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POLYVINYL CHLORIDE FILM ADHERED TO A SELF-BONDED WEB OF
CONTINUOUS ORIENTED POLYPROPYLENE FILAMENTS

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FIG. 2

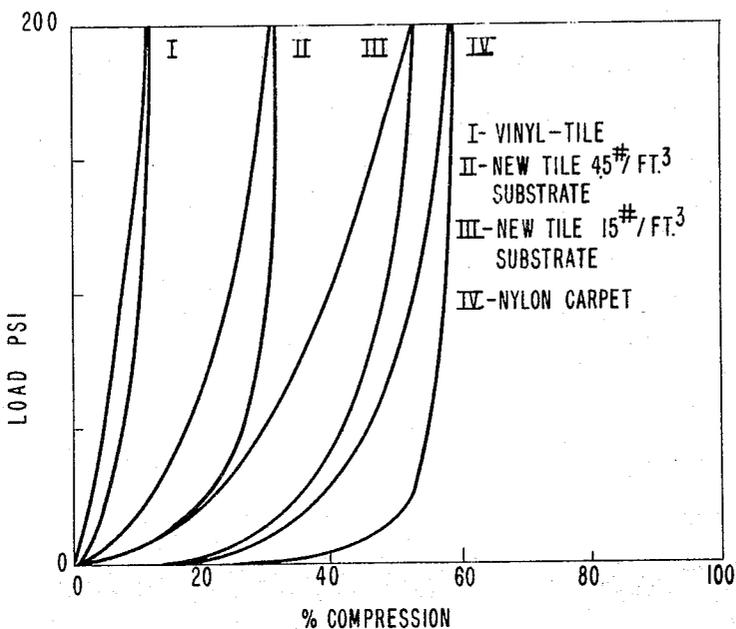
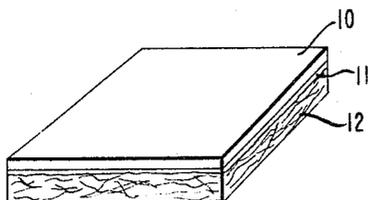


FIG. 1



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POLYVINYL CHLORIDE FILM ADHERED TO A SELF-BONDED WEB OF CONTINUOUS ORIENTED POLYPROPYLENE FILAMENTS

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1 Claim. (Cl. 161—150)

This invention relates to laminates of fibrous webs with a continuous coating, particularly suitable for use as floor tiles and wide width floor covering materials.

Use of natural and synthetic polymeric materials in structures suitable for use as floor covering is well known. Asphalt, linoleum and vinyl compositions are well known examples of such uses. In addition, the applicability of structures of fibrous materials in connection with floor coverings is also known. The most widely accepted and utilized examples of this are the rug cushions or underlayment pads which are employed in conjunction with conventional woven and tufted rugs to provide extra softness and to reduce wear. The use of asphalt-impregnated felts containing short length cellulose pulp and other staple fiber materials in more rigid, less flexible structures is also known.

More recently, specific floor covering sheet structures have been developed which are based on asphalt-impregnated or other polymer saturated felts, surfaced with a smooth coating to provide abrasion resistance. This latter aspect of the art is exemplified in U.S. Patent 2,836,528, issued May 27, 1958, to D. E. Ford and in Canadian Patent 587,376, issued November 17, 1959 to Rainar. While these articles have met with commercial success, it has been recognized that further improvements in the art of floor covering materials are desirable.

In the past, efforts have been made to produce floor covering materials which approximated the cushioning effect of soft carpets. Toward this end, for example, tiles have been made with a core of foam material. However, such tiles lack resilience and form recovery after load. These deficiencies become apparent in the so-called "edge-effect" which means that of two adjacent tiles, the one which is loaded will become permanently depressed at the edge, presenting a sharp difference in vertical level. This vertical level leads to collection of dirt, tripping, and extensive damage to the exposed edge of the tile.

It is an object of this invention to provide new and improved forms of fiber-containing laminated materials suitable for floor coverings. It is a further object to provide floor covering materials derived from fibrous webs which combine resilience, abrasion resistance, recovery from deformation, and a low level of noise generation. Other objects will become apparent from the specification which follows:

In accordance with this invention, there is provided a novel resilient fibrous multilayer structure suitable for use as a floor-covering material having an overall thickness of between about 20 and 200 mils and comprising:

(1) As a first element, a synthetic polymer film, preferably calendered, said film being at least one mil thick, but not more than 15 mils thick, and constituting between 1% and 40% of the total weight of the whole and

(2) As a second element firmly bonded to said film, a bonded fibrous batt having a density of at least 15 lbs./ft.³, and not over 45 lbs./ft.³, comprising molecularly oriented fiber members, the batt constituting the remainder of the total weight of the structure, except for laminating adhesive.

Up to 30% of a filler material may be present in the laminate for decorative or other purposes.

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The fibers of the batt may be self-bonded, i.e., no extraneous binder, or may be bonded with up to about 120% of a binder based on the weight of fiber and are characterized by:

- (a) Average fiber length of at least 1.0 inch,
- (b) Filament size between 1 d.p.f. (denier per filament) and 20 d.p.f.,
- (c) Initial modulus of at least 10 g.p.d. (grams per denier),
- (d) Break extensibility over 5%, but less than 400%,
- (e) Moisture regain, based on dry weight, less than 5% at 65% RH (relative humidity) and 21° C.,
- (f) Creep of less than 1% per hour under a tensile load of 20% of the breaking strength of the fiber,
- (g) Dimensional stability (under temperature change) less than $\pm 2\%$ variation in length over the interval between 0° C. to 100° C.

FIGURE 1 is an enlarged sectional view of a laminate of the invention.

FIGURE 2 is a graphic representation of the resilient properties of the present invention.

The structure of FIGURE 1 shows film 10 bonded through adhesive 11 to bonded fibrous batt 12.

The materials of the invention exhibit excellent behavior as flooring materials. In the properties of indentation resistance, abrasion resistance and dimensional stability, the floor coverings of the present invention are at least equivalent and in most cases substantially superior to commercial materials. In addition, they offer outstanding improvements in regard to resilience, warmth and quietness. These behavior characteristics, combined with light weight, durability, and a modest cost, combine to make flooring materials of a superior nature.

As already suggested, there are a number of critical limitations which must be observed in constructing the floor materials of the present invention. The effect of these critical limitations will now be discussed. It is necessary, first, that there be a polymeric surface film at least one mil thick and not over 15 mils thick. The film is found to provide a dirt-resistant strong long-wearing surface. If the film is less than one mil thick, or less than 1% of the total structure weight, the flooring material will be deficient in wear life. If, on the other hand, the material is too thick, that is, more than 15 mils thick, the structure will be characterized by inadequate softness and, of course, will be uneconomical. If the film is over 40% of the total structure, the resilience of the composite will be poor, the indentation resistance will be low, and the overall structure will behave substantially like conventional floor tile materials. The film in the composite structure of the present invention coats with the base material and provides recovery from deformation, low indentation and permanent set. Illustrative of films which may be employed are oriented polyethylene terephthalate, oriented nylon, oriented polypropylene oriented polyoxymethylene, and other oriented synthetic polymer films, as well as calendered and cast films.

The critical limitations which apply to the fiber element of this composite flooring material are equally significant. First, the fiber elements must be bonded to one another in order to provide a stable, firm structure which is adequately resistant to delamination and acts as a network rather than as independent filler particles. Where necessary or desired, needle punching may be employed to prevent delamination. In addition, the fibers must be oriented, that is, they must be drawn as is taught in conventional synthetic fiber technology. Undrawn fibers are unsatisfactory since they give low form recovery and high residual set. Undrawn fibers also lead to a brittle structure which has poor resistance to shock.

The fiber elements will lie substantially in the plane of the batt and will be bonded at numerous points throughout the batt. The batts may be self-bonded, i.e., the bond consists of the same material as the fiber elements, as is accomplished by heat fusing or solvent fusing of contacting filaments at spaced points, or the fibers in the batt may be bonded to each other by an adhesive binder in a weight ratio of 100 parts of fiber to maximum of about 120 parts of binder.

Illustrative of the synthetic organic polymeric fibers that may be employed are e.g., polyethylene terephthalate, polyhydrocarbons, e.g., polypropylene, etc. Where binder is used, it may be selected from any of the large group of conventional binders. Illustrative of such bonding agents are polyurethane binders, copolyester binders, etc. The modulus of the binder selected should preferably be not greater than that of the fiber employed and will ordinarily be adjusted for proportions as will be obvious to those skilled in the art.

At least 30% by weight of the composite structure is fibrous material. If less than 30% of fibrous material is employed based on the weight of the overall structure, the flooring material will have poor resilience and poor form recovery.

There are also a number of other critical requirements for the individual fibers which go to make up the bonded fiber element. The fiber may be continuous filament material or may be staple fiber, but the length of the fiber should be at least about 1.0 inch. If shorter fibers are used, it is found that the composite structure requires excessive binder. This, in turn, leads to a material which has poor resilience and poor form recovery. The fiber denier must be greater than one denier per filament. Finer fibers present difficulties in fabrication. If the filaments are heavier than 20 d.p.f., they give inadequate support for the film surface and the film surface is easily damaged.

Furthermore the oriented fiber material employed in the fiber element of the present flooring materials should have an initial modulus in excess of 10 g.p.d. This characteristic is necessary for high form recovery and high impact resistance. The break extensibility of the fibers must be greater than 5% to avoid a brittle final product. In addition, the moisture regain and creep of the fibers must be low and the dimensional stability must be high as is apparent to those skilled in the art when consideration is given to these factors. Furthermore, there must be a firm adherent bond between the film element and the fibrous element in order to provide a strong structure resistant to delamination.

The present advance requires a combination of factors and interaction between components which are involved to permit the formation of a superior flooring structure.

The most critical and specific novel structural requirement of the present invention resides in the density range of the bonded fibrous portions of the laminat. While it is known to incorporate fiber stock in many floor covering products, this is generally done by adding scrap material to a semifluid mass of polymer, such as asphalt, or vinyl compound and casting therefrom a solid, high-density slab or sheet. It has now been found that a different type of flooring material can be prepared, wherein an open bonded-fiber substrate provides resilience, warmth and quietness. These advantages are achieved only when the density of the fibrous layer, including the bonding material, is in the range of 15 lbs./ft.³ and 45 lbs./ft.³.

Among the characteristics which distinguish the products of the invention from tiles and other hard surface flooring materials of the prior art, are low set, i.e., below about 30% and never higher than 30 mils set, measured after compression at 3000 p.s.i. for 1 hour, low compressional modulus (softness) combined with high form recovery, an impact resistance as measured by energy

to damage, greater than 2×10^3 g. cm., low thermal conductivity and high abrasion resistance. When using a low density base, i.e., below 15 lbs./ft.³, both the indentation and permanent set are too high to yield the form recovery which is desired. Similarly, a base density of above 45 lbs./ft.³ results in too low an indentation and in the diminution of any cushioning effect. While high form recovery is desired, it is preferred that this be accompanied by relatively low (e.g., below 70%), work recovery.

Perhaps the most unusual quality of the laminate of the present invention is its similarity to carpeting in its feel underfoot and its similarity to high quality hard surface flooring coverings in wear, i.e., dirt and abrasion resistance. Thus a plot FIGURE 2 of load vs. compression on application and removal of load shows that the hysteresis loop for the products of the invention approaches that for carpeting as distinguished from conventional hard surface floor coverings, such as asphalt tile, etc. The resilience is measured on an Instron applying a 200 lb. load through a 1 inch ram at 0.05 in./min.

The practice of the present invention can more readily be appreciated with reference to examples showing specific flooring compositions and structures.

EXAMPLE I

Polypropylene flake at a melt index of 12 is screw-melted through a $\frac{1}{4}$ " layer sand filter bed consisting of 4 mls. A sand, 4 mls. 60/80, 4 mls. 40/60 balance A sand and metered at 6 grams/minute. It is spun from a 2" spinneret having 60 capillaries of 15 mils diameter. The spinneret temperature is controlled at 275° C. A set of feed rolls pulls the yarn away from the spinneret at 200 yards/minute and the draw rolls draw the filament 5.5× at 1100 yards/minute. The fibers are triboelectrically charged over three brass bars and are dispersed by an air jet operating at 94 p.s.i.g. These dispersed, drawn, charged filaments are deposited on a grounded table which reciprocates in two planes to form a web. Table motion is adjusted to obtain desired web weight and uniformity. Webs are collected for approximately 1.8 minutes each. Sheet basis weight is approximately 3 oz./yd. Typical fiber properties are as follows: denier, 2 d.p.f.; tensile, 3.8 g.p.d.; break elongation, 48%; M_i (initial modulus) 35 g.p.d. The web formed in this manner is coherent even before bonding. The appearance of the web is uniform. Similar results are obtained when a continuous moving belt is used in place of the reciprocating table. Six 3 oz./yd.² batts are plied together and consolidated and then needle-punched on a Waterman needle loom (20 H.P.) having 25 gauge needles at a needle density of 740/ft.². The machine is run at 84 strokes/minute and the multi-ply web is subjected to one pass at 0.14 in./sec. Following needle-punching, the structure is thermally bonded by the application of steam for 5 min. at 95 p.s.i.g. leading to a final material having a weight of 18 oz./yd.². This bonded fibrous structure is then ready for lamination to form a flooring material, as shown in the following example.

EXAMPLE II

The fibrous polypropylene web prepared as in Example I is made into a laminated, smooth-surface flooring construction. The laminate is formed from the web and a 6 mil thick layer polyvinyl chloride precalendered commercial film (Style FC-638, supplied by Elm Coated Fabrics, New York, N.Y.). Lamination is brought about by first applying a contact type vinyl emulsion-thickened adhesive. A light coating of the adhesive is spread over the web and the calendered film is placed over the adhesive-coated fibrous structure and lightly pressed. The laminate is allowed to air-dry several hours at room temperature for maximum strength. The resulting floor tile is a sheet of material $\frac{1}{8}$ " thick, the density of the bonded batt being about 17 lbs./ft.³. The properties of this

sample (New Tile A) are compared with those of commercial flooring in Table I below. Samples B, C, D, and E were prepared as in Example II and differed from each other in that the density of these several samples was varied as shown by changing the amount of fiber and the level of compression during bonding.

from a 2" spinneret having 40 capillaries of 9 mils diameter each. The pack block is kept at 270° C. and a spinneret temperature of 248° C. is maintained. A set of feed rolls pulls the yarn away from the spinneret at 300 yds./min. and a set of draw rolls stretch the filaments 3×. The fibers are triboelectrically charged

Table I
COMPARISON OF PROPERTIES OF FLOORING MATERIALS

	New Tile			Asphalt Tile	Vinyl Asbestos	Vinyl	Rubber	Linoleum	Experimental tiles	
	A	B	C						D ¹	E ²
Thickness (mils).....	125	61	125	125	125	125	125	125	50	125
Weight (oz./yd. ²).....	20			181	188	124	188	128		
Density (lb./ft. ³).....	17	44	16	120	125	82	125	85	59	14
1 hr. at 3,000 p.s.i.....	91	16	94	37	33	78	106	109		86
And residual set.....	25	9	30	25	11	8	14	23		40
Impact Resistance (Energy to Damage, g. cm. X10 ³).....	14			0.225	1.35	11		2.25		
Abrasion Resistance (wt. loss, gms./10 ³ cycles).....	0.25			2.4	2.1	1.9	2.7	1.5		
Thermal Conductivity B.t.u./ft. ² /hr./° F./in.....	0.39			3.32	4.15	2.40	3.32	1.62		
Percent Compression at 200 p.s.i.....		28	43			10			27	43
Percent Work Recovery.....		66	49	87	86	75	86		87	52

¹ High batt density gives high work recovery and therefore decreased comfort.

² Low batt density leads to high residual set.

³ Mils.

The measurements referred to in Table I were made as follows:

Indentation: A load of 3000 lbs./in.² was applied to the sample for one hour through a 1/4" brass rod having a round bottom. The indentation is directly measured in mils. The load is removed for one hour and the residual indentation is then measured in mils.

Impact resistance: Impact to cause cracking is determined by dropping a steel ball on a sample which bridges supports separated by a distance of one inch.

Abrasion resistance: ASTM-D1242-56 Procedure B, Abradant E, 2 1/2 lb. load.

Thermal conductivity: Determined on a modified Cenco-Fitch Heat Conductivity Apparatus, Cat. No. 77,555.

EXAMPLE III

The polypropylene fiber web described as in Example I is made into a flooring surface material using each of several different smooth surface calendered film synthetic polymer compositions. Each of these experimental film surfaces is adhered to the fibrous substrate with a polyester resin adhesive as indicated. The film layer thickness is shown in each case and, as a test of utility, the abrasion resistance of each of the composite structures is tested and is given in Table II.

Table II

Film	Thickness (mils)	Abrasion Resist. Wt. Loss (g./10 ³ cycle)
1. "Delrin" ¹	3.5	0.13
2. Saran ² F-220.....	2.2	0.16
3. Nylon.....	7.3	
4. "Mylar" ³	4.8	0.25
5. "Adiprene" ⁴	12.0	
6. Vinyl.....	6.5	0.25

¹ Trademark for Du Pont's acetal resins.

² Trademark of Dow Chemical Company.

³ Trademark for Du Pont's polyester film.

⁴ Trademark for Du Pont's urethane rubbers.

EXAMPLE IV

Polypropylene polymer flake with a melt index of 10 is screw-melted at a maximum temperature of 248° C. through a 1/4" layer sand filter bed consisting of 4 mls. A sand, 4 mls. 60/80, 4 mls. 40/60, and balance A sand and is metered at a rate of 6 g./min. It is spun

over three brass bars and are dispersed by an air jet operating at 10 ft.³/min. at room temperature. The dispersed drawn charged filaments are deposited for three minutes on a grounded plate 12" x 24" in area which reciprocates in two planes to form a web. Web weights vary between 12 and 17 g. Typical fiber properties are as follows: tensile strength 2.15 g.p.d., elongation 215%, initial modulus 19, denier 1.6. The web formed in this manner is strong even before bonding. The appearance of the web is uniform. Similar results are obtained by collecting the web continuously between a pair of rolls. Sheets so collected may be plied together to give a fibrous batt of any desired basis weight. The material can then be consolidated by needle-punching or by impregnation with a binder. After bonding, the sheets are suitable for lamination as described in Example II above.

EXAMPLE V

The following example shows the preparation of a floor covering material which is impregnated. The procedure of Example I is followed up to the point of web formation and needle-punching. At this point, instead of bonding by thermal means, the web is impregnated with an aqueous polyvinyl chloride emulsion (25%) applied by dipping. The weight of polyvinyl chloride impregnant is approximately equal to the weight of the fiber which is present before impregnation. Thus the resulting fibrous impregnated web is a 50% fiber and 50% vinyl chloride resin. This resin-impregnant is then cured by heating at 100-120° C. in a forced draft oven between steel plates for 1 to 2 hours. Following impregnation, the substrate having a density of 21 lb./ft.³ and thickness of 140 mils is coated with a calendered polyvinyl chloride film of 6 mils, as described in Example II to give a floor covering material with a density 35 lbs./ft.³ and a thickness of 100 mils. The tile had excellent properties.

EXAMPLE VI

Polymer flake of polyethylene terephthalate, relative viscosity 38.5 semi-dull, is melted in a screw melter at a maximum temperature of 302° and metered at a rate of 7.5 g./min. through a sand 1" A sand filter bed and a 2" 20 capillary 9 mil diameter spinneret. The pack block is held at 280° C. and the spinneret temperature is controlled at 265° C. The bundle of fibers is ad-

vanced through an airjet operating at 92 p.s.i.g. in which the air moves at a rate of 19 c.f.m. The fiber bundle is drawn across brass bars to triboelectrically charge them. The distance from the spinneret face to the first bar is 123 cm., to the second bar 135 cm., and to the third bar 148 cm. The distance from the spinneret face to the air jet advancing assembly is 191 cm. The second brass static charging bar is displaced laterally from the plane of the fibers a distance of 6 cm. so that sufficient tension is applied to the fibers as they pass down. The charged polyethylene terephthalate filaments are deposited on a reciprocating table similar to that described in Example I. Typical filament properties obtained are as follows: tensile strength 2.28 g.p.d., elongation 134.8%, initial modulus 26.9 g.p.d., denier 1.54%. Following the fiber deposition, the webs are plied together, needle-punched, and impregnated as described in Example V.

EXAMPLE VII

Using the bonded fibrous substrate of Example VI, floor covering material is prepared in a manner similar to that described in Example II. The fibrous web is coated with an adhesive emulsion of polyvinyl chloride and is then laminated with a 6 mil sheet of vinyl polymer similar to that employed in Example V. The resulting floor tile is quite comparable to that contained in the earlier example, having the same excellent level of behavior properties.

An impregnated polyethylene terephthalate substrate coated with a 6-mil polyvinyl chloride film to give a laminate having a thickness of 56 mils and a density of 25 lbs./ft.³ had an indentation at 3000 p.s.i. for 1 hour of 39 mils and a residual set of 17 mils. A similar laminate having a thickness of 65 mils and a density of 37 lbs./ft.³ had an indentation of 26 mils and a residual set of 16 mils at the same loading.

In a modification of the above procedure, a flooring material composed entirely of polyester components is prepared by using an oriented film, "Mylar" polyester film 4.8 mils thick as the surface of the laminate.

The floor covering materials of this invention are suitable for a wide variety of specific utilities. Most logically, they may be employed in the shape of well-known tiles which are adhered to a sub-flooring surface with conventional adhesives. In the form of such tiles, they bond well to the floor, provide a high level of durability, and, as already indicated, superior aesthetic properties. In addition, because of their smooth surface, they are suitable for use in heavy-wear and high-soilage areas such as kitchens, laundries and utility rooms. However, it is also possible and desirable to use them in areas where aesthetic requirements are more highly emphasized. For example, it is convenient to apply decorative patterns, coloring material, marbled effects, metal, flakes, fiber fragments and similar ornamental materials to the surface of the fibrous substrate at the same time as the adhesive is applied. In this way, when the film surface is deposited on top of the fiber substrate, the ornamental materials are clearly visible in depth and provide attractive ornamental designs.

In addition, it is equally suitable to apply marbled or patterned designs within the interior or on the surface of the oriented film which comprises the wear surface

on the floor covering. In this way other patterned effects may be readily obtained. Further modifications of the present invention are, of course, obvious and will be readily perceived by those skilled in the art.

In addition to separate individual tile elements, the products of the present invention may be made in the form of wide roll goods, such as smooth-surfaced rugs and the like, which can be employed for smooth uniform coverings of large areas. In this application, the products of the present invention are normally in the thin portion of the ranges indicated earlier in the specification. As roll goods, these materials perform the same function as linoleum, vinyl roll goods and other similar materials. However, in accordance with the teaching of this invention, they provide, in addition to a smooth wearing surface, the additional and desirable features of superior aesthetic appeal and comfort which arise by virtue of the specified structure as well as increased ability to hide floor surface imperfections and greatly increased tear strength.

In addition to these end uses, other applications are also possible. For example, the products of the present invention may be used as coverings for stairs and stairways, as automobile floor coverings, as elevator floor coverings, as covers for bars and other serving areas, as resilient tops for desks and other articles of furniture, and as wall coverings for padded cells in hospitals, jails, mental institutions, office buildings and the like. In all of these applications, it is possible to employ these products, by virtue of their thermoplastic nature, as coverings for curved and three-dimensional surfaces as well as for flat two-dimensional surfaces. In this regard, they offer substantial advantages over flooring materials which are not sufficiently thermoplastic to be molded and shaped to three-dimensional contours. In application to curved and shaped surfaces, the products of the present invention have the further advantage that they have no tendency to crack or disintegrate under the compressive and extensive forces of the molding operation.

What is claimed is:

A laminate having a thickness of between about 20 and 200 mils comprising a film of calendared polyvinyl chloride and a base layer comprising a self-bonded web of continuous oriented polypropylene filaments adhered to said film, the filaments being between 1 and 20 denier per filament and having an initial modulus of at least 10 grams per denier, the film having a thickness of between about 1 and 15 mils and constituting between about 1% and 40% of the weight of the laminate and the bonded web having a density of between about 15 lbs./ft.³ and 45 lbs./ft.³ with at least 30% by weight of the composite structure being fibrous material.

References Cited by the Examiner

UNITED STATES PATENTS

2,537,126	1/1951	Francis	161—151 X
2,836,528	5/1958	Ford	161—231
2,875,115	2/1959	Lott et al.	161—151
2,905,585	9/1959	Hubbard et al.	161—402 X
2,949,394	8/1960	Rodman	161—151
3,015,595	1/1962	Moser et al.	161—151 X

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