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PHOTOSENSITIVE PRODUCTS FOR PREPARING GRAVURE RESISTS

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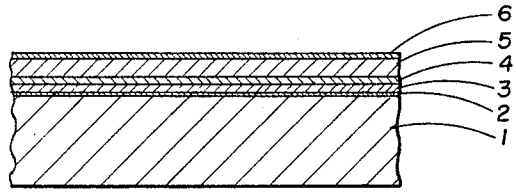


FIG. 1

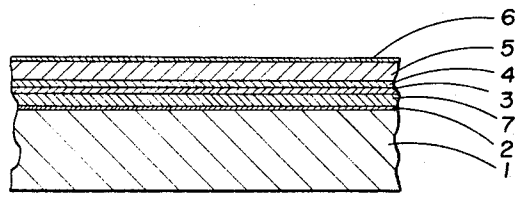


FIG. 2

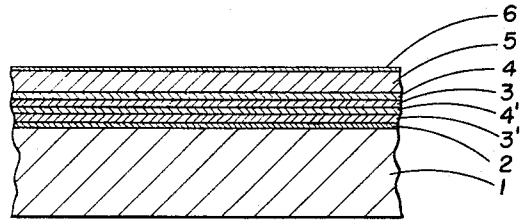


FIG. 3

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PHOTOSENSITIVE PRODUCTS FOR PREPARING GRAVURE RESISTS

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ABSTRACT OF THE DISCLOSURE

A gravure resist film comprises a dimensionally, stable, temporary, subbed film support containing thereon (1) a water-impermeable layer, (2) a stripping layer comprising a hydrosol of an acrylic copolymer as for example a mixture of gelatin with a copolymer of ethyl acrylate and acrylonitrile, (3) an antihalation layer composed of a gelatin layer containing a dye or Carey Lea silver and (4) an unhardened gelatin-silver halide emulsion layer.

This invention relates to an improvement of the light-sensitive element described and claimed in French Patent No. 1,282,219.

That patent describes a photosensitive product useful in the preparation of gravure resist and comprises a temporary support of good dimensional stability which can be easily stripped without the aid of an organic solvent. The photo sensitive product consists of a customary film support such as of cellulose acetate, polystryrene or polyethylene terephthalate overcoated with layers in the following order: (1) a subbing layer, (2) a stripping layer comprising a hydrosol of an acrylic copolymer as for example a mixture of gelatin with a copolymer of ethyl acrylate and acrylonitrile, (3) an antihalation layer composed of a gelatin layer containing a dye or Carey Lea silver and (4) an unhardened gelatin-silver halide emulsion layer. In use this product after exposure and tanning development is applied onto the wet surface of a gravure cylinder, the emulsion being in contact with the cylinder. The cylinder is washed by sprinkling it with water at 5–15° C. and the film support is carefully stripped from the cylinder permitting the emulsion to adhere to the cylinder following which the emulsion is washed in water of 38–48° C. to remove the non-tanned areas and to eliminate layers which might remain on the emulsion layer following the stripping. The cylinder is then ready for the etching procedure.

The light sensitive element described in that patent yields excellent results and constitutes an improvement over previous products. However its use ordinarily necessitates cold water washing and unless special care is taken blisters might form on the emulsion layer.

One object of our invention is to provide a photographic product for gravure purposes which does not necessitate the use of cold water or organic solvents in the stripping and removing operations. Another object of our invention is to provide a photosensitive product of a type whereby resists without blisters are readily obtained without the use of special precautions. A further object of our invention is to provide a photographic product for gravure purposes which is sufficiently sensitive that it does not necessitate exposure to intense light sources such as a carbon arc in obtaining useful images. Other objects of our invention will appear herein.

In its broadest aspects our invention relates to adding to the photosensitive product described in French Patent No. 1,282,219 one or several layers which serve to prevent water from the gravure cylinder, during application of the photosensitive product to the cylinder, from penetrating across the stripping layer and moistening the interface of the support and the stripping layer. Such moisture

in products of this nature has the adverse effect of increasing the stripping resistance of the product. For best results the stripping strength of the support should be lower than the adhesive strength of the emulsion to copper and the purpose of the present invention is to provide a product having that lower stripping strength whereby the formation of blisters is avoided upon stripping the support from the emulsion. Accordingly, in the preferred form of the invention a coating impermeable to water is placed between the support and the hydrosol layer of a product as described in French Patent No. 1,282,219. In a modification of the invention, in addition to the layers forming the product as described in said French patent a second hydrosol layer and a second gelatin layer containing an antihalation dye are placed in that order between the support and the primary hydrosol layer. If desired, one can advantageously use the supplementary underlying gelatin layer not in contact with the emulsion to introduce a magenta antihalation dye useful during yellow exposures as any tendency of the dye to diffuse in the emulsion is avoided because of its lack of its proximity to the emulsion layer in the composition. If desired, an antiabrasion layer may be applied over the photographic emulsion layer.

In the attached illustrative drawings FIG. 1 represents a greatly enlarged cross section of the photosensitive product of the type described in French Patent No. 1,282,219.

FIG. 2 represents a cross section of a product in accordance with the preferred embodiment of the invention, and

FIG. 3 represents in similar fashion a cross section of a photographic product according to a modification of the invention.

FIG. 1 illustrates a product comprising a support **1** carrying successively and in superimposed relationship subbing layer **2**, hydrosol layer **3**, antihalation layer **4**, silver halide emulsion layer **5**, and antiabrasion layer **6**.

Illustrated by FIG. 2 is a photosensitive product in accordance to the preferred embodiment of the invention in which a water impermeable coating **7** is placed between subbing layer **2** and hydrosol layer **3**.

FIG. 3 illustrates a photographic product of a modified structure in that layers **3'** and **4'** constituting respectively a hydrosol layer and an antihalation layer are placed between layers **2** and **3**.

The water impermeable layer may be a layer of a polymer or a synthetic resin applied either from an organic solvent solution thereof or in the case of a synthetic resin which is insoluble or little soluble in organic solvents in the form of an aqueous dispersion. Some synthetic resins useful in this connection and which are applicable by application from organic solvents are polyisobutyl methacrylate, polyvinyl butyral, polyvinyl chloride-acetate, poly condensation products of bisphenol and maleic or fumaric anhydride, polyvinylidene, acrylonitrile copolymers and the like. As illustrative of synthetic resins which are better applied in the form of their aqueous dispersions are synthetic resins such as styrene-butadiene resins, vinylidene chloride-acrylonitrile copolymers, modified polyvinyl chloride, the terpolymers of methacrylate vinyl chloride and vinyl isobutyl ether, polyacrylonitrile, etc.

The thickness of the layers represented in FIGS. 1, 2 and 3 are on an approximate scale. For example, the support may have a thickness of about 100 microns, the antihalation layers and the hydrosol layers a thickness of about 5 microns, the emulsion layer a thickness of about 15–20 microns, the antiabrasion layer of about one micron and the water impermeable coating **7** a thickness of 5–8 microns. It is apparent that some latitude should be allowed in the compounding of these various layers to form the products described.

By the present invention moisture from the gravure cylinder during the procedure for forming the gravure resist is prevented from penetrating the stripping layer and thus the stripping operation is facilitated. We have found that the resistance to stripping of the product is much greater in the moist state than in the dry state and an increase of this resistance can be the cause of formation of blisters on the emulsion. By means of the procedures described in accordance with the invention and because of the difference between the stripping when the stripping layer is in the dry state and when moist, difficulties in transfer to the gravure cylinder are greatly reduced or even eliminated. Also tolerance to water of the product is greatly increased.

The support for the photosensitive product can be any film support customarily used such as cellulose acetate, cellulose acetate butyrate, polystyrene and the like, wherein the thickness usually runs from 0.125–0.375 mm. In case of polyester supports even thinner sheets may be used. Before applying the various layers to a support, it may be desirable to treat the surface of the support with trichloroacetic acid prior to coating with the various layers which facilitates the adherence of the layers thereto. The polyester support layer may if desired have a subbing layer such as of the type shown in French Patent No. 1,278,273 or that shown in French Patent No. 1,064,786, the subbing layer of the latter being generally overcoated with a gelatin layer.

In preparing products in accordance with the invention, an impermeable coating can be applied from an aqueous suspension of the polymer rather than from its solution in organic solvent particularly if the polymer is insoluble or little soluble in those solvents. This method of coating avoids the use of volatile and inflammable solvents. This layer is desirably of a thickness between 4 microns and 30 microns, preferably between 5 and 10 microns.

The hydrosol layers are of the type described in the patent referred to. They are obtained for instance starting with an aqueous composition composed of 10–15 parts of gelatin and 85–90 parts dry weight of copolymer latex in which a surface active agent is also present. The other layers such as the antihalation layer, emulsion layer and antiabrasion layer are of the type described in French Patent No. 1,282,219 in which details are provided concerning the composition of those layers. These layers may be applied by one of the well-known techniques such as hopper, scraper, bead coating, transfer by immersed rollers or many of the other procedures well known in the art for the application of layers in preparing photographic products. The product after photographic exposure is developed in a tanning developer, fixed, washed and dried. After exposure and development the film is applied to a moist surface of a gravure cylinder, the emulsion surface (or the antiabrasion layer if the product had one) being in contact with the cylinder, which is then washed with luke warm water for 3 or 4 minutes and thus remains on the cylinder as an image composed of silver and differentially tanned gelatin. In the use of the modified product in accordance with the invention, the emulsion is applied to the cylinder as described but the two thin hydrosol layers can be easily separated by treating with luke warm water which serves to remove the non-tanned gelatin, this operation being after the support had been stripped from the gelatin layer.

It may be seen that the stripping of the film temporary support from the emulsion layer is easily achieved without the formation of blisters.

The following examples illustrate the invention:

Example 1

A solution of Lucite 45 (isobutyl polymethacrylate) was dissolved in 960 grams of methyl ethyl ketone and was applied respectively to (1) a Mylar support of 0.12 mm. thickness, (2) a Mylar support which had been pretreated with trichloroacetic acid in solution in methyl ethyl ketone and then dried and (3) an Estar support of 0.10 mm. thickness having a subbing layer of a copolymer of vinylidene chloride methacrylate and itaconic acid. The Mylar and Estar supports were composed of poly-(ethylene glycol terephthalate). The coating of Lucite 45 was of 5 microns thickness. A layer of a hydrosol comprising a dispersion of 453 grams of gelatin in 7200 grams of hydrosol having about 40% solids content and comprising a copolymer of ethyl acrylate-acrylonitrile was then applied. Gelatin was then applied over this layer in an amount of 4 mg. per square decimeter, resulting in a gelatin layer having a thickness after drying of approximately 5 microns. There was then applied in succession, an antihalation layer of gelatin containing Carey Lea silver dispersion, a gelatin-silver halide emulsion layer and if desired an antiabrasion layer.

In order to evaluate the qualities of the resulting material the stripping force of a 1 cm. wide bit of the unexposed and dry material and of the material developed at the time of stripping, after wet transfer of the emulsion side onto the copper cylinder was measured with a dynamometer at an angle of 45°. The presence or absence of blisters resulting from stripping on the emulsion was also evaluated by a practical test on a larger sheet of film. The stripping force of the dry unexposed material and of the material developed at the time of stripping for the respective products were respectively 4 grams and 10–20 grams for the untreated Mylar support, 5 grams and 25–30 grams for the pretreated Mylar support and 2 grams and 15–20 grams for the Estar support. No formation of blisters was observed after the support had been stripped off.

After stripping the support from the element on the cylinder the non-tanned gelatin of the emulsion layer was removed with water of about 40° C. by simultaneously treating the antihalation layer and the hydrosol layer. The gravure cylinder is then ready for etching. If Example 1 is repeated using a product without the impermeable layer of the polyisopropyl methacrylate, the stripping force when dry and after transfer to the moist copper cylinder are respectively 5–6 grams and 70–80 grams. In practice, with the latter product stripping can be carried out only after treating with cold water (4–5° C.). The products are also prone to the formation of blisters upon stripping and removal of the non-tanned portions of the image layer with warm water.

Example 2

30 grams of polyvinyl butyral were dissolved in 490 grams of ethanol and 480 grams symmetrical dichloroethane. This solution was then applied on film support at a thickness of 5–6.5 microns. The various other layers to form the photosensitive element of the invention were also applied in the same manner as described in Example 1. The dry stripping forces and the stripping forces after wet transfer with respectively 5 grams and 10–15 grams on raw Mylar film base, 5 grams and 50–75 grams on pretreated Mylar film base, 5 grams and 15–25 grams on subbed Estar film base. The removal of the coating composition which still adheres to the emulsion on the copper cylinder was carried out with water at 40° C. simultaneously with the removal of the resin coat during the wash-off operation. Blister formation was nil except in the case of the pretreated Mylar film base.

Example 3

40 grams of polyvinyl acetate chloride modified by treating with maleic acid was dissolved in a solvent composed of 480 grams of cyclohexanone. This solution was then coatd onto raw Mylar film base and subbed Estar film base to give a coating having a thickness of 4–5 microns upon drying. The product was incorporated and was applied to a copper cylinder in the same manner as described in Example 1. The dry stripping forces and those after

wet transfer were determined and were found to be 10 grams and 30 grams in the case of the raw Mylar film base and 8 grams and 25 grams in the case of the subbed Estar film base. 1 or 2 blisters were formed on the emulsion which was considered to be due to defects of the coating composition.

Example 4

30 grams of a high molecular weight polyester (polyethylene glycol terephthalate) was dissolved in a solvent of 700 grams of methylene chloride and 270 grams of toluene. The composition was applied onto a subbed Estar film base to give a coating having a thickness of 4–5 microns upon drying. The product was completed as described in Example 1, applied to a copper cylinder and subjected to stripping, all as described in that example. The dry stripping forces and those after wet transfer were determined to be respectively 25 grams and 75–100 grams. Blisters were formed on the emulsion because the resistance to wet stripping was high in this particular case.

Example 5

On raw and on pretreated Mylar film base there was applied a coating of latex composed of a styrene-butadiene copolymer having a high styrene content. The dry thickness of the coating applied was 7–8 microns. Upon drying the various coatings which constitute the complete product were applied in a similar manner as described in Example 1. The product was then applied to a copper cylinder in the manner described there and the film support was stripped from the emulsion layer upon the copper cylinder. The material on the copper cylinder was then given the prescribed washing. It was determined that the dry stripping forces and those upon wet-transfer, in the case of these two products were respectively 10 grams and 2–3 grams on the raw Mylar film base and 6 grams and 2–3 grams in the case of the pretreated Mylar film base. No blisters were formed upon stripping but the polymer coating had to be removed from the emulsion layer by spraying with denaturated alcohol followed by washing off with water at 40–45° C.

Example 6

In a procedure identical with the preceding, there was coated a latex composed of a plasticized vinylidene chloride-acrylonitrile copolymer containing 50% of dry material. On both the raw Mylar film base and a pretreated Mylar film base the dry thickness of the layer was 8–12 microns. The dry stripping forces and those upon wet transfer were determined and were respectively as follows: 10 grams and 2–3 grams in the case of raw Mylar film base, 70 grams and 10–25 grams in the case of pretreated Mylar film base. No stripping blisters were formed on the emulsion. The polymer coating that still adhered to the emulsion was removed with spraying with denaturated alcohol followed by washing off with water at 35–40° C.

Example 7

In a procedure identical with that of Example 5 an aqueous dispersion of a modified unplasticized polyvinyl chloride polymer having 50% dry material was coated onto raw Mylar film base and pretreated Mylar film base, the coatings having dry thickness of 5–6 microns. The dry stripping forces and those upon wet transfer were determined and were respectively as follows: 20 grams and 263 grams on raw Mylar film base, 5 grams and 2–3 grams of pretreated Mylar film base. No blisters were formed on the emulsion upon stripping. The polymer coating still adhering to the emulsion layer was removed by spraying with denaturated alcohol followed by washing off with water at 40° C.

Example 8

Using the same procedure as the preceding, a pretreated Mylar film base was coated with an aqueous dispersion of unplasticized polyacrylic ester containing 40% of dry material. The dry thickness of the coating was 4 microns. The dry adhesion forces and those upon wet transfer are respectively 170 grams and 25–30 grams. No blisters were formed on the emulsion upon stripping. The polymer coating that adhered to the emulsion layer after stripping was removed by spraying with denaturated alcohol followed by washing off with water at 40° C.

Example 9

An Estar base of 0.20 mm. thickness was coated with a latex composed of an aqueous dispersion of unplasticized modified polyvinyl chloride, the proportion of dry material being 50%. The dry thickness was 18 microns. After drying there was then applied the series of coatings as described in Example 1. The product was transferred to a wet copper cylinder and the film support was stripped off. The dry stripping forces and those upon wet transfer of this product were determined and were respectively 200 grams and 2–3 grams. No blisters were formed on the emulsion upon stripping. The polymer coating was removed from the emulsion layer by spraying with denaturated alcohol followed by washing-off with water at 50° C. which simultaneously removed the resin-coat, the Carey Lea silver and the unexposed emulsion areas.

Example 10

The preceding procedure was repeated except a latex composed of styrene-butadiene copolymer having 47% of dry material was applied. The dry thickness of the coating was about 30 microns. The dry stripping forces and those after water transfer were respectively about 200 grams and 20 grams. No blisters were formed on the emulsion upon stripping. The polymer coating was removed by spraying the emulsion layer with denaturated alcohol followed by wash-off with water at 40–45° C.

Using the same latex the various dilution rates gave similar results.

Example 11

The preceding procedure was followed except that a latex of an anionic aqueous dispersion of a terpolymer comprising ⅓ of methyl acrylate, ⅓ of vinyl chloride and ⅓ of vinylisobutilic ether, having 50% dry material was used. The dry thickness of the coating was 18 microns. The dry stripping forces and those upon wet transfer were respectively 12 grams and 3 grams. No blisters were formed on the emulsion. Upon stripping the polymer coating was removed from the emulsion layer by spraying with denaturated alcohol followed by washing-off with water 40–50° C.

Example 12

The procedure described in Example 9 was used except that the latex coating was comprised of a medium molecular weight polyacrylonitrile having about 40% dry material. The dry thickness of the coating was about 14 microns. The dry stripping forces and those upon wet transfer are respectively 5 grams and 15–20 grams. No blisters were formed on the emulsion upon stripping. The polymer coating could be removed dry by a slight pull after which a wash-off with water at 35° C. removes the resin coat, the Carey Lea silver layer and the unexposed emulsion areas.

Example 13

Example 9 was repeated except that the latex used was that of a styrene-butadiene copolymer having a high styrene content. The latex containing 40% of dry material. The dry thickness of the coating was 19 microns. The dry stripping forces and those upon wet transfer were respectively about 200 grams and 50–70 grams. The polymer coating was removed with the temporary support and a wash-off with water at 35° C. removed the remaining materials from the emulsion layer on the copper cylinder.

Example 14

Example 9 was repeated using as the latex an anionic aqueous dispersion (pH 7.5) of a styrene polymer plasticizer with butyl phthalate having 47% of dry material. The dry thickness of the coating was 5 microns. The dry stripping forces and those upon wet transfer were respectively 200 grams and 15–20 grams. The polymer coating was removed upon stripping off of the temporary support. A wash-off with water at 35° C. was then sufficient to clear the emulsion layer on the copper cylinder.

Example 15

A Mylar support having a gelatin subbing layer thereon of the type described in French Patent No. 1,278,073 was coated with a hydrosol of polyethyl acrylate-acrylonitrile copolymer and gelatin then with a Carey Lea silver antihalation coating and again with a hydrosol coat and a Carey Lea silver coating and finally with a silver halide emulsion and an antiabrasion layer. The product obtained was used in forming a gravure resist. It was determined that the dry stripping force and that after wet transfer was respectively 5 grams and 15–18 grams. No blisters were formed on the emulsion in the stripping operation.

Example 16

The preceding example was repeated and 4 samples were obtained each comprising a double Carey Lea silver and resin coat association. The dry stripping force and that after wet transfer of the 4 samples obtained were in each case respectively 4–5 grams and 5–6 grams. No blisters were formed on the emulsion after stripping.

Example 17

The procedure of Example 15 was repeated but the Carey Lea silver present in the gelatin sublayer of the association not in contact with the silver halide layer was replaced by an antihalation magenta dye. Four samples were prepared. The dry stripping force and that after wet transfer of the four samples were in each case respectively 4–6 grams and 7–8 grams. No blisters were formed on the emulsion after stripping. The same results are obtained using normally subbed triacetate supports and Estar supports.

The invention has been described in considerable detail with particular reference to certain preferred embodiments thereof, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention as described hereinabove, and as defined in the appended claims.

We claim:

1. A gravure resist film comprising dimensionally, stable, temporary, subbed film support containing thereon (1) a water impermeable layer (2) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (3) an antihalation layer and (4) a hardener-free light sensitive gelatin-silver halide photographic emulsion all in that order.

2. A gravure resist film comprising dimensionally stable, temporary, subbed film support containing thereon (1) a layer of an organic solvent soluble synthetic resin which is impermeable to water (2) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (3) an antihalation layer and (4) a hardener-free light sensitive gelatin-silver halide photographic emulsion all in that order.

3. A gravure resist film comprising dimensionally stable, temporary, subbed film support containing thereon (1) a layer of a synthetic resin which is no more than poorly soluble in organic solvent, which layer is impermeable to water (2) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (3) an antihalation layer and (4) a hardener-free light sensitive gelatin-silver halide photographic emulsion all in that order.

4. A gravure resist film comprising dimensionally stable, temporary, subbed film support containing thereon (1) a layer of polyisobutyl methacrylate (2) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (3) an antihalation layer and (4) a hardener-free light sensitive gelatin-silver halide photographic emulsion all in that order.

5. A gravure resist film comprising dimensionally stable, temporary, subbed film support containing thereon (1) a layer of polyvinyl butyral (2) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (3) an antihalation layer and (4) a hardener-free light sensitive gelatin-silver halide photographic emulsion all in that order.

6. A gravure resist film comprising dimensionally stable, temporary, subbed film support containing thereon (1) a layer of maleic acid modified polyvinyl acetate chloride (2) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (3) an antihalation layer and (4) a hardener-free light sensitive gelatin-silver halide photographic emulsion all in that order.

7. A gravure resist film comprising dimensionally stable, temporary, subbed film support containing thereon (1) a lower alkyl acrylate copolymer hydrosol layer (2) an antihalation coating (3) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (4) a second antihalation coating and (5) a hardener-free light sensitive gelatin-silver halide photographic emulsion all in that order.

8. A gravure resist film comprising dimensionally stable, temporary, film support subbed with a cellulose nitrate layer containing over the subbing layer (1) a water-impermeable layer (2) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (3) a Carey Lea silver layer and (4) a hardener-free light sensitive gelatin-silver bromoiodide photographic emulsion all in that order.

9. A gravure resist film comprising dimensionally stable, temporary, film support subbed with a cellulose nitrate layer containing over the subbing layer (1) a lower alkyl acrylate copolymer hydrosol layer (2) a Carey Lea silver layer (3) a stripping layer composed of 10–15 parts of gelatin and 85–90 parts (dry weight) of an ethylacrylate-acrylonitrile copolymer latex in which ethyl acrylate units comprise 60–90 weight percent of the total polymer (4) an antihalation layer and (5) a hardener-free light sensitive gelatin-silver bromoiodide photographic emulsion layer all in that order.

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