

SPECIFICATION

Pressure-sensitive products

This invention relates to pressure-sensitive products.

5 Pressure-sensitive products which may be bonded to a desired substrate with the application of pressure are well known. In order to ensure a more permanent bonding of such products to the substrate, it is desirable for such products to exhibit the highest possible adhesion values. Although it is possible to achieve a greater degree of adhesion by chemical modification of the polymers which normally constitute the pressure-sensitive adhesive layer, such an approach requires some degree of sophisticated chemical capability which might not be possessed by some of the ultimate end users of those conventional pressure sensitive adhesives which are commercially available. Hence, a need exists for a simple means to increase the adhesion values of pressure-sensitive products without a need to resort to actual modification of the polymeric constituents of the pressure-sensitive adhesive.

10 The present invention provides a composition which comprises a pressure-sensitive adhesive and, homogenously dispersed therein, non-leaving metallic flakes.

The present invention also provides, in one embodiment, a pressure-sensitive product which comprises:

(a) a substantially transparent plastic film; and attached thereto

(b) a layer of pressure-sensitive adhesive containing homogenously dispersed non-leaving metallic flakes.

15 The present invention further provides, in another embodiment, a pressure-sensitive product which comprises:

(a) a plastic film and attached thereto optionally via an intermediate backing layer.

(b) a layer of pressure-sensitive adhesive containing homogenously dispersed non-leaving metallic flakes.

20 Furthermore, the present invention relates to a process for the production of such products and to the products produced thereby.

In the products according to the present invention, an optional release liner may be attached to the surface of the adhesive layer (b).

Referring to the accompanying drawings,

30 *Figure 1* is an enlarged cross-sectional view of one type of pressure-sensitive product in accordance with the present invention; and

Figure 2 is an enlarged cross-sectional view, partially broken, of another pressure-sensitive product in accordance with the present invention which contains a backing material between the adhesive layer and the film.

In *Figure 1*, one preferred embodiment of the present invention is shown in cross-section, wherein a plastic film 1 has a layer of pressure-sensitive adhesive 2, containing non-leaving metallic flakes 3, laminated to one of its sides. A release liner 4 covers the side of the adhesive layer 2 which is opposite to the side attached to film 1 in order to protect the adhesive from contamination from dirt or other solid or liquid contaminants.

The plastic film 1 may be a conventional, flexible film known to persons skilled in the art of fabricating pressure-sensitive products. If desired, it may be a "substantially transparent" film if a decorative product is desired. The term "substantially transparent" as used herein is intended to refer to those plastic films which are transparent enough to allow a viewer to perceive the decorative effect generated by the metallic flakes 3 in the adhesive 2. The film thickness in either a decorative or non-decorative product will generally range from 0.05 to 0.5 mm, (from 2 to 20 mils). The film may be a homo- or co-polymer of vinyl chloride, the preferred film material, or, for example, a polyester resin, a cellulose resin, an acrylic resin, a urethane resin, a polyolefin resin or a compatible blend thereof. Films of this type are well known and have been used in the past in forming pressure-sensitive products which differ in construction from the product shown in *Figure 1*.

The layer of pressure-sensitive adhesive 2 which is used in the present laminate may also be formed using the conventional pressure-sensitive adhesives which are known in the art. Such an adhesive may be based upon an elastomeric adhesive material, such as: (1) natural rubber; (2) synthetic rubbers, for example, styrene-butadiene copolymers, polyisobutylene, butadiene-acrylonitrile copolymers, polychloroprene and polyisoprene; (3) acrylic copolymers containing at least 50 percent, by weight, of a C₄-C₁₂ alkyl acrylate ester together with a hardening comonomer, for example vinyl acetate, styrene, methyl methacrylate, methyl acrylate, ethyl acrylate, ethyl methacrylate and vinyl chloride; and (4) polymers of alkyl vinyl ethers, for example polymethyl vinyl ether and polyethyl vinyl ether. Acrylic pressure-sensitive adhesives are most preferred since they have the best balance of adhesive and cohesive properties for the present laminate. The thickness of the adhesive layer 2 will generally be from 0.013 to 0.05 mm, (from 0.5 to 2.0 mils).

The pressure-sensitive adhesive layer 2 contains an effective amount of non-leaving metallic flakes 3 surprisingly to increase the adhesion of the product after it has been bonded to a desired substrate by the application of pressure and, in the case of a product containing a "substantially transparent" film 1, also to produce a decorative effect in the product by being visible through the plastic film 1. These flakes 3 are homogeneously dispersed throughout the adhesive layer 2 by means of conventional mixing techniques.

The term "non-leaving metallic flakes" is well known in the art and such products may be formed by means well known to persons skilled in the art. For the purposes of the present invention, non-leaving aluminium flakes are preferred.

(14)

The aluminium flake which may be employed in the preparation of the present laminate may be prepared in several ways. The most common means of obtaining aluminium flake is to atomize molten aluminium and subsequently grind it or hammer it in the presence of various lubricants. If the lubricant is a long chain saturated fatty acid, the prepared flake is not easily wetted by a resinous adhesive vehicle and tends to float 5 to the surface subsequent to application, giving a more or less continuous layer of aluminium flakes. This phenomenon is called "leafing". Such leafing flakes are not suitable for use in accordance with the present invention. It is therefore desirable to remove the long chain fatty acid or to grind or hammer the atomized aluminium in the presence of other materials, such as short chain fatty acids. The thus-produced flakes are more easily wetted by the resinous adhesive vehicle and tend to become randomly dispersed, rather than to 10 float. They are, therefore, less likely to layer at the surface during the curing or drying of the adhesive.

The aluminium flake may, if desired, be subjected to various treatments which impart a gloss or polish thereto. These polished aluminium flakes may then be utilized when a highly reflective decorative effect is to be generated.

These non-leafing aluminium flake particles are plate-like in appearance, but have a ragged edge which 15 follows no geometrical design. The flatness or plate-like appearance of the flake is generally determined by the method by which the flakes were prepared. Hammered flakes, for example, tend to be flatter than those that are ball-milled. However, both types may be used in accordance with the present invention. For a more complete description of non-leafing aluminium flake and the methods of preparation of such flakes, reference may be made to "Organic Coating Technology" by Payne, Vol. II, 1961.

20 The particle size of the flakes should generally be predominantly from 100 to 200 mesh (U.S. Standard Sieve) although a certain proportion may be above 100 mesh and up to 60 mesh. A predominant amount, preferably about 80 percent of the flakes, should generally be from 200 to 60 mesh. Excellent compositions may be made using flake having as much as 15 percent below 325 mesh in size which aids suspension, although as a substantial number of the particles approach a smaller size (below 325 mesh), there may be an 25 insufficient number of large planar surfaces provided in the adhesive to give the desired degree of opacity needed when a decorative product is desired. One suitable non-leafing aluminium flake which is commercially available from Alcan Metal Powder, Inc. is known under the designation "Grade MD-3100".

The aluminium flake particles are generally employed in amounts of at least 5%, by weight, based on the adhesive layer, generally with a maximum of 20%, by weight. Optimum effects have been obtained when 30 about 12%, by weight, of flake is present. Use of too little flake will not give the desired effects, whereas amounts higher than described above will weaken the adhesive/substrate bond. It is more preferred that from 10 to 15%, by weight, of flake be used.

If desired, a tinting pigment may be added to the flake-containing adhesive composition or to the flakes themselves prior to the incorporation thereof in the adhesive layer when a decorative product is desired.

35 Examples of pigments which may be used include:

phthalocyanine green, phthalocyanine blue, indanthrene yellow, burnt sienna, indo orange, phthalocyanine blue green tone, carbon black, phthalocyanine blue red shade, quinacridone red and hydrated iron oxide.

If the product of the present invention illustrated in Figure 1 is to be manufactured at one location and used at another location, a release liner 4 is preferably affixed to the side of the pressure-sensitive layer 2 which is 40 furthest removed from film 1. The release liner 4 may be one of the release liners known to those skilled in the art, for example removable, water-soluble protective coatings. One preferred liner material is silicone-coated release paper having a thickness of from 0.05 to 0.3 mm. Of course, if the film/adhesive composite is to be manufactured and applied to a desired substrate at the same manufacturing location, a release liner may not be needed.

45 The laminate according to the present invention may be formed by the conventional techniques used to form pressure-sensitive films. In a preferred embodiment, when a release liner, such as release paper, is to be used, the adhesive liner, such as release paper, is to be used, the adhesive formulation in solvent form, containing the non-leafing metallic flakes, is applied to the release liner and the adhesive/release liner composite is then heated to dry the adhesive layer. This combination is laminated to the plastic film to form 50 the final product. In those few applications wherein the product is not intended to have a release liner, the adhesive/metallic flake composite may be cast onto the vinyl film and the adhesive layer in the resulting laminate may then be dried.

Another embodiment of the present invention is shown in Figure 2 wherein a backed pressure-sensitive product is shown. Element Nos. 11, 16, 17 and 19 in Figure 2 correspond to Element Nos. 1, 2, 3 and 4, 55 respectively in Figure 1. The backing material 13, which may comprise synthetic fibres 15 needle punched into a thin layer of foam 14, is the added component in this article and may be attached to the film 11 by a suitable adhesive layer 12, if desired.

The backing 13 may be selected from a wide variety of conventional backing materials, such as non-woven, woven, knit or composite materials (e.g. non-woven/foam combinations, non-woven/knit combinations, non-Woven/woven/knit combinations) to provide a sufficiently thick layer for the intended purpose. For 60 example, in Figure 2, the backing (which is shown in broken form) will aid in camouflaging any surface irregularities that may exist on the substrate to which the composite article is attached. If the intended use of the product is as an exterior roofing material for motor vehicles, for example, its weight will range from 68 to 680 gm./m.², and its density from 32 to 320 kg./m.³. The thickness of such a backing and film composite will 65 generally range from 0.5 to 5.1 mm., preferably from 1.4 to 2.2 mm.

The backing material 13 shown in Figure 2 is a commercially available backing of a non-woven fabric of synthetic fibres 15 which have been needle punched into a thin layer 14 of foam. Some preferred examples of fibres which may be used in such a backing include: polypropylene, rayon, polyester, acrylonitrile, nylon and polyphenylene sulphide or mixtures thereof. Suitable foam materials which may be used include urethane and polyvinyl chloride foams. The denier of such fibres in this type of backing may range from 1 to 20. "Denier" is defined as a unit of fineness equal to the fineness of a yarn weighing one gram for each 9000 metres. The foam in such a backing may have a thickness of from 0.25 to 2.5 mm.

The backing 13, if intended for outdoor use, may be made water-repellent by treatment with an effective amount of at least one conventional water-repellent agent. Fluorocarbon emulsions, which are available as "Zepel" (Registered Trade Mark) (from E.I. DuPont de Nemours Co.) or "Scotchguard" from 3M Co.), are representative types of materials which may be used to confer such water-repellency. In order to treat the selected type of backing material, it may, for example, be placed in a fluorocarbon bath, dried and the resulting product may be cured. The product that results from either this or alternative conventional treating procedures will resist being wetted by water, oil and other contaminants. Further information on how backing 13 might be rendered water-repellent may be ascertained from "Waterproofing and Water Repellency", Kirk-Othmer Encyclopedia of Chemical Technology, 2nd Edition, Vol. 22, pp. 135-156 (1970).

If desired, one or more corrosion inhibitors may be used to treat the backing 13, if the article is to have exterior uses, in order to confer or enhance corrosion protection on the article/substrate combination. The corrosion inhibitor may be applied in conjunction with the aforementioned water-repellent agent. For example, when a fluorocarbon bath treatment is employed, such known corrosion inhibitors as the nitrites, phosphates, chromates and sulphates might be present in the bath to act as a corrosion inhibitor when the article is subjected to exterior use in the final article/substrate combination that will be produced. One corrosion inhibitor which is preferred for economic reasons is sodium nitrite. Further information on how the backing 13 might be treated to render the article/substrate combination corrosion resistant may be ascertained from "Corrosion Inhibitors", Kirk-Othmer Encyclopedia of Chemical Technology, 2nd Edition, Vol. 6, pp. 317-346 (1965).

The composite article of film 11 and backing 13 which is known may be formed by a number of well-known processes, such as direct calender lamination or extrusion coating, post-lamination or casting.

Direct calender lamination or extrusion coating produce a composite article without the use of adhesive layer 12. The plastic film 11 acts as the adhesive because it is brought in contact with the backing 13 shortly after formation while the film 11 is still hot and sticky.

Post-lamination is another well known method and differs from the extrusion coating or direct calender lamination techniques in that the plastic film 11 is not usually formed just prior to the bonding step and an adhesive layer 12 is applied directly by known coating techniques (e.g. gravure cylinder, knife, roller or reverse roller) to the film before the film is contacted with the backing 13. A roll or pre-formed film is generally the source of the film and a thin layer of adhesive is applied to the side of the film which is to be bonded to the backing. The adhesive is applied by a suitable means usually in an amount of from 34 to 170 gm./m.² depending upon the adhesive. The coated film is then contacted with the backing to form the known composite article.

Adhesives suitable for use in the post-lamination technique are well known in the art. Some examples include plastisol, epoxy, acrylic and urethane adhesives. Plastisols are one of the best known materials. These are dispersions of finely divided polymeric materials in non-volatile organic liquids and low melting solids, generally referred to as plasticizers. Suitable plasticizers include phthalate, adipate and sebacate esters and polyols such as ethylene glycol and derivatives thereof. A typical plastisol composition is 50 parts of polymer material and 50 parts of plasticizer which forms a paste as a result of solvation of the resin particles by the plasticizer. This paste gels and fuses when heated to about 300°F. If a volatile solvent is included in the plastisol, then the adhesive is generally referred to as an organosol, which also is suitable for use in accordance with the present invention.

In order to form the final pressure-sensitive, composite article according to the present invention, the exposed side of backing 13 is coated with a substantially continuous layer 16 of a suitable tacky, pressure-sensitive adhesive comprising adhesive 18 and metallic flakes 17, such as described above in connection with Element No. 2 in Figure 1.

The product shown in Figure 2 may find utility without the presence of release liner 19, for example, if it is used shortly after application of the adhesive layer and care is taken to avoid contamination of the exposed surface of the adhesive layer 16 with foreign matter. If this embodiment of the present invention is desired, the adhesive in the form of a viscous fluid may be cast onto the backing 13 and dried by the application of heat. In most end uses however, the composite article will be manufactured with a suitable release liner 19 by a manufacturer and the product will be sent to a customer for use.

The release liner 19 may be one of the release liners known to those skilled in the art. One preferred liner material is silicone-coated release paper having a thickness of from 0.05 to 0.25 mm. Polyolefinic (e.g. ethylene), ethylene/vinyl acetate copolymer, ethylene/acrylic copolymer and ethylene/chlorinated polyethylene blends which have been suitably release coated, e.g. with a silicone release agent, may also be employed as release liners.

A preferred process for affixing the film 11/backing 13 combination and the pressure sensitive adhesive layer 16 and release liner 19 comprises transfer lamination. In such a process, a solvent solution, an aqueous

dispersion or a hot melt of the adhesive layer 16 is applied to the release liner 19 and the resulting composite (except when a hot melt technique is used) is heated to dry the adhesive layer 16. The adhesive side of this adhesive/liner laminate is then laminated to the exposed surface of the backing 13, preferably at a heat lamination nip, to form the product shown in Figure 2. The use of a heated nip ensures good bonding between the adhesive layer 16 and the backing 13 which may have been treated with water-repellents and/or corrosion inhibitors.

Besides transfer lamination, the adhesive layer 16 may be applied to the backing by use of conventional direct application methods using either knife, roller, reverse roller or extrusion coating techniques provided a substantially continuous and uniform coating of pressure-sensitive adhesive results.

10 The following Examples illustrate the present invention:

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Example 1

This Example illustrates a procedure for forming a decorative pressure-sensitive product in accordance with the present invention.

15 A transparent, plasticized polyvinyl chloride (PVC) film was heat laminated to itself on a lamination machine by passing two layers of such film over a series of hot drums in order to achieve a heat seal temperature of about 149°C. The PVC film was simultaneously embossed on one side with a brushed silk pattern at the lamination point. The film that resulted had a thickness of about 0.2 mm.

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An adhesive formulation was then prepared which comprised 100 gm. of a 30%, by weight, solids acrylic pressure-sensitive adhesive polymer and 3 gm. of a non-leafing, finely divided aluminium flake. The adhesive that was used was a high molecular weight thermoplastic acrylic terpolymer available as "Durotak (Registered Trade Mark) 80. 1053" from National Starch and Chemical Corp. The aluminium flake that was used is commercially available from Alcan Metal Powder, Inc. as "Grade MD-3100".

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The adhesive and aluminium flake were mixed together for from 5 to 10 min. at high speed to effectively disperse the aluminium flake particles. The adhesive/aluminium flake composition was then coated onto silicone-coated release paper using a laboratory coating blade. Removal of the volatiles from this coated layer was accomplished by drying the layer in an oven for 2 minutes at 121°C. The dried adhesive thickness was about 0.03 mm.

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The resultant adhesive-coated release paper was then laminated to the unembossed side of the transparent PVC film by passing both the film and release paper through a laboratory nip roller at sufficient pressure to ensure intimate contact of the adhesive with the film. This PVC film/adhesive/release liner laminate was then conditioned for 16 hours at laboratory ambient conditions to allow the adhesive to form a strong bond to the film. The opacity and aesthetics of the laminate were judged to be satisfactory.

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Peel adhesion of the product was then determined by bonding 2.54 cm. strips of the PVC/adhesive laminate (after removal of the release liner) to a stainless steel panel using the standard Pressure Sensitive Tape Council 2.04 kg. roller. Peel adhesion at 180° was then measured after a 24 hour wetout of "dwell" period on a Thwing-Albert type tester. The value achieved was about 1.34 kg. per linear cm. Virtually all of the adhesive remained on the film thereby demonstrating both high adhesive strength to the film and high cohesive strength, the former being of more importance when the produce is to be used as a permanent decorative film on a desired substrate.

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A Control laminate, lacking the aluminium flake, yielded a peel adhesion of only 0.98 kg. per linear cm.

One advantage of the aluminium flake-containing laminate as compared to the control laminate was the greater ease of repositionability that it exhibited. The presence of the flake reduced the "quick grab" of the adhesive allowing for initial repositioning of the laminate.

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Example 2

In another experiment, two other leafing, rather than non-leafing, aluminium flakes ("Grade MD-2100" and "Grade MD-5100" from Alcan Metal Powder, Inc.) were tried in similar amounts, but they produced poor adhesion of the adhesive to the vinyl film when tested as above after 20 min. and 24 hr. dwell times on the substrate. The results of all tests are set forth in the Table which follows. All values are given in kg. per linear (abbreviated "l.") cm.

50

No.	Metallic Flakes	20 min. Dwell	24 hr. Dwell
1	None (control)	0.43	0.82
2	Non-leafing (1)	0.58	1.61
3	Leafing (2)	0.58	0.8
4	Leafing (3)	0.82	1.07

(1) "Grade MD-3100" from Alcan Metal Powder, Inc.

(2) "Grade MD-2100" from Alcan Metal Powder, Inc.

(3) "Grade MD-5100" from Alcan Metal Powder, Inc.

Each of the above samples had the following peel adhesions (in kg./1.cm.) after one minute: No. 1: 0.63; No. 2; 0.23; No. 3: 0.43; and No. 4; 0.52. No. 2, the product of the present invention, has the lowest initial tack, but the highest adhesion after 24 hr. dwell times.

After 20 minutes, then the film/adhesive laminates were peeled back from the panel, product Nos. 1 and 2 showed no transfer of adhesive to the plate from the film whereas products Nos. 3 and 4 showed transfer of adhesive. After 24 hours, product No. 1 showed no transfer, product No. 2 showed cohesive failure of the adhesive as illustrated by adhesive remaining on both film and panel and products Nos. 3 and 4 showed failure of the adhesive to the film as illustrated by most of the adhesive being left on the panel. In those end uses where the product is to be left permanently on the object, these observations are merely of interest as to how the internal strength of the adhesive (cohesive strength) and bond strength of adhesive to film and/or substrate (adhesive strength) are related. Adhesion strength would be unquestionably the most important property.

Example 3

Approximately 1835 metres of a 0.01 mm. transparent PVC film was heat laminated to itself and was simultaneously embossed on one side with a shallow brushed silk roll.

An adhesive formation was prepared by mixing 300 gm. of the acrylic adhesive used in Example 1, 15 gm. of ethyl acetate, 15 gm. of toluene and 12 gm. of the non-leaving aluminium flake used in Example 1. The mixing was performed at high speed on a Cowles type dissolver until the flake was completely dispersed in the adhesive solution. The viscosity of the formulation after mixing was about 2000 cps. (Brookfield viscosity, 25°C., No. 2 spindle, 20 rpm).

The adhesive formulation containing the aluminium flakes was then applied to 90 pound basis weight, bleached kraft, silicone-coated release paper using a reverse roll coater. A streak-free, uniform coating of adhesive on the release paper was produced and the adhesive coating was dried by passing the adhesive/release paper laminate through an oven at 60°C. for 40 seconds, followed by passage through a second oven at 132°C. for 45 seconds.

This adhesive/release paper laminate was then laminated to the unembossed side of the transparent PVC film as described in Example 1.

The resulting product was then tested against a control product that did not contain aluminium flakes in the adhesive using the test procedures described in Example 1. The adhesion at 180° was measured on stainless steel and lacquered panels using the same test method as described in Example 1. The results are given below in kilograms per linear centimetre:

Stainless Steel Adhesion

<u>Film</u>	<u>20 min. Dwell</u>	<u>24 hr. Dwell</u>
Present Film	0.75	1.84
Control	0.61	1.18

The 20 min. dwell readings showed no adhesive transfer from the film to the substrate, whereas both 24 hour dwell readings showed slight transfer using the same units given above:

Lacquer Painted Panel Adhesion

<u>Film</u>	<u>30 min. Dwell</u>	<u>24 hr. Dwell</u>
Present Film	1.0	2.04
Control	1.0	1.16

At 30 minutes no adhesive transfer was noted for the present film as compared to slight transfer for the control film. After 24 hours on the panels, both films showed cohesive failure as evidenced by transfer of substantial amounts of adhesive to the panel.

The films were also tested to determine the resistance by the adhesive to shrinkage of the vinyl film. This was evaluated by bonding a 2.54 cm. x 25.4 cm. adhesive-coated film cut in the direction of travel of the film in the laminator to an aluminium panel using a standard 2.04 kg. roller. The ends of the film were scored with a razor blade and after a 2 hour wet out period at room temperature the assembly was placed in a 121°C. oven for 30 minutes. Growth of the razor cut was measured using a magnifier after the film was cooled to room temperature. The present film had a mounted shrinkage of only 0.04 cm., whereas the control film had a shrinkage of 0.08 cm.

Example 4

This Example compares the adhesion of a film according to the present invention (Film A) to that exhibited by the type of decorative film known in the prior art (Film B).

Film A was a 0.2 mm. transparent polyvinyl chloride film that had been prepared by laminating two 1.0 mm. films together, followed by application of adhesive containing non-leafing aluminium flakes as described in Example 1.

Film B was a 0.174 mm. transparent polyvinyl chloride film made by laminating a 0.075 mm. transparent film to the vinyl side of a 0.10 mm. film that had previously been printed on one side with a silver ink for decorative purposes. The same adhesive used in making Film A was applied to the silver ink side of the 0.175 mm. film without the presence of the aluminium flakes.

Both films were applied to lacquer painted stainless steel panels and the peel adhesion was measured after the two films had been subjected to various environmental conditions for various lengths of time. The procedure described in Example 1 was employed to mount the films and measure the peel adhesion. The Table that is given below sets forth the results that were obtained in kg./linear centimetre:

15	Condition	Time	Adhesion of Film		15
			A	B	
	Room Temperature	72 hrs.	1.88	0.98	
	70°C	7 days	1.84	1.16	
	70°C	30 days	1.66	0.95	
20	37.8°C) 100% Relative Humidity)	7 days	1.88	1.21	20

These data demonstrate the uniformly higher adhesion values for the present film as compared to the type of film known in the prior art.

Example 5

This Example illustrates the present invention in connection with a plastic film/adhesive laminate containing an opaque plastic film.

The laminate according to the present invention was first formed by initially forming the following adhesive formulation containing non-leafing aluminium flakes:

35	Ingredient	Parts by Weight	35
	Acrylic Pressure-Sensitive Adhesive Solution*	100	
	Ethyl acetate	25	
	Isopropyl alcohol	25	
40	Non-leafing aluminium flakes**	2	40

* "GELVA RA 1753" from Monsanto Co.

** "Grade MD-3100" from Alcan Metal Powder, Inc.

The adhesive solution was knife coated onto release paper, dried at 121°C. and the release paper/adhesive layer was laminated to an opaque polyvinyl chloride film. The adhesive layer was 0.02 mm. thick.

A similar process was used to form the Control film, with the exception that the non-leafing aluminium flakes were not used.

The two products were tested for 180° peel adhesion to stainless steel (as described in Example 1) after having been allowed to remain for 1 minute, 10 minutes and 24 hours, respectively, on the substrate. The following values (in kg./linear cm.) were obtained with higher values being more desirable:

55	Peel Adhesion After	Control	Present Film	55
	1 min applied	—	0.51	
	10 min. applied	0.41	0.60	
	24 hrs. applied	0.60	1.14	

The static creep resistance of each film was also measured. This is expressed as the time (in minutes) for the film/adhesive laminate to fall from the stainless steel substrate when a 1000 gm. load is attached to a sample having an area of 1.61 cm². Higher numbers are more desirable. The present film had a value of 2493 min., whereas the control film only had a value of 520.1 min.

The 180° peel adhesion and static creep resistance were also measured after both laminates had been aged for 7 days at 70°C. The following results were obtained. The peel adhesion values are given in units of kg./linear cm. and the creep resistance value in minutes:

	<u>Peel Adhesion at 180° After</u>	<u>Control</u>	<u>Present Film</u>	
5	1 min. applied	—	0.04	5
	10 min. applied	0.24	0.06	
	24 hrs. applied	0.22	0.09	
10				10
	<u>Peel Adhesion at 180° After</u>	<u>Control</u>	<u>Present Film</u>	
15	Static Creep Resistance	47.7	342	15

CLAIMS

1. A composition which comprises a pressure-sensitive adhesive and, homogenously dispersed therein, non-leafing metallic flakes. 20
2. A composition as claimed in claim 1 in which the metallic flakes are aluminium flakes.
3. A composition as claimed in claim 1 or claim 2 in which the pressure-sensitive adhesive is an acrylic pressure-sensitive adhesive.
4. A composition as claimed in any of claims 1 to 3 in which from 5 to 20%, by weight, based on the adhesive of metallic flakes are present. 25
5. A composition as claimed in claim 4 in which from 10 to 15%, by weight, based on the adhesive of metallic flakes are present.
6. A composition as claimed in claim 1 substantially as herein described.
7. A composition as claimed in claim 1 substantially as herein described with reference to any one of the Examples. 30
8. A pressure-sensitive product which comprises:
 - (a) a substantially transparent plastic film; and, attached thereto
 - (b) a layer of pressure-sensitive adhesive containing homogeneously dispersed non-leafing metallic flakes. 35
9. A pressure-sensitive product which comprises:
 - (a) a plastic film and, attached thereto, optionally via an intermediate backing layer,
 - (b) a layer of pressure-sensitive adhesive containing homogeneously dispersed non-leafing metallic flakes. 40
10. A product as claimed in claim 8 or claim 9 in which the plastic film (a) is a vinyl film.
11. A product as claimed in any of claims 8 to 10 in which the pressure-sensitive adhesive (b) is an acrylic pressure-sensitive adhesive. 45
12. A product as claimed in any of claims 8 to 11 in which the metallic flakes are aluminium flakes.
13. A product as claimed in any of claims 8 to 12 in which the plastic film (a) has a thickness of from 0.05 to 0.5 mm.
14. A product as claimed in any of claims 8 to 13 in which the thickness of the pressure-sensitive adhesive (b) is from 0.013 to 0.05 mm. 50
15. A product as claimed in any of claims 8 to 14 in which the amount of metallic flakes in the adhesive is from 5 to 20%, by weight, based on the adhesive layer.
16. A product as claimed in any of claims 8 to 15 which further comprises a release liner.
17. A product as claimed in claim 8 or claim 9 substantially as herein described. 55
18. A product as claimed in claim 8 or claim 9 substantially as herein described with reference to any one of the Examples.
19. A process for the production of a product as claimed in claim 8 or claim 9 substantially as herein described.
20. A product when produced by a process as claimed in claim 19.



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	<u>Peel Adhesion at 180° After</u>	<u>Control</u>	<u>Present Film</u>	
5	1 min. applied	—	0.04	
	10 min. applied	0.24	0.06	5
	24 hrs. applied	0.22	0.09	

	<u>Peel Adhesion at 180° After</u>	<u>Control</u>	<u>Present Film</u>	
10				10
	Static Creep Resistance	47.7	342	

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2. A composition as claimed in claim 1 in which the metallic flakes are aluminium flakes.
3. A composition as claimed in claim 1 or claim 2 in which the pressure-sensitive adhesive is an acrylic pressure-sensitive adhesive.
4. A composition as claimed in any of claims 1 to 3 in which from 5 to 20%, by weight, based on the adhesive of metallic flakes are present. 25
5. A composition as claimed in claim 4 in which from 10 to 15%, by weight, based on the adhesive of metallic flakes are present.
6. A composition as claimed in claim 1 substantially as herein described.
7. A composition as claimed in claim 1 substantially as herein described with reference to any one of the Examples. 30
8. A pressure-sensitive product which comprises:
 - (a) a substantially transparent plastic film; and, attached thereto
 - (b) a layer of pressure-sensitive adhesive containing homogeneously dispersed non-leaving metallic flakes.
9. A pressure-sensitive product which comprises:
 - (a) a plastic film and, attached thereto, optionally via an intermediate backing layer,
 - (b) a layer of pressure-sensitive adhesive containing homogeneously dispersed non-leaving metallic flakes. 35
10. A product as claimed in claim 8 or claim 9 in which the plastic film (a) is a vinyl film.
11. A product as claimed in any of claims 8 to 10 in which the pressure-sensitive adhesive (b) is an acrylic pressure-sensitive adhesive. 40
12. A product as claimed in any of claims 8 to 11 in which the metallic flakes are aluminium flakes.
13. A product as claimed in any of claims 8 to 12 in which the plastic film (a) has a thickness of from 0.05 to 0.5 mm.
14. A product as claimed in any of claims 8 to 13 in which the thickness of the pressure-sensitive adhesive (b) is from 0.013 to 0.05 mm. 45
15. A product as claimed in any of claims 8 to 14 in which the amount of metallic flakes in the adhesive is from 5 to 20%, by weight, based on the adhesive layer.
16. A product as claimed in any of claims 8 to 15 which further comprises a release liner.
17. A product as claimed in claim 8 or claim 9 substantially as herein described. 50
18. A product as claimed in claim 8 or claim 9 substantially as herein described with reference to any one of the Examples.
19. A process for the production of a product as claimed in claim 8 or claim 9 substantially as herein described.
20. A product when produced by a process as claimed in claim 19. 55