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R. K. PETRY  
RESILIENT DECORATIVE SURFACE COVERING  
AND METHOD OF MAKING SAME  
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3,239,365

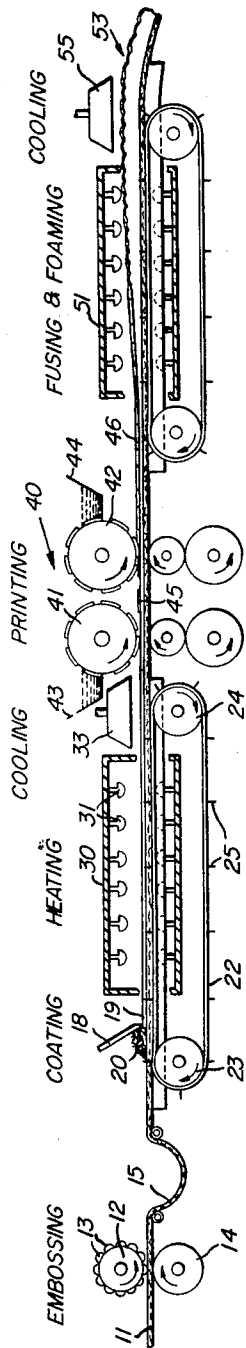


FIG. 1

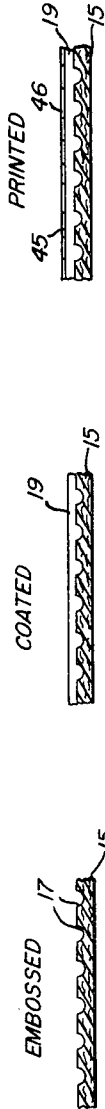


FIG. 2

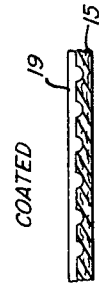


FIG. 3

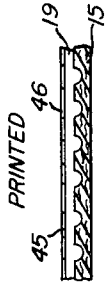


FIG. 4

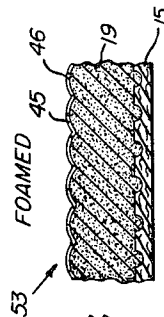
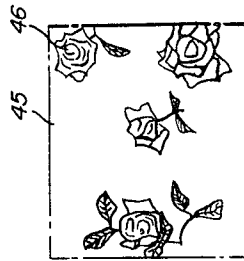
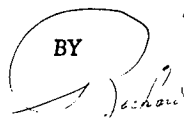


FIG. 5

FIG. 6

INVENTOR.  
ROBERT K. PETRY

BY  ATTORNEY

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## RESILIENT DECORATIVE SURFACE COVERING AND METHOD OF MAKING SAME

Robert K. Petry, Mountain Lakes, N.J., assignor to  
Congoleum-Nairn Inc., Kearny, N.J., a corporation of  
New York

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16 Claims. (Cl. 117—111)

This invention relates to flexible plastic surface coverings which have a decorative surface and particularly to such products having a resilient foam layer.

This application is a continuation-in-part of my co-pending application Serial No. 168,477, filed January 24, 1962, now abandoned, which, in turn, is a continuation-in-part of application Serial No. 841,409, filed September 21, 1959, and now abandoned.

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

Printed felt base has a hard, smooth decorative wearing surface. Although this renders the product easily cleaned, the hard surface tends to result in excessive noise from foot traffic. In addition, the hard surface can cause fatigue to those who must stand for long periods of time upon such products. The comfort and quietness 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, an effect augmented by the smooth and glossy wearing surface.

Rubber floor coverings such as one-eighth inch thick tile are available which are quiet and comfortable underfoot. Rubber tile, however, is expensive and tends to be cool due to its thermal conductivity.

Efforts have been directed toward improving the resilience of smooth surface floor coverings of which printed felt base is an example. Products with improved resilience can be made by the application of a thin layer of foam rubber to the back of the surface covering. Although this does improve the properties of products such as printed felt base, there are certain disadvantages. Foam rubber is subject to deterioration and chemical attack, particularly if it is installed upon a concrete floor. The resulting breakdown of the cell structure causes the product to lose its resilience. 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 some of the effect of resilience.

A major source of competition for smooth surface floor coverings, such as printed felt base, is from woven or tufted soft surface carpeting. Carpeting is not only soft and comfortable underfoot but also has a three-dimensional textured appearance which is particularly attractive in certain areas in the home. Soft surface carpeting, although highly attractive, has a serious disadvantage in that it readily soils and once soiled it is difficult to clean.

In my United States Patent 2,943,949, issued July 5, 1960, there is disclosed a product having a textured surface and the resiliency of soft surface covering such as

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tufted carpets while still retaining the unitary readily cleanable surface of printed felt base. This product is produced by coating or printing a foamable thermoplastic resinous composition on a textured backing such as an embossed flooring felt and thereafter heating the foamable layer to fuse the resin and foam the thermoplastic composition. The result of this procedure is a surface covering having the three-dimensional appearance caused by the embossing being reproduced in reverse in the surface and a very resilient nature caused by the foam to simulate carpet while having the ease of cleaning attribute of printed felt base. This product can be coated with a resinous material such as by spray coating to increase the service life of the product. The textured surface, however, makes it difficult to apply a relatively thick coating.

One of the most sought after properties of floor covering is good indent recovery. If a floor covering has poor indent recovery, it quickly becomes covered with small concave impressions from heels and the like which causes rapid deterioration in appearance. A resinous foam layer should have the best recovery because of its resilient nature.

An object of the invention is to produce a printed decorative surface covering characterized by excellent indent recovery and comfort underfoot while having increased wear resistance. Another object of the invention is to provide a process for producing such a product in a simple and economical manner. A further object of the invention is to produce a surface covering with a resilient wearing surface of substantial thickness and which is relatively low in cost. Other objects and the advantages of the invention will appear hereinafter.

In accordance with the invention, a surface covering having a solid wear layer of substantial thickness, a cellular foam interlayer and a flexible backing is produced by coating a flexible backing with a thermoplastic composition containing a blowing agent, applying a wear layer second coating of thermoplastic resinous composition of substantial thickness over the first coating thereby completely covering the foamable coating and heating to fuse the resins and foam the coating to form a foamed structure having a solid, unfoamed surface layer of substantial thickness. It is essential to this invention that the thickness and compositions of the foam layer and wear layer come within defined limits to give the product an acceptable combination of indent recovery and cushioning. It has been discovered that the thickness of the foam layer must be from about 0.020 inch to about 0.100 inch with a preferred range of about 0.030 inch to about 0.060 inch. The solid wear layer must be at least 0.003 inch to about 0.020 inch and preferably about 0.004 inch to about 0.006 inch in thickness.

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

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

FIGURE 2 is an enlarged cross-sectional view of a textured flexible backing for use in preparing products of the invention;

FIGURE 3 is an enlarged cross-sectional view of the backing of FIGURE 2 with a coating composition applied thereto;

FIGURE 4 is an enlarged cross-sectional view of the coated backing of FIGURE 3 having a printed design on its surface;

FIGURE 5 is an enlarged cross-sectional view of a

three-dimensional printed surface covering produced by the method of FIGURE 1; and

FIGURE 6 is a portion of the surface of the surface covering shown in FIGURE 5.

With reference to FIGURE 1, a backing sheet 11, which can be of felted fibrous backing material and embossed in one surface by passing through an embossing unit, is passed to a coating apparatus. The embossing unit comprises an upper embossed roll 12 bearing a plurality of spaced protuberances 13 which are provided in the pattern to be embossed in the surface of the backing sheet. In the embossing unit, the felt sheet is contacted by a back-up roll 14. The coating apparatus can comprise a doctor blade 18 which allows a uniform layer of resinous composition 19 containing a blowing agent from reservoir 20 to be applied to the surface of the felt 15 as it passes beneath the blade. The coating is sufficient to fill the depressions 17, if present, and form a smooth surface. The coated felt passes to an endless belt 22 provided with pins 25 which project vertically from the belt at spaced points throughout its length. The belt 22 passes around and is driven by wheels 23, 24. The web of backing material 15 is engaged by the pins 25 which pierce the side edges of the web and advance it along the machine. The coated felt is carried by the endless belt 22 to an oven 30 provided with infrared heat lamps 31. The oven supplies sufficient heat to the resinous composition to gel or partially gel the composition without decomposing the blowing agent contained in the composition. The gelled composition which is a solid is then passed through a cooling chamber 33. The cooled coated felt is then conveyed to an apparatus for applying an overall wear layer. The wear layer can be a transparent or opaque coating or it can be applied by printing to form a design as by using a printing apparatus generally indicated at 40. The printing or coating can be performed by any of the conventional coating or printing techniques which will allow the application of a uniform layer of substantial thickness. The printing apparatus as shown comprises two printing cylinders 41 and 42. A resinous composition which forms the printing ink is supplied to the printing cylinders 41 and 42 from reservoirs 43 and 44. As the printing cylinders revolve, portions of the printing composition are picked up by the design elements engraved or otherwise formed on the cylinders and placed on the coated felt. The printing 45 and 46 has to be sufficient to completely cover the coated felt to form a uniform coating. The printed sheet passes through an oven 51 in which the composition is fused and foamed, thereby producing a decorative foamed product 53 which, if an embossed base was used, bears a plurality of raised foamed portions which correspond in location to the depressed portions in the embossed felt backing. The product has a solid resinous surface formed by the printed coatings 45 and 46 and a resilient foam interlayer 19. The product is withdrawn from the oven and passed through a cooling chamber 55. The product can be used in sheet form as produced or can be cut up into tiles or other appropriate shapes.

The operation of the printing step is conventional and in place of the rotary method shown, a block printing machine or any other printing method can be used. As indicated alternately, a design can be printed on the surface of the gelled foamable coating such as with a rotogravure printing press and conventional printing inks and then a transparent or colored wear layer coating of substantial thickness is applied by means of reverse roll coater, doctor blade or the like coating apparatus.

If the backing is to be removed from the final product as an alternate method, the solid coating which will form the wear layer of the product can be first applied to the surface of the backing material. The wear layer coating is then heated to gel the composition, a design can be printed on the gelled coating and the foamable coating is applied to the surface of the gelled wear layer over the

design. After fusing the two coatings and foaming the composition, the product is stripped from the base and inverted for use.

The backing sheet is preferably a flexible strong material which will remain part of the product although, as indicated, backing materials can be used which are subsequently stripped from the final product, such as stripable coated release paper or the like. A paper having a coating such as disclosed in United States Patent 2,273,040 which issued on February 17, 1942, is particularly suitable as a release paper. If the backing remains, flexibility is important since the product is conventionally stored in closely wound rolls and must be capable of being rolled and unrolled without cracking or tearing. Strength is important in a backing in view of the strains to which the product is subjected when handled both during manufacture and immediately prior to installation. If the product is to be used in sheet form, rigid backings can be used, such as a metal sheet of aluminum or steel, rigid sheets of resinous material, such as rigid polyvinyl chloride sheets, and the like. Other suitable backings will readily occur to those skilled in the art.

Suitable backing sheets include those formed of flexible resinous composition 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 which can be textured for use in the invention. Such resins as butadiene-styrene copolymer, polymerized chloroprene, polyvinyl chloride, polyvinyl acetate, vinyl chloride-vinyl acetate copolymer and the like can be compounded with plasticizers and fillers and sheeted to form a flexible sheet. In some cases, scrap and degraded resinous compositions can be salvaged by forming them into sheets which can be embossed and used as backing sheets in producing products in accordance with the invention. With such solid backings, it is usually necessary to allow for release of any gases given off during processing. This can be done by having a multitude of small holes passing through the backing.

As indicated, suitable backing sheets also include woven fabrics formed of such fibers as cotton, wool and various synthetic fibers. Where loosely woven fabrics, such as burlap are used, the fabric can be sized to prevent passage of the printing composition through the openings between the fibers.

It has been found that felted cellulose or asbestos fibrous sheets impregnated with a water-resistant and strengthening saturant yield desirable backing sheets for the production of products 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. In addition, 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 asbestos, other fibers can be used including synthetic fibers and those of mineral and animal origin.

The particular impregnant or saturant chosen must not only be capable of imparting strength and water resistance to the sheet of felted fibers, but must also meet other requirements as to its physical and chemical behavior at the high processing temperatures. The coating composition applied to the backing in accordance with the invention must be heated to temperatures as high as 300 to 400° F. in order to fuse the resin and 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 saturant should not be subject to appreciable detrimental chemical changes such as oxidation.

The conventional impregnant used in the manufacture of printed felt base coverings of the prior art has been asphalt. Although asphalt is very low in cost, it is a highly thermoplastic substance and, in general, is too fluid at high temperatures for use as an impregnant for felt in the production of products in accordance with the invention. Asphalt saturated felt can only be used as a backing sheet in the invention where the level of impregnation is controlled not to exceed 115 percent by weight based on the weight of dry felt and where the surface on which the composition is applied bears a plurality of seal coats of paint comprising such binders as butadiene-styrene copolymer, vinylidene chloride copolymers and the like.

There are other saturants which can be used in place of asphalt to impart strength and water resistance to felt and which can withstand temperatures up to 400° F. for short periods of times, i.e., two to five minutes, without the necessity of providing special seal coats. Fibrous sheets impregnated with resinous materials are particularly suitable for use as backing sheets in the invention. Suitable resins include vinyl resins, such as copolymers of vinyl chloride, polymerized acrylic and methacrylic acids and their polymerized derivatives, polyethylene, polystyrene, butadiene-styrene copolymer, butadiene-acrylonitrile copolymer, natural rubber, polymerized chloroprene and the like. Thermosetting resins which under the influence of heat cure by polymerizing and cross-linking 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 suitable.

Such resins can be incorporated into a felted fibrous sheet by impregnation of the finished 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.

Some resin impregnants which produce a felted sheet with excellent physical properties are not compatible with the foamable composition to be applied. This may result in poor adhesion of the foam to the base. In such cases, it is desirable to size the surface of the impregnated felt sheet to which the foamable composition is to be applied with a thin coating of material which has good adhesion to both the felt impregnant and the foamable composition. Where a plasticized polyvinyl chloride polymer foam is used, excellent results have been obtained over a wide variety of felt impregnants using a size of acrylic polymer latex. A mixture of equal parts of a soft acrylic polymer latex and a hard acrylic polymer latex has been found particularly effective in aiding adhesion and fidelity of the textured design, without causing sticking of the sized surface to the rolls during the processing. The coating is effective in small amounts, an application of only 0.02 pound dry weight per square yard being sufficient to obtain the improvements. Other vinyl resins can also be used, depending upon the type of felt impregnant and foam applied thereon. A butadiene-acrylonitrile polymer latex either alone or in combination with hard resin emulsions is effective.

The backing sheet in one of the embodiments has a texture in the form of depressed and raised portions in accordance with the particular three-dimensional overall effect desired in the finished product. The contrast between the depressed and raised portions should be at least 5 mils; that is, the depressed portions should lie at least 5 mils below the raised portions in the textured backing sheet. Where a backing naturally bearing a smooth surface is used, this backing sheet can be embossed to produce the desired texture. Any of the con-

ventional techniques of embossing such as flatbed or rotary embossing can be used. Where a backing sheet in its natural state already possesses the desired type and character of texture, no embossing is necessary. Coarse woven fabrics possess natural texture and when they are used as backings, the outline and effect of the weave is formed in the foam structure in the finished product. Where other types of three-dimensional effects are desired in the finished product, it is preferable to use a natural smooth backing sheet which is embossed in the desired pattern. The embossing can be in any desired pattern, but the best appearance is created by a uniform overall embossing, for example, a series of evenly spaced depressions as illustrated in FIGURE 1 or more elaborate embossing, if such an effect is desired. The shape and position of the depressed areas in the backing will conform to the raised areas in the finished product. The deeper the embossing or texture in the backing, the greater will be the three-dimensional effect in the product.

In accordance with the invention, a coating of foamable composition is applied to the base and subsequently a second wear layer 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 for the foamable composition since it is unnecessary to remove the carrier as is necessary with water and organic solvent carriers. Organosols are preferred for the wear layer since they contain less plasticizer and are, therefore, less subject to staining.

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 percent of the extraneous comonomer is copolymerized therein. Suitable extraneous comonomers 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, idene, 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 ethylenically 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 comonomer for use with vinyl chloride to produce copolymers containing 60 percent or more of vinyl chloride is that, on a mol percentage basis, an initial charge of 96 percent vinyl chloride, balance comonomer, shall yield an 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:

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

$$\frac{1.33Q_2e^{.2(.3-e_2)}}{.96}$$

Instead of the single unsaturated comonomers of the types above indicated, mixtures of such comonomers 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 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 calender 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 calender 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 200° 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 the higher 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

$$[\eta] = C \lim_{C \rightarrow 0} \frac{(\ln \eta \text{ rel.})}{C}$$

when  $\eta$  rel. is relative viscosity and  $C$  is the concentration of polymer in grams per 100 cc. the concentration being such that  $\eta$  rel. has value of from 1.15 to 1.4.

In the formulation of coating compositions for use in the invention, the resin is uniformly dispersed in a mass of fluid plasticizer in a plastisol or with plasticizer and organic solvent with an organosol. 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. Coating compositions for use in the invention contain from about 45 to about 80 parts plasticizer per 100 parts resin with a range of about 60 to about 70 parts plasticizer per 100 parts resin being particularly effective for the foamable coating. The plasticizer level of the wear layer composition can be as low as 28 parts per 100 parts of resin up to about 50 parts with 30 to 40 parts of plasticizer per 100 parts of resin being preferred. The viscosity of the compositions can be reduced by the addition of a volatile diluent. Plastisols usually contain less than 10 parts per 100 parts resin of

diluents. Useful diluents include benzene, toluene, methyl ethylketone, petroleum solvents such as V.M. & P. naphtha (boiling range of 190–275° F.) and the like. If the composition is to be applied by a printing step, it is usually applied as a plastisol which requires reducing its 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 flatbed 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, stain resistance 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, alkyl derivatives of rosin, chlorinated paraffine, high molecular weight hydrocarbon condensates and the like can also be used. The plasticizer 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.

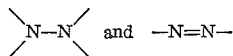
Minor amounts of stabilizers are usually incorporated in the coating compositions to reduce the effects of degradation by light and heat. Suitable light stabilizers include resorcinol disalicylate, resorcinol dibenzoate, phenyl phthalate, phenyl benzoate, o-tolyl benzoate, eugenol, guaiacol, o-nitrophenol, 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, recinoleates, abietates, salicylates and the like. Normally, the compositions contain about 0.5 to about 5 parts stabilizer per 100 parts resin. Care should be exercised in the selection of a stabilizer since some types will catalyze the decomposition of the blowing agent which will alter the operating temperature.

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 about 12 to about 40 pounds per cubic foot can be readily attained. Such results are attainable with from about 1 to about 20 parts blowing agent per 100 parts resin. About 2 to 10 parts blowing agent per 100 parts resin is 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. The amount of blowing agent will depend in large measure on the efficiency of the agent.

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



linkages decompose at elevated temperatures to yield a gas mixture high in nitrogen. Typical compounds include substituted nitroso compounds, substituted hydrazides, substituted azo compounds and the like, such as are tabulated in Table 1:

TABLE 1

| Blowing Agent                                                                                               | Decomposition Temperature, ° F. |
|-------------------------------------------------------------------------------------------------------------|---------------------------------|
| Dinitrosopentamethylenetetramine.....                                                                       | 355-375                         |
| Azodiformamide ( $\text{NH}_2-\text{C}(=\text{O})-\text{N}=\text{N}-\text{C}(=\text{O})-\text{NH}_2$ )..... | 325-400                         |
| p,p'-Oxybis (benzene sulfonyl hydrazide).....                                                               | 300-340                         |
| Azobisisobutyronitrile.....                                                                                 | 220-250                         |
| N,N'-dimethyl-N,N'-dinitrosoterephthalamide.....                                                            | 190-300                         |

The decomposition temperature depends in large measure on the particular composition. Catalyst can be added to aid in the decomposition and change the temperature range.

Blowing agents for use in the invention must be decomposed at an effective rate at a temperature below the decomposition temperature of the resin used but above the elastomeric point of the resin composition. A layer of resinous foam has heat insulating properties with the result that fusion of a prefoamed layer is very difficult and extremely slow. Therefore, in the case of compositions formulated with the preferred vinyl chloride polymers, a blowing agent decomposing between about 300 and 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 step. In the event the coating is to be fused before the wear layer is applied, then it is necessary to use a blowing agent which decomposes above the fusion temperature of the resin. In general, vinyl chloride polymer compositions attain body through partial gelation when heated to about 200° F. Thus, the minimum decomposition temperature should be about 200° F. or higher.

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 adhesion of the coating to the foam layer. After gelling the first coat, the product is cooled.

The degree of foaming of a typical plastisol formulation using different concentrations of blowing agent is shown in the Table 2:

TABLE 2

| Parts Azodiformamide per 100 parts resin | Ratio of foam thickness to original thickness | Density—lbs. per cu. ft. |
|------------------------------------------|-----------------------------------------------|--------------------------|
| 0.0.....                                 | 1/1                                           | 80                       |
| 0.1.....                                 | 1.33/1                                        | 60                       |
| 0.5.....                                 | 2/1                                           | 40                       |
| 1.0.....                                 | 3/1                                           | 27                       |
| 2.0.....                                 | 4.5/1                                         | 17.5                     |
| 3.0.....                                 | 6.5/1                                         | 12.3                     |
| 5.0.....                                 | 9.3/1                                         | 8.6                      |

It has been found that density of from about 15 to 20 pounds per cubic foot produces the most useful products.

For the product of this invention to be used as a surface covering, such as floor covering, it is essential for the product to recover from indentation or be substantially free from permanent indentation. The product is considered to meet these requirements if the permanent indentation is less than 0.010 inch after seven days following the removal of a load of 300 pounds per square inch applied with a ¼ inch diameter rod for a 72-hour period. This depth of permanent indentation would include any indentation placed in the felt base, if present. In order for the product to have this quality, it is necessary to carefully balance the thickness of the layers and the formulations of the foam and wear layers. It has been discovered that when utilizing the preferred foam formulation of about 60 to about 70 parts of plasticizer per 100 parts of resin, a product having an excellent combination of indent resistance and cushioning properties for both short and long duration time of load is obtained with wear layers of from about 0.004 to about 0.006 inch containing from 30 to 40 parts of plasticizer per 100 parts of resin when used with a foam thickness of from 0.04 to about 0.06 inch. It has also been discovered that the foam thickness should not exceed 10 times the thickness of the wear layer to minimize wrinkling of the sheet caused by winding the sheet in rolls.

In the embodiment wherein the base is embossed, it is preferred that both the coating composition containing the blowing agent and the coating composition which does not contain the blowing agent have substantially the same degree of softness or plasticity at elevated temperatures. This result can be accomplished by balancing the plasticizer efficiency in both compositions. That is, the amount and kind of plasticizer utilized in the composition should result in the respective compositions having the same degree of plasticity at high temperature. This is essential since if the solid layer is substantially softer than the foamable coating, the gas given off by the blowing agent can pass into the solid layer thereby reducing or eliminating its solid thickness. In addition, if the solid layer is too hard, it will depress the gas given off and severely hinder the reproduction of any embossed areas in the flexible backing if the textured effect is desired. It will also interfere with the formation of a uniform foam. The plasticity at elevated temperatures can also be influenced by the molecular weight of the resin used, less plasticizer being required with the lower molecular weight. As a general rule, the two compositions should fuse at approximately the same temperature which would indicate that the compositions have substantially the same degree of softness. The wear layer composition is preferably substantially the same composition as the foamable coating without the foaming agent. It has been discovered that the thickness of the solid composi-

tion is also critical. If the thickness is less than three mils the gas from the lower coating can pass right through it thereby completely losing the desired solid surface layer. In addition, the thicker the coating over eight mils, the more depressed is the fidelity in producing the textured effect and in the uniformity of the foam.

After gelling, the temperature of the composition should be reduced so the wear layer coating composition will not be affected. After cooling and the application of the wear layer, the composition is heated to a temperature sufficient to fuse the resins and 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 can be attained. In addition, the entire mass of foamable composition must be heated to a point where the blowing agent is decomposed. Where a preferred high temperature blowing agent is used, blowing does not occur until the resinous compositions have been completely fused.

If volatile diluents are used to reduce the viscosity of the coating composition, care must be taken that they are essentially completely removed from the film prior to fusion and foaming. If they are not removed, poor cell structure and blister formation will result. This removal can be accomplished by heating the composition at a temperature substantially below the fusion temperature and minimum decomposition temperature of the foaming agent for sufficient time to remove the volatile material. For example, if 5 percent of V.M. & P. naptha (boiling range 190–275° F.) is used, heating at 300° F. for three minutes will remove sufficient material so that fusion and foaming 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 under infrared heat lamps as shown in the drawing or other types of heating such as forced hot air oven or dielectric heating units can be used. In some instances, it is desirable to heat the web from both sides to offset the insulating effect of the foam.

When the base is embossed, expansion of the coating due to foaming yields a three-dimensional textured effect which duplicates in reverse the texture in the backing. That is, depressions in the backing appear as raised areas of foam. The size of the raised areas in the foamed product depends on the depth of the depressed areas in the backing and the amount of expansion in the foaming process. The decorative design is unimpaired by the foaming, in that the foamed surface accurately reproduces the printed pattern applied. The decorative effect is enhanced by the three-dimensional texture present in the surface of the foamed product.

There are certain limitations in reproducing the embossed felt design in the foam surface. Very fine and delicate embossings where the depth and width are less than about .005 inch are difficult to reproduce with good fidelity by this process. Similarly, embossings deeper than .050 inch produce large texture effects in the foam surface and loss detail. Embossings which have an average depth and width of from .015 inch to 0.030 inch have given best results.

If the expansion of the foamable composition applied to the embossed felt is too low, there will not be enough contrast between the high and low areas to produce a good textured effect. On the other hand, too great an expansion will result in a coarser foam structure which impairs design fidelity and too much lateral flow which depresses the design. For embossed felt depth of .015 inch to .030 inch, a foam expansion of 300 percent to 600 percent can give good design fidelity. With the preferred composition, a plasticized polyvinyl chloride foam, expansions of 400 percent to 500 percent give the best combination of good design fidelity and foam structure.

The textured design is also affected by the total thickness of the foam layer. In general, the foam thickness

should be from 3 to 10 times the average depth of felt embossing, but this is largely dependent upon the design selected. For the preferred embossing depths of from 0.015 inch to 0.030 inch, the foam thickness should not exceed 0.100 inch and 0.30 inch to 0.050 inch is preferred.

In order that the texture of the backing be accurately reproduced in the foamed product, it is important that the coating flow into and fill all the depressions in the textured backing and become completely level so as to have a smooth surface prior to printing and foaming. In certain instances, it is desirable to first fill the impressions and then apply a second coating to the desired thickness. This latter system greatly aids the leveling of the top coating.

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 can be adjusted so that the 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.

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 wear and soil resistance because of the solid layer and excellent resiliency in view of the foamed layer. In one form, they are also characterized by having a marked three-dimensional textured appearance conforming to the texture of the backing. 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 of the prior art.

Table 3 gives the preferred temperature and time relationship using the preferred polyvinyl chloride resin:

TABLE 3

| Film Condition <sup>1</sup> | Resin Temperature (° F.) | Oven Temperature (° F.) Circulating Air | Exposure Time Range (seconds) |
|-----------------------------|--------------------------|-----------------------------------------|-------------------------------|
| Elastomeric Point.....      | 240-300                  | 250-400                                 | 10-300                        |
| Fused.....                  | 300-375                  | 350-450                                 | 60-240                        |
| Blown.....                  | 340-400                  | 350-450                                 | 60-240                        |

<sup>1</sup> 0.014 inch plastisol on 0.25 inch cellulosic felt base impregnated with 20% vinyl acetate and 10% petroleum hydrocarbon.

The time required to reach the elastomeric point will depend in part on the film thickness and particular base as shown in Table 4:

TABLE 4

| Base                 | Film Thickness (inch) | Time/Temperature (second/° F.) |
|----------------------|-----------------------|--------------------------------|
| A <sup>1</sup> ..... | 0.008                 | 45/300                         |
| A.....               | 0.014                 | 60/300                         |
| B <sup>2</sup> ..... | 0.014                 | 90/300                         |

<sup>1</sup> Base A is a cellulosic felt of 0.025 inch thickness impregnated with 80 percent vinyl acetate homopolymer.

<sup>2</sup> Base B is a cellulosic felt of 0.043 inch thickness impregnated with synthetic rubber and urea-formaldehyde.

The following examples are given for purposes of illustration:

Examples I to VII are typical foamable compositions:

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## Example I

A foamable plastisol was formulated by grinding the following ingredients on a conventional three-roll mill:

|                                                                 | Parts |
|-----------------------------------------------------------------|-------|
| Vinyl chloride-vinyl acetate copolymer (dispersion grade) ----- | 100   |
| Diocetyl phthalate -----                                        | 30    |
| Dipropylene glycol dibenzoate -----                             | 30    |
| Stabilizer -----                                                | 6     |
| Finely divided titanium dioxide -----                           | 2.5   |
| Azodiformamide foaming agent -----                              | 2.5   |

The plastisol had a viscosity of 16,800 centipoises at 25° C. as measured with a Brookfield viscometer using a No. 6 spindle at 10 r.p.m. This plastisol can be pigmented as desired.

## Example II

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

|                                                     | Parts |
|-----------------------------------------------------|-------|
| Polyvinyl chloride (dispersion grade) -----         | 100   |
| Petroleum hydrocarbon condensate <sup>1</sup> ----- | 18    |
| Butyl benzyl phthalate -----                        | 52    |
| Pigment -----                                       | 3     |
| Stabilizers -----                                   | 4     |
| Azodiformamide foaming agent -----                  | 3.5   |

<sup>1</sup> Conoco 300—Continental Oil Company, 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 III

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

|                                                     | Parts |
|-----------------------------------------------------|-------|
| Polyvinyl chloride (dispersion grade) -----         | 100   |
| Petroleum hydrocarbon condensate <sup>1</sup> ----- | 18    |
| Butyl benzyl phthalate -----                        | 52    |
| Pigment -----                                       | 3     |
| Stabilizers -----                                   | 4     |
| Azodiformamide foaming agent -----                  | 1     |
| V.M. & P. naphtha (boiling range 190–275° F.) ----- | 5     |

<sup>1</sup> Conoco 300—Continental Oil Company.

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.

## Example IV

|                                                                 | Parts |
|-----------------------------------------------------------------|-------|
| Polyvinyl chloride (dispersion grade) -----                     | 100   |
| Didecyl phthalate -----                                         | 80    |
| Stabilizers -----                                               | 5     |
| Pigment -----                                                   | 2     |
| Wetting agent -----                                             | 3.5   |
| N,N'-dimethyl-N,N'-dinitrosoterephthalamide foaming agent ----- | 5     |

The plastisol was prepared on a three-roll mill.

## Example V

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

|                                                                 | Parts |
|-----------------------------------------------------------------|-------|
| Polyvinyl chloride (dispersion grade) -----                     | 100   |
| Didecyl phthalate -----                                         | 50    |
| Didecyl adipate -----                                           | 25    |
| Stabilizer -----                                                | 5     |
| Pigment -----                                                   | 2     |
| Wetting agent -----                                             | 3.5   |
| N,N'-dimethyl-N,N'-dinitrosoterephthalamide foaming agent ----- | 5     |

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## Example VI

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

|                                                     | Parts |
|-----------------------------------------------------|-------|
| 5 Polyvinyl chloride (dispersion grade) -----       | 100   |
| Petroleum hydrocarbon condensate <sup>1</sup> ----- | 18    |
| Butyl benzyl phthalate -----                        | 52    |
| Pigment -----                                       | 3     |
| Stabilizers -----                                   | 4     |
| 10 Azodiformamide foaming agent -----               | 4.5   |

<sup>1</sup> Conoco 300—Continental Oil Company.

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 VII

A foamable plastisol was prepared having the following composition:

|                                                     | Parts |
|-----------------------------------------------------|-------|
| 20 Polyvinyl chloride (high molecular weight) ----- | 100   |
| Polyvinyl chloride (low molecular weight) -----     | 100   |
| Azodiformamide -----                                | 3     |
| Pigment (TiO <sub>2</sub> ) -----                   | 7     |
| Dibasic lead phosphite -----                        | 7     |
| 25 Butyl benzyl phthalate -----                     | 100   |

The plastisol had a Brookfield viscosity of 6,400 (2 r.p.m., No. 3 spindle).

Examples VIII to XIV are typical wear layer compositions.

## Example VIII

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

|                                                | Parts |
|------------------------------------------------|-------|
| 35 Polyvinyl chloride (dispersion grade) ----- | 100   |
| Di(2-ethylhexyl) hexahydrophthalate -----      | 50    |
| Epoxidized soya oil -----                      | 5     |
| Wetting agent -----                            | 2     |
| Stabilizer -----                               | 3     |
| 40 Pigment -----                               | 3     |

The plastisol had a viscosity of 2,500 centipoises as measured with a Brookfield viscometer using a No. 6 spindle at 10 r.p.m. It was suitable for coating or printing by the flat bed method.

## Example IX

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

|                                                        | Parts |
|--------------------------------------------------------|-------|
| Polyvinyl chloride (dispersion grade) -----            | 100   |
| 50 Petroleum hydrocarbon condensate <sup>1</sup> ----- | 13    |
| Butyl benzyl phthalate -----                           | 37    |
| Pigment -----                                          | 3     |
| Stabilizers -----                                      | 4     |

<sup>1</sup> Conoco 300—Continental Oil Company.

The plastisol was suitable for coating or printing by the flat bed method.

## Example X

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

|                                                                                   | Parts |
|-----------------------------------------------------------------------------------|-------|
| Polyvinyl chloride (dispersion grade high molecular weight) <sup>1</sup> -----    | 50    |
| 65 Polyvinyl chloride (dispersion grade high molecular weight) <sup>2</sup> ----- | 50    |
| Petroleum hydrocarbon condensate <sup>3</sup> -----                               | 11    |
| Butyl benzyl phthalate -----                                                      | 35    |
| Pigment -----                                                                     | 3     |
| 70 Stabilizer -----                                                               | 4     |
| Wetting agent -----                                                               | 1     |
| Petroleum naphtha -----                                                           | 2     |

<sup>1</sup> Exon 654—Firestone Plastics Co., Pottstown, Pa. Relative viscosity of 2.65 (1% in cyclohexenone).

<sup>2</sup> Exon 650—Firestone Plastics Co. Relative viscosity of 2.30 (1% in cyclohexenone).

<sup>3</sup> Conoco H-300—Continental Oil Co., Ponca City, Okla.

The plastisol was suitable for coating or printing by the flat bed method.

#### Example XI

A transparent organosol was prepared by mixing the following ingredients:

|                                                 | Parts |
|-------------------------------------------------|-------|
| Vinyl chloride polymer (dispersion grade) ----- | 100   |
| Diocetyl phthalate -----                        | 16    |
| Tricresyl phosphate -----                       | 8.5   |
| Polyester plasticizer -----                     | 8.5   |
| Stabilizer -----                                | 4.0   |
| Mineral spirits -----                           | 24    |

The organosol had a Brookfield viscosity of 8,000 (2 r.p.m., No. 3 spindle).

#### Example XII

|                                                                                   | Parts |
|-----------------------------------------------------------------------------------|-------|
| Polyvinyl chloride (dispersion grade) -----                                       | 100   |
| Butyl benzyl phthalate -----                                                      | 43.7  |
| Alkyl aryl hydrocarbon -----                                                      | 4.4   |
| Barium-zinc complex -----                                                         | 3.5   |
| Titanium dioxide (58% finely divided TiO <sub>2</sub> in dioctyl phthalate) ----- | 3.0   |
| Hydrocarbon diluent -----                                                         | 4.4   |

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 XIII

A transparent organosol was prepared by mixing the following ingredients:

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

#### Example XIV

A transparent organosol was prepared by mixing the following ingredients:

|                                             | 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 XV

A size coat is prepared having the following formula:

|                                                     | Parts |
|-----------------------------------------------------|-------|
| Polyvinyl chloride latex (preplasticized) -----     | 53    |
| Carboxy vinyl polymer (thickener 2% in water) ----- | 35    |
| Water -----                                         | 12    |
| Ammonia to raise pH to 7-8.                         |       |

#### Example XVI

A sheet of felted cellulose fibers (.045 inch thick) was produced containing 25 percent by weight of the fibers of polyvinyl acetate dispersed in fine particulate form at junctions of fibers within the sheet. The size coat of Example XV was applied to the surface of the sheet at a thickness of about 0.001 inch and dried. The size surface of the sheet was embossed to a depth of 0.020 inch in a plurality of evenly spaced depressions. The foamable plastisol composition of Example

II was coated on the surface of the embossed sheet by means of a doctor blade to form a coating having a thickness of 0.010 inch. The depth of coating over one of the embossed areas was, therefore, 0.030 inch thick.

The coating was then subjected to heat at 375° F. for a period of fifteen seconds to gel the coating without decomposing the blowing agent. A four color pattern was printed by conventional block printing technique on a surface of the coated felt sheet. A film having a thickness of 15 mils was printed and leveled. The printing composition was made in accordance with Example VIII. The sheet was then passed through an oven maintained at 400° F. with a residence time of three minutes, thereby fusing the resin and expanding and foaming the composition to produce a foamed product. The foamable composition expanded to yield a product having a minimum foam thickness of about 60 mils with a plurality of raised areas conforming in location to the embossed depressions in the backing. The surface of the product had a solid vinyl composition of about 15 mils thickness free of any gas or foam areas. The product has a four-color block-printed design with a three-dimensional textured appearance and the presence of the foam composition results in a soft and resilient feel to the product underfoot.

#### Example XVII

A sheet of felted cellulose fibers having a thickness of 0.045 inch containing 25 percent by weight of the fibers of polyvinyl acetate dispersed in fine particulate form at junction of fibers within the sheet. The sheet was embossed to a depth of 0.010 inch to provide a plurality of evenly spaced depressions in the surface of the felt. The foamable plastisol composition of Example VI was prepared and pigmented in a neutral color. The plastisol composition of Example IX was prepared in four separate batches, each pigmented a different color in accordance with the colors desired in the finished product. The plastisol of Example VI was coated by means of a doctor blade over the surface of the felt bearing the depressions in such a way that the composition completely filled the depressions and gave a uniform coating of 0.005 inch. The coated sheet was then heated to a temperature of 300° F. for ninety seconds to gel the coating without decomposing the foaming agent. The four pigmented batches of the plastisol composition of Example IX were used as printing compositions in printing a four-color pattern by conventional block printing technique on the surface of the felt bearing the depressions filled with the pigmented plastisol of Example VI. A printed film having a thickness of 8 mils was produced. The printed film had a smooth and level surface. The sheet was then passed through an oven at 400° F. for three minutes, thereby fusing the polyvinyl chloride in the plastisols and decomposing the blowing agent to foam the plastisol composition. The product bore a layer of foam over the raised unembossed areas of the felt having a maximum thickness of about 70 mils with a plurality of raised areas conforming to the embossed depressions in the backing. The entire surface of the product had a solid vinyl composition layer of about 0.008 inch in thickness. The textured appearance of the product was greater than that of the product of Example XVI due to the deeper embossing of the backing and the larger proportion of blowing agent in the plastisol used in filling the embossed depressions. The finished product has a four-color, block-printed design with a highly textured surface as a result of the raised foamed areas. The presence of the foam imparts a high degree of resiliency to the product.

## Example XVIII

A sheet of felted cellulose fibers having a thickness of 0.045 inch containing 25 percent by weight of the fibers of polyvinyl acetate dispersed in fine particulate form at junctions of fibers within the sheet. The sheet was embossed to a depth of 0.010 inch to provide a plurality of evenly spaced depressions in the surface of the felt. Four batches of the foamable plastisol composition of Example III were prepared and pigmented in different colors. The plastisol composition of Example XII was prepared in four separate batches, each pigmented the same color as the foamable composition in accordance with the colors desired in the finished product. The four plastisols of Example III were printed by the flat bed technique to form an overall four-color design having a minimum thickness of 0.005 inch. The printed design was then heated to gel at 300° F. for 90 seconds. The four pigmented batches of the plastisol composition of Example XII were used as printing compositions in printing a duplicate four-color pattern in register with the first printed design by conventional block printing technique. A printed film having a thickness of 8 mils was produced. The printed film had a smooth and level surface. The sheet was then passed through an oven at 400° F. for three minutes thereby fusing the polyvinyl chloride in the plastisol compositions and decomposing the foaming agent to expand the foamable layer. The product bore a layer of foam over the raised unembossed areas of the felt having a maximum thickness of about 50 mils with a plurality of raised areas conforming to the embossed depressions in the backing. The entire surface of the product had a solid vinyl composition layer of about 0.008 inch in thickness.

## Example XIX

A first plastisol composition is formulated by mixing the following ingredients on a conventional Cowles mixer:

| Parts                                                           |     |
|-----------------------------------------------------------------|-----|
| Vinyl chloride-vinyl acetate copolymer (dispersion grade) ----- | 100 |
| Dioctyl phthalate -----                                         | 30  |
| Dipropylene glycol dibenzoate -----                             | 30  |
| Stabilizer -----                                                | 6   |
| Pigment -----                                                   | 4   |

The plastisol of Example I is applied as a uniform coating of about 0.008 inch in thickness on the surface of a release paper. The release paper has a coating of a complex compound of the Werner type in which a trivalent nuclear chromium atom is coordinated with an acyclic carboxylic acid group having at least 10 carbon atoms. Such a composition is disclosed in United States Patent 2,273,040, issued February 17, 1942. The plastisol coating is then heated to a temperature of 300° F. for 1 minute to gel the composition. The gelled coating is then cooled. The first plastisol composition is then coated on the surface of the cooled, gelled coating to form a uniform coating of 0.005 inch in thickness. The coatings are then heated to a temperature of 400° F. for a period of 2 minutes to fuse the compositions and completely decompose the blowing agent to form a foam layer of 0.040 inch thickness integrally bonded to the wear layer of 0.005 inch thickness. The fused and foamed product is then cooled and the release paper is stripped from the back of the product. The resulting product has a foam underlayer with a solid resinous wear layer integrally bonded thereto.

## Example XX

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

| Parts                                                           |     |
|-----------------------------------------------------------------|-----|
| Vinyl chloride-vinyl acetate copolymer (dispersion grade) ----- | 100 |
| Dioctyl phthalate -----                                         | 60  |

|                                       |   |
|---------------------------------------|---|
| Alkyl aryl hydrocarbon -----          | 5 |
| Dibasic lead phosphite -----          | 1 |
| Finely 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 0.008 inch on the surface of a 0.025 inch thick cellulose felt sheet impregnated with 9 percent vinyl acetate and 30 percent hydrocarbon resin. The felt sheet had previously been coated with the size coating of Example XV at the rate of 0.025 pound per square yard and dried. The plastisol coating is then heated to a temperature of 310° F. for a period of 50 seconds to obtain a composition temperature of 250° F. thereby gelling the coating into a film having an elongation of 100 percent. The organosol composition of Example XI was applied to the surface to form a uniform coating of 0.004 inch in thickness. The sheet was then passed through an oven which gradually raised the temperature of the coating to 400° F. The foamable coating and the organosol coating fused and the blowing agent decomposed to form a product having a foam layer of 0.040 inch in thickness and a solid layer covering the foam layer of 0.004 inch in thickness. The product made and excellent floor covering having high wear and stain resistance and excellent indent recovery.

## Example XXI

A foamable plastisol is prepared according to Example VII. The plastisol had a Brookfield viscosity of 6,400 at 25° C. using a No. 3 spindle at 2 r.p.m. The plastisol was applied as a uniform layer of 0.008 inch in thickness on the surface of a felt sheet 0.045 inch in thickness. The felt sheet was composed of cellulosic fibers impregnated with 5% of a urea-formaldehyde resin and 25% of a copolymer of butadiene-acrylonitrile. The sheet had been heated to cure the urea-formaldehyde resin. The felt sheet had previously been coated with the size coating of Example XV at the rate of 0.025 lbs. per square yard followed by drying the size coat. The plastisol coated felt was then passed through an oven heated at 300° F. at the rate of 60 ft. per minute. The oven was 90 ft. long. The sheet was then allowed to cool and fed to a conventional rotogravure printing machine which applied a decoration on the surface of the sheet with conventional vinyl printing inks. Because of the soft surface of the gelled foamable coating, excellent fidelity of print was obtained. The printed sheet was then passed to a reverse roll coater where an organosol coating of 0.005 inch in thickness was applied having the composition of Example XI. The sheet was passed from the coater at a speed of 30 ft. per minute into an oven having three zones each of 30 ft. in length. The zones were heated to 325 F., 425° F., and 425° F., respectively. During this heating operation, the compositions became fused and the blowing agent decomposed to foam the foamable layer to a thickness of 0.040 inch. The product was then cooled and wound on a collecting roll. The product could be utilized directly as a floor covering having excellent indent recovery. The solid transparent wear layer of 0.005 inch in thickness had excellent resistance to wear and staining and the design printed on the surface of the foamable composition was clearly visible through the transparent wear layer.

The product showed instantaneous recovery from spike heel shoes. Utilizing an Armstrong indent tester with a  $\frac{15}{16}$  inch diameter dome of silence type pin with 100 lbs. of dead load for 60 seconds dwell, 90% recovery showed within 60 seconds and 100% recovery within 10 minutes. A long term indent test utilizing  $\frac{3}{4}$  inch diameter domes of silence with 112 and 224 lbs. per square inch for 9 days dwell showed complete recovery in one day for the lesser weight and approximately 90% recovery in one week for the higher weight.

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

What is claimed is:

1. A method of producing a resilient decorative surface covering which comprises applying a first coating of a thermoplastic resinous composition containing an effective amount of foaming agent to one surface of a flexible sheet which contains depressed areas in its surface, applying a second coating of a thermoplastic resinous composition to cover said first coating, heating said first and second coatings to fuse the resinous compositions and decompose the foaming agent thereby creating a textured decorative surface bearing a plurality of raised areas which conform in location to the depressed areas in said surface of said flexible sheet and having a resinous surface formed by said second coating and thereafter cooling the fused resinous compositions.

2. The method according to claim 1 wherein said thermoplastic resinous composition containing a foaming agent is a foamable plastisol of vinyl chloride polymer comprising about 45 to about 80 parts plasticizer per 100 parts vinyl chloride polymer.

3. The method according to claim 1 wherein said first and second coating compositions fuse at approximately the same temperature.

4. The process of claim 1 wherein said flexible sheet having depressed areas in its surface is stripped from said coatings after decomposing the foaming agent.

5. A method of producing a resilient decorative surface covering which comprises applying a first coating of a vinyl resinous composition containing an effective amount of foaming agent to one surface of a flexible sheet which contains depressed areas in its surface, applying a second coating of vinyl resinous composition to cover said first coating, heating said first and second coatings to fuse the resinous compositions and decompose the foaming agent thereby creating a textured decorative surface bearing a plurality of raised areas which conform in location to the depressed areas in said surface of said flexible sheet and having a vinyl composition surface formed by said second coating and thereafter cooling the fused resinous compositions.

6. The process of claim 5 wherein said vinyl resinous compositions comprise vinyl chloride resin and plasticizer in a ratio of about one part of resin to 0.5 to 1.5 (parts) of plasticizer.

7. The method according to claim 5 wherein said foaming agent has a minimum initial decomposition temperature of at least about 200° F. and is substantially completely decomposed at a temperature below 450° F.

8. A method of producing a resilient decorative surface covering which comprises applying a first coating of a thermoplastic resinous composition containing an effective amount of foaming agent to one surface of a flexible sheet which contains depressed areas in its surface, heating said composition to gel the composition, applying a second coating of thermoplastic resinous composition to cover said first coating, heating said first and second coatings to fuse the resinous compositions and decompose the foaming agent thereby creating a textured decorative surface bearing a plurality of raised areas which conform in location to the depressed areas in said surface of said flexible sheet and having a resinous surface formed by said second coating and thereafter cooling the fused resinous compositions.

9. A method of producing a resilient decorative surface covering which comprises applying a first coating of a vinyl chloride resinous composition containing an effective amount of foaming agent to one surface of a flexible sheet which contains depressed areas in its surface, heating said composition to gel the composition, cooling said gelled composition, applying a second coating of vinyl chloride resinous composition to cover said first coating, heating said first and second coatings to fuse the resinous

compositions and decompose the foaming agent thereby creating a textured decorative surface bearing a plurality of raised areas which conform in location to the depressed areas in said surface of said flexible sheet and having a vinyl chloride resinous composition surface formed by said second coating and thereafter cooling the fused resinous compositions.

10. A resilient decorative surface covering having a textured surface which comprises a flexible sheet having in one surface thereof a plurality of areas depressed below said one surface, a layer of expanded and foamed plasticized thermoplastic resinous composition covering said one surface of said flexible sheet, and a layer of plasticized thermoplastic resinous composition bonded to the opposite surface of said foamed layer, said surface covering having a plurality of raised and depressed portions on its upper surface, said raised portions conforming in location to said depressed areas in said flexible sheet.

11. A resilient decorative surface covering having a textured surface which comprises a flexible sheet having in one surface thereof a plurality of areas depressed below said one surface, a layer of expanded and foamed plasticized vinyl resinous composition covering said one surface of said flexible sheet, and a layer of plasticized vinyl resinous composition bonded to the opposite surface of said foamed layer, said surface covering having a plurality of raised and depressed portions on its upper surface, said raised portions conforming in location to said depressed areas in said flexible sheet.

12. A resilient decorative surface covering having a textured surface which comprises a flexible sheet having in one surface thereof a plurality of areas depressed below said one surface, a layer of expanded and foamed plasticized vinyl chloride resinous composition covering said one surface of said flexible sheet, and a layer of plasticized vinyl chloride resinous composition bonded to the opposite surface of said foamed layer, said surface covering having a plurality of raised and depressed portions on its upper surface, said raised portions conforming in location to said depressed areas in said flexible sheet.

13. A resilient decorative surface covering having a textured surface which comprises a flexible sheet having in one surface thereof a plurality of areas depressed at least 5 mils below said one surface, a layer of expanded and foamed plasticized thermoplastic vinyl resinous composition covering said one surface of said flexible sheet, said foamed layer is from about 0.020 to about 0.100 inch in thickness and a layer of plasticized thermoplastic vinyl resinous composition bonded to the opposite surface of said foamed layer, said surface covering having a plurality of raised and depressed portions on its upper surface, said raised portions conforming in location to said depressed areas in said flexible sheet.

14. A resilient decorative surface covering having a textured surface which comprises a flexible sheet having in one surface thereof a plurality of areas depressed below said one surface, a layer of expanded and foamed plasticized vinyl resinous composition comprising 45 to 80 parts plasticizer per 100 parts of vinyl resin covering said one surface of said flexible sheet, and a layer of plasticized vinyl resinous composition comprising 30 to 40 parts plasticizer per 100 parts vinyl resin bonded to the opposite surface of said foamed layer, said surface covering having a plurality of raised and depressed portions on its upper surface, said raised portions conforming in location to said depressed areas in said flexible sheet.

15. A resilient decorative surface covering having a textured surface which comprises a flexible sheet having in one surface thereof a plurality of areas depressed below said one surface, a layer of expanded and foamed plasticized vinyl chloride resinous composition comprising 45 to 80 parts plasticizer per 100 parts vinyl chloride resin covering said one surface of said flexible sheet, and a layer of plasticized vinyl chloride resinous composition comprising 28 to 50 parts plasticizer per 100 parts vinyl

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chloride resin bonded to the opposite surface of said composition layer, said surface covering having a plurality of raised and depressed portions on its upper surface, said raised portions conforming in location to said depressed areas in said flexible sheet.

16. A resilient decorative surface covering having a textured surface which comprises a flexible sheet having in one surface thereof a plurality of areas depressed below said one surface, a layer of expanded and foamed plasticized vinyl resinous composition comprising 45 to 80 parts plasticizer per 100 parts of vinyl resin covering said one surface of said flexible sheet, and a layer of plasticized vinyl resinous composition comprising 28 to 50 parts plasticizer per 100 parts vinyl resin bonded to the opposite surface of said foamed composition layer, said surface

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covering having a plurality of raised and depressed portions on its upper surface, said raised portions conforming in location to said depressed areas in said flexible sheet, the ratio of plasticizer to resin being substantially the same in both resinous compositions.

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EARL M. BERGERT, *Primary Examiner.*