinyl chloride (dispersion grade) -----	100
Dioctyl phthalate -----	15
Tricresyl phosphate -----	15
Petroleum mineral spirits -----	20
Methylethyl ketone -----	2
Stabilizers -----	5

The composite structure is then heated for two minutes at 300° F. to remove the solvent and gel the coating into a film. The film reached a temperature of about 250° F. The gelled coating is then embossed as described in Example X, with the embossing extending to a maximum depth of 0.012 inch. The embossed coatings are heated to 400° F. for 2½ minutes to decompose the blowing agent and fuse the compositions. The product is identical to that of Example X except that it has a clear solid wear layer of about 0.004 inch formed by the second coat and a decorative design visible through the wear layer.

Example XII

The foamable plastisol is formulated by mixing the following ingredients on a conventional Cowles mixer:

	Parts
Vinyl chloride (dispersion grade) -----	100
Dioctyl phthalate -----	30
Dipropylene glycol dibenzoate -----	30
Stabilizer (dibasic lead phosphite) -----	1
Finely divided titanium dioxide -----	2.5
Azodicarbonamide blowing agent -----	2.5

The plastisol has a viscosity of 16,800 centipoises, measured with a Brookfield viscometer using a No. 6 spindle at 10 r.p.m. The plastisol is applied as a uniform coating of 0.014 inch on the surface of a felt sheet approximately 0.030 inch thick, formed of asbestos fibers impregnated with about 20 percent of neoprene rubber. The coating is then heated to a temperature of 250° F. by subjecting the coating to 60 seconds of a 300° F. oven to gel the coating into a film. A second plastisol formulation is prepared having the following composition:

	Parts
Polyvinyl chloride (dispersion grade) -----	100
Petroleum hydrocarbon condensate -----	18
Butyl benzyl phthalate -----	52
Pigment -----	3
Stabilizers -----	4
Azodicarbonamide blowing agent -----	3.5

The second plastisol had a viscosity of 4,000 centipoises as measured with a Brookfield viscometer using a No. 6 spindle at 10 r.p.m. The composition was separated into three batches which were each pigmented to contrasting colors. The composition was then printed on the surface of the gelled first coating to form a uniform layer having a thickness of 0.010 inch in a three-color decoration. The composite structure was then heated to a temperature of about 250° F. by subjecting it for a period of 60 seconds to an oven at a temperature of 310° F. to gel the printed coating into a film. The product is then further heated to a temperature of about 325° F. and immediately em-

bossed in a textured overall design, utilizing 500 pounds per lineal inch of pressure. The embossing roll was maintained at a temperature of 150° F. The embossed design extended to a maximum depth of 0.017 inch. The embossed product was then heated for 2½ minutes at 400° F. to completely decompose the blowing agent and fuse the composition to produce a product having a foamed structure. The surface of the product reproduced the embossed design with excellent fidelity and the product had a wear layer of 0.010 inch formed by the printed three-color design.

Any departure from the foregoing description which conforms to the present invention is intended to be included within the scope of the claims.

15 What is claimed is:

1. A process for producing a decorative surface covering having a cellular foam layer and a textured surface which comprises applying on one surface of a base sheet a coating of a liquid foamable thermoplastic resinous composition containing a blowing agent, gelling said coating by heating to at least the elastomeric point of said resinous composition without decomposing said blowing agent, applying a second coat of a liquid thermoplastic resinous composition to the surface of said gelled coating, gelling said second coat by heating said second coat to at least the elastomeric point of said second coating composition without decomposing said blowing agent, embossing a design into the exposed surface of the second gelled coating to a depth greater than the thickness of said second coating and heating the embossed coatings to completely fuse the compositions and decompose the blowing agent causing the foamable composition to form a cellular structure containing an embossed design on its surface.

2. The process of claim 1 wherein both the first and second coating compositions are plastisols of a vinyl chloride polymer.

3. The process of claim 1 wherein the first coat is a plastisol of a vinyl chloride polymer and the second coat is an organosol of a vinyl chloride polymer.

4. The process of claim 1 wherein a decorative design is printed on the surface of said first coat after heating to its elastomeric point and said second coat is at least partially transparent so that said decorative design is visible from the surface of the product.

5. The process of claim 1 wherein said base sheet is felted fibrous sheet of cellulosic fibers.

6. The process of claim 1 wherein said base sheet is felted fibrous sheet of asbestos fibers.

7. The process of claim 1 wherein said second coating is applied by printing a number of resinous compositions of contrasting coloration.

8. The process of claim 1 wherein each of said thermoplastic resinous compositions contain polymers of vinyl chloride.

9. The process of claim 8 wherein both the first and second coatings are heated to a temperature of about 240° F. to about 300° F. to gel said coatings.

10. The process of claim 8 wherein the coatings are heated to a temperature of about 250° F. to about 325° F. at the time of embossing said coatings.

11. The process of claim 8 wherein the coatings are fused and the blowing agent decomposed by heating to a temperature of about 340° F. to about 400° F.

12. A process for producing a decorative surface covering having a cellular foam layer and a textured surface which comprises applying on one surface of a base sheet a coating of a plastisol of vinyl chloride polymer containing a blowing agent which is stable at the gelling temperature of the plastisol, gelling said coating by heating to a temperature of about 240° F. to about 300° F. without decomposing the blowing agent, applying a second coating of a plastisol of vinyl chloride polymer to the surface of said gelled coating, gelling said second coating by heating said second coating to a temperature of about 240° F. to about 300° F. and raising the temperature of said

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coatings to about 250° F. to about 325° F. without decomposing the blowing agent, embossing the heated coated base to a depth greater than the thickness of said second coating and heating the embossed coatings to a temperature of about 340° F. to about 400° F. to completely fuse the compositions and decompose the blowing agent thereby forming a cellular structure containing an embossed design on its surface.

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