

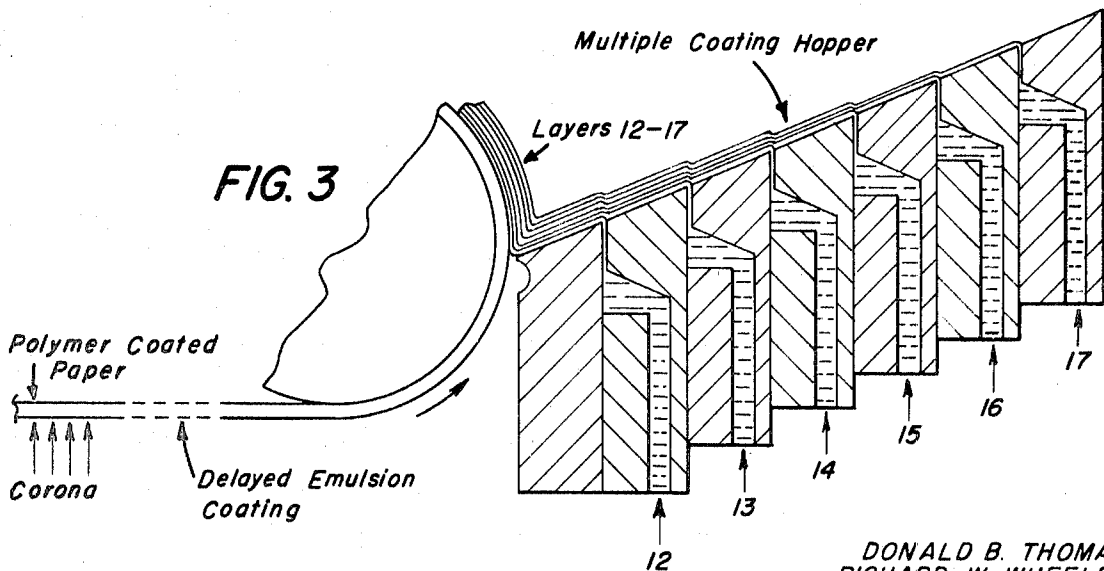
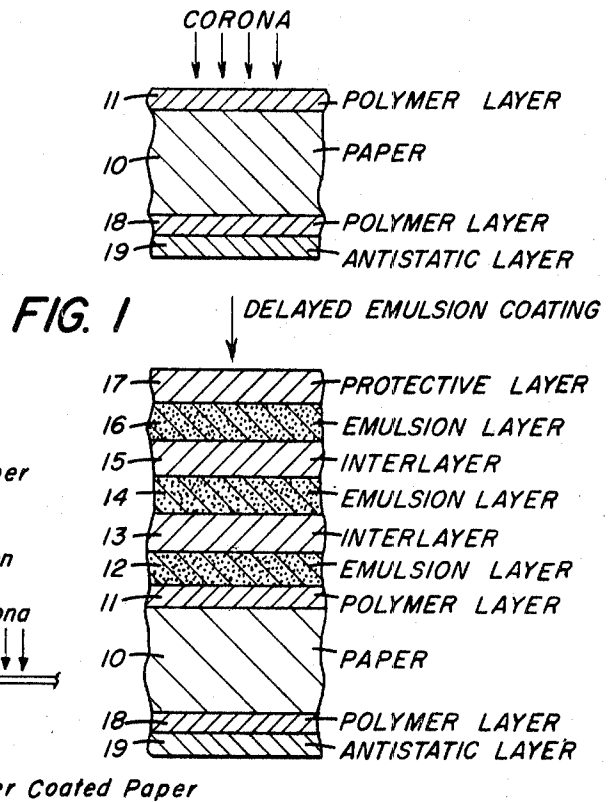
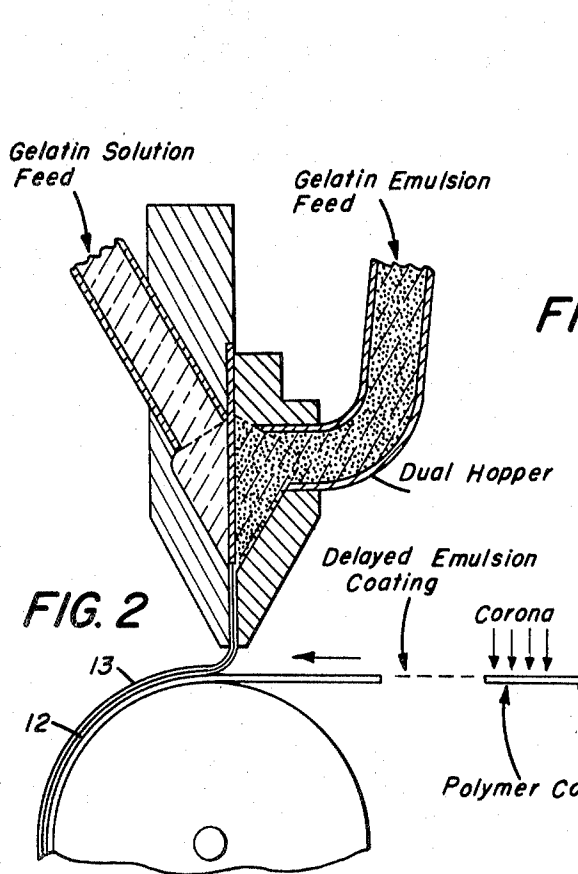
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[54] **PROCESS FOR COATING PHOTOGRAPHIC
EMULSION LAYERS**
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G03c 1/78, C03c 1/00
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ABSTRACT: Photographic emulsion layers are coated on corona activated polymer surfaces more than about 30 minutes after activation of the polymer surface for improvement of coating uniformity and stability of emulsion layers



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PROCESS FOR COATING PHOTOGRAPHIC EMULSION LAYERS

Photographic emulsions have been coated on transparent film base or reflective film or paper surfaces for preparation of black-and-white or color images for projection or viewing against the reflective surface for many years. There has long been a need for a waterproof paper support to prevent processing solutions and wash water from penetrating the paper and thus to decrease the washing and drying time of the processed paper prints. Paper coated with organic solvent solutions of synthetic polymers is useful for this purpose but is fairly costly among other disadvantages. More recently, synthetic polymers such as the polyolefins and linear polyesters have been found to be very useful for waterproofing photographic paper and are readily coated upon paper without use of organic solvents, using relatively cheap polymer extrusion methods.

However, as in the case of photographic film base, since the polymer coated paper surface is hydrophobic, the hydrophilic photographic emulsions, especially gelatin-silver halide emulsions, will not adhere unless the polymer surface has been subbed using a composition usually containing a polymer which will adhere to the polymeric paper surface and to which the photographic emulsion layer will in turn adhere. More recently, it has been found that if polymeric surfaces are activated by a strong corona discharge, the photographic emulsions and similar organic colloid layers can be coated with good adherence onto the corona activated polymer surface. This method of adhering the photographic emulsions to the polymer surface materially reduces the cost of manufacture of the waterproof, photographic paper but has several serious disadvantages. That is, when the surface of polymer films, and paper coated with the polymers such as polyethylene, is activated with corona discharge and then immediately coated in-line with either a single emulsion layer for black-and-white photography, or with a plurality of differently sensitized color emulsions for color photography, the emulsions are adversely affected. While the adhesion of the emulsions to the activated polymer surface is good, when the corona activation of the polymer surface is followed immediately by the coating of an emulsion layer, this layer is partially fogged. Thus, if a negative emulsion layer designed for use in black-and-white photography is coated on the freshly activated surface and is exposed and processed as usual with a black-and-white developer solution, substantial silver density appears in highlight areas which have received no image exposure. A similar effect may be obtained in the highlight areas if the emulsion layer comprises a direct-positive emulsion. In coating products for color photography wherein several emulsions are coated on the freshly corona activated polymer surface, the emulsions are affected by the activated surface in a manner such that an emulsion layer (usually the first coated emulsion in direct contact with the support) may be partially fogged, and another emulsion layer (usually the second coated emulsion) may contain coating irregularities such as a mottled pattern. The terms "mottle" or "mottled pattern" as used herein describe a defect readily observed when a silver halide emulsion layer containing a coupler compound is given a uniform exposure to light, color developed to provide a dye image, and the dye image, which should have even dye density throughout, instead exhibits marks which are areas having distinctly higher dye density than that of the remaining areas of the dye image. The areas having the higher dye density exhibit greater density than would be expected or predicted from the photographic speed of the particular system. Mottle is also frequently accompanied by a lower speed than would be predicted in areas outside the marks.

The solution to the problem is not readily apparent especially when it is noted that some photographic emulsions are not materially affected by the corona activated polymer surface. It is possible that the mentioned defects arise because of the presence of substances reactive with the activated polymer surface such as the particular silver halide used, color-forming coupler compounds, optical sensitizing agents, chemical sen-

sitizing agents or other addenda present in the emulsion layers.

However, it has been discovered that the mentioned adverse effects of the corona activated polymeric supports upon the light-sensitive emulsions can be overcome by treating the polymer surfaces with corona discharge which activates the surface sufficiently to effect adhesion of the emulsion layers, and at some later time, e.g. more than about 30 minutes later, when the adverse effect of the activated polymer surface has been dissipated but before substantial decrease in emulsion adhesion has taken place, coating the emulsions onto the activated polymer surface. Accordingly, it is usually desirable to activate the polymer surface such as polyethylene out-of-line on one machine equipped to supply the corona discharge, and at a later time on a different machine to coat the emulsions onto the activated polymer surface. As will be seen from the following description in reference to FIG. 3, the invention allows as many as six photographic layers for subtractive color photography to be coated simultaneously onto the corona activated polymer surfaces with good uniformity of coating. Moreover, the emulsions do not show fog usually obtained by coating the emulsions on the freshly corona activated polymer surfaces.

The accompanying drawings show the appearance of representative products and apparatus useful in the process of the invention and methods for carrying out the invention.

FIG. 1 shows in greatly enlarged cross-sectional view the appearance of a representative multilayer photographic element being prepared by the process of the invention as described in Example 3 below. The element including paper support 10, polymer layers 11 and 18 and antistatic layer 19 is corona activated at the surface of layer 11 and after suitable delay, e.g. 24 hours later, is coated with the emulsion, interlayer and protective layers 12-17 separately or simultaneously in the manner shown in FIG. 2 and FIG. 3.

FIG. 2 shows in diagrammatic form one method (using a dual coating hopper) wherein the polymer coated paper is activated with corona discharge, and after a suitable delay, simultaneously coating two hydrophilic organic colloid layers such as a gelatin emulsion layer 12 (the emulsion layer tending to become fogged by the activated polymer surface) and gelatin interlayer 13, upon the corona activated polymer surface of the polymer coated paper in production of the photographic element shown in FIG. 1. In a similar manner, the pairs of layers 14 and 15 (layer 14 tending to show mottle in response to the corona activated surface) and layers 16 and 17 are separately or simultaneously coated on layer 13. Alternately, the process of the invention allows all layers 12-17 to be coated simultaneously upon the corona activated polymer surface (after suitable delay) using a multiple coating hopper as shown in FIG. 3. The construction and operation of this type of hopper is described in greater detail in U.S. Pat. 2,671,791.

As discussed above, the corona activated polymer surfaces tend to fog a light-sensitive layer coated immediately thereon, and in the case of multilayer coatings for subtractive color photography, the second emulsion layer commonly exhibits coating irregularities such as mottle. According to the invention, by delaying the emulsion coating of the activated polymer surface longer than about 30 minutes, these defects are avoided. However, emulsion coating should not be delayed too long or a substantial decrease in adhesion of the emulsion layers to the activated polymer surface is obtained. For example, typical gelatin-silver halide emulsions containing color-forming couplers, adhere satisfactorily to samples of a corona activated polyethylene surface which have been aged about 30 minutes to 36 hours. It can be expected that other emulsions, e.g. common gelatin-silver halide emulsions free of color couplers, may adhere well when the activated polymer surface has been aged longer than 36 hours. However, it is advisable to run tests with each organic colloid photographic emulsion and each activated polymer surface to determine the optimum or useful time one can delay the emulsion coating

operation to avoid the fog and mottle problems and still obtain good adhesion of the emulsion layer to the activated polymer surface.

The photographic elements which are activated with corona as described above include films or surfaces of various polymers, including hydrophobic addition and condensation polymers which can be corona activated to effect adhesion of hydrophilic polymer materials such as gelatin. These polymers include polyolefins such as polyethylenes and polypropylenes and ethylene-propylene copolymers, polystyrene, polybutenes, polypentenenes, polyacrylic acid esters, linear polyesters such as polyethylene terephthalates, polycarbonates, polyamides such as nylon, cellulose esters, polyacrylonitrile, polyvinylidene chloride and other copolymers of the indicated monomers such as ethylene-vinylacetate copolymers. Paper coated with the polymers is especially useful.

The invention is used with these polymeric films, and paper coated with the polymers, and particularly when a conductive antistatic compound is incorporated into the support, i.e. in any of layers 10, 11, 18 or 19 as illustrated in the Examples below. Antistatic material particularly useful in the external layer 19 is a mixture of a (1) colloid such as hydroxymethyl cellulose, (2) colloidal silica and (3) either an alkali metal salt of 2,5-naphthalene disulfonic acid, an alkali metal salt of a lower alkyl naphthalene sulfonic acid, an alkali metal salt of the condensation product of formaldehyde and 2,5-naphthalene disulfonic acid, an alkali metal salt of an alkyl aryl polyether sulfonate or an alkali metal salt of polymeric carboxylic acid. The compound p-(1,1,3,3-tetramethylbutyl)phenoxyethoxyethyl sodium sulfonate is especially useful. Carbon black can also be used in the polymer layers. The antistatic layers are described in more detail in Miller et al., U.S. Pat. application Serial No. 594,226 filed Nov. 14, 1966. An internal antistatic material can be incorporated into the polymer support, into the paper or on the surface of paper which is coated with the above polymeric materials or into the polymer layers on the paper. Antistatic materials especially useful for this purpose include salts such as sodium sulfate and the like salts of organic compounds, such as the alkali metal and ammonium salts of the condensation products of an aldehyde such as formaldehyde with naphthalene sulfonic acids, organic antistatic agents, such as triethanolamine oleate, triethanolamine stearate, and various polyalkylene polyamine derivatives. Oxyalkylene amine derivatives of phosphorous, polyacryloxyalkyl trialkyl ammonium alkylsulfate salts, diethanol amine salts of phosphate esters, carbon black and the like may also be used. When antistatic materials are used in this way, inside the support, to provide antistatic protection, condenser geometry is present. That is, it can be shown that friction generates a charge on the polymer coated paper but when the internal antistat is present, the charge is stored in the element rather than being periodically discharged to the air or being conducted away to ground. The above external antistatic layers augment the internal antistatic method for overcoming static problems. Chu et al., U.S. Pat. 3,253,922 describes this method for providing internal antistatic protection to polymer coated papers.

The photographic layers which are coated upon the corona activated polymer surfaces in the manner described above include organic colloid layers such as gelatin interlayers, gelatin filter layers and differently sensitized gelatin-silver halide emulsion layers, etc. as shown in FIG. 1. The silver halide can be, e.g. silver bromide, silver iodide, silver chloride or mixed crystals of these silver halides, e.g. silver chlorobromide. The hydrophilic organic colloid of these layers is preferably gelatin but it may be replaced wholly or in part by known gelatin derivatives, water-soluble polymers such as partially hydrolyzed cellulose acetate, cellulose methyl ether, polyvinyl alcohol, hydrolyzed vinyl acetate copolymers, vinyl acetal-vinyl alcohol copolymers, alkylacrylate-acrylic acid copolymers, etc. The color emulsion layers coated on the corona activated polymer surfaces are preferably the incorporated coupler gelatin-silver halide emulsion layers well

known in the art. These emulsions contain colored or colorless nondiffusing cyan, magenta and yellow dye-forming coupler compounds, reactive with the oxidation products of p-phenylenediamine silver halide developing agents to form dyes.

Representative elements for color photography comprise superposed on the corona activated surfaced, red, green and blue light-sensitive gelatin-silver halide emulsion layers containing respectively, a cyan-forming coupler (e.g. a phenolic compound), a magenta-forming coupler (e.g. a 5-pyrazolone compound) and a yellow-forming coupler (e.g. an open chain ketomethylene compound) which are reactive with the p-phenylenediamine silver halide color developing agents to form dyes.

Suitable nondiffusing couplers are disclosed in U.S. Pat. Nos. 2,407,293, 2,640,776, 2,600,788, 2,801,171, 2,875,057 and 2,956,876. The couplers can be incorporated into the emulsion layers in accordance with well-known procedures, e.g. using coupler solvents as described in U.S. Pats. 2,304,939, 2,322,027, 2,801,170, 2,801,171 and 2,949,360. Accordingly, the terms "coupler compound" or "color-forming coupler compound" refer to well-known compounds such as disclosed by the above patents which react with the p-phenylenediamine color developing agents to form dyes in the development of silver halide emulsion layers.

The elements of the invention, prepared as described by delayed coating of the incorporated coupler emulsion layers on the corona activated polymeric surfaces can be exposed to color negatives and processed directly to color positives using the solutions and process disclosed at columns 5 and 6 of Van-Campen U.S. Pat. 2,956,879. Thus, a conventional color developer solution containing a primary aromatic amino silver halide developing agent is used and the silver images obtained are bleached and removed along with the residual silver halide leaving the subtractively colored dye images in the layers. It is in this negative color process that it is necessary that the emulsion in contact with the support (and, of course, all emulsions) not be fogged or otherwise adversely affected when coated on the corona activated polymer surface, since fog produces, e.g. yellow stain in highlights and on the borders of the prints. As mentioned, this fogging of the emulsion layers is most unexpectedly avoided by delaying the coating of the emulsion layers until at least about one-half hour after corona activation of the polymeric support, or at least until the fogging action of the activated surface has been dissipated. This delayed coating procedure also prevents formation of mottled dye images in the emulsion layers.

These multilayer color elements can also be processed by reversal to color positives. Thus, the element is exposed, for example to a subtractive color transparency, and as usual, developed with a black-and-white developer solution (a so-called MQ developer), the residual silver halide is then rendered developable by fogging with light or using chemical fogging and a p-phenylenediamine type of color developing solution is used to form the positive dye images in the layers. Silver and residual silver halide are removed by known methods leaving the dye images in the layers. In this process a small amount of fog in a layer due to the corona activated surfaces is not as objectionable since it only causes loss of density in the shoulder region of the yellow color scales.

The corona discharge applied to the polymer surfaces is supplied by well-known power sources. The spark gap type power source for the corona has current supplied to the electrodes by a spark gap excited oscillator in a well-known manner. Variation in fundamental frequency of the corona is obtained by changing the primary power frequency of the oscillator. As mentioned, a high voltage corona is desirable, e.g. 25,000 to 50,000 volts or higher, to obtain adequate adhesion of the emulsion layer to the corona activated surface. Voltages of this range are adequate for corona activation of polymers at web speeds of about 100 to 1,000 feet per minute or higher. Voltage is varied by spacing the spark gaps and by varying the primary voltage to the oscillator.

Continuous wave corona is advantageously used for activating the polymer surfaces prior to coating the emulsions. Continuous wave corona is obtained by using as the power source a motor-generator set whereby a sinusoidal waveform generator with a fixed number of poles is driven by a variable speed motor, giving a variable frequency sine wave output. Variation in frequency of the continuous wave corona of from about 1,000 to 10,000 or higher cycles per second is obtained by varying the speed of the driving motor. Voltage of the continuous wave corona which is stepped up in value by a multitap transformer and varied by field control can vary from about 5,000 volts to 30,000 volts or higher at web speeds of about 100 to 1,000 f.p.m.

If desirable, the corona discharge can be regulated as described in the Ambusk, U.S. Pat. application Ser. No. 724,491, filed Apr. 26, 1968. That is, the current frequency for the corona can be selected so that the ribbon of silver halide emulsion or other photographic layer being coated on the activated polymer surface does not appreciably respond to the pattern of the static charge on the activated polymer surface and coating irregularities are avoided. Thus, if a 60 Hz. corona is used and the emulsions coated at about 125 f.p.m., an objectionable nonuniform emulsion coating may be obtained. When corona of current frequency beyond about 200 Hz. is used, the emulsions usually can be coated at at least 125 f.p.m. without obtaining coating defects. Moreover, corona current frequencies in the range of 200 Hz. to 10,000 Hz. or higher are very useful since emulsion coating speeds of 1,000 f.p.m. and higher can be used to obtain uniform emulsion coatings as shown by the above Ambusk invention.

The corona can be applied to the polymeric surface, for example, by means of several metal electrodes positioned close to the polymeric surface at a point where the polymeric surface is passing over a grounded metal roll coated with a dielectric metal such as a linear polyester. Similarly, a metal roller may be used to support the web with the other electrode array being in planetary disposition equidistant from the surface of the metal roller and each being coated with a dielectric, at least on the surface nearest the metal roller. As mentioned above, the spacing of the electrodes to the polymer surface and ground roll should be adequate to produce the corona at the voltage used and yet allow for free passage of polymeric sheet through the activating zone. Corona supplied by DC current, or a combination of AC superimposed on DC can be used. However, there appears to be no advantage in using DC corona and, in fact, AC is preferred since the continuous wave AC corona requires much less power and is, thus, considerably cheaper to use.

The following examples will serve to illustrate the invention.

EXAMPLE 1

A photographic paper stock is provided sized with gelatin and aluminum stearate and extrusion coated on each side with about 8 lbs. polyethylene/1,000 sq. ft. One polyethylene surface is activated at 125 f.p.m. by a 400 L Hz. corona discharge at about 50,000 volts supplied by a spark gap type AC high frequency generator. Apparatus used is as described above and includes a series of electrodes positioned close to the polyethylene surface and connected to the generator. A dual hopper such as shown in FIG. 2 is used to coat the blue sensitive gelatin-silver bromiodide emulsion layer 12 and gelatin interlayer 13 simultaneously onto the freshly activated polyethylene surface at about 125 f.p.m. The pairs of layers 14, 15 and 16, 17 are coated similarly several hours later. Layers 12, 14 and 16 are gelatin-silver chlorobromiodide emulsion layers adapted to reversal color processing and primarily sensitive, respectively, to blue, green and red light and containing, respectively, nondiffusing yellow, magenta and cyan-forming couplers. Layers 13, 15 and 17 are gelatin layers. The element appears as shown in FIG. 1. The resulting element is exposed to a color original and processed by known reversal color development methods described above to form

dye images in the emulsion layers. As a result, the emulsion layers appear to have been uniformly coated and the dye images have satisfactory density and contrast. Adhesion of the emulsion layers to the corona activated polyethylene surface is also good.

EXAMPLE 2

PROCESS A

A photographic element is prepared as in Example 1 having coupler-containing gelatin-silver halide emulsion layers of the type described in Example 2 of VanCampen U.S. Pat. 2,956,879 adapted to development directly to negative dye images (positive images with respect to a color negative used for exposing the layers), except more than 30 minutes after 400 Hz. corona activation of the polyethylene surface all layers 12 to 17 are coated simultaneously as shown in FIG. 3 using a multiple coating slide hopper. U.S. Pat. 2,761,791, gives detail of the construction and operation of the hopper. Thus, solutions for layers 12, 13, 14, 15, 16 and 17 are extruded onto the slide surfaces of the hopper and in superposed relation are coated simultaneously onto the corona activated polyethylene surface. The resulting element having the structure shown in FIG. 1 is exposed to a color negative and color developed directly to produce positive subtractive colored dye images in the layers. As a result, emulsion layer 12 has low fog and emulsion layer 14 does not show significant mottle. The dye images have good density and contrast. The adhesion of the layers to the activated polyethylene surface is also good.

PROCESS B

When the same process is carried out except coating the layers 12-17 simultaneously immediately after activation of the polyethylene surface, substantial fog and mottle is found in emulsion layers 12 and 14 respectively. Moreover, a substantial loss in green speed is obtained in layer 14 as shown by the data in the following table.

| | Yellow Stain | Mottle Layer 14 | Green Speed (Rel. Log E) Layer 14 |
|-----------|-----------------|--------------------|---|
| Process A | 0.15 | No | 1.25 |
| Process B | 0.17 | Yes | 1.16 |

In obtaining this data the yellow stain density was measured as usual by reading the density of a highlight area of the processed element using a filter of contrasting color. Mottle was determined by observation.

EXAMPLE 3

The process of Example 2 is carried out except the paper support, before polyethylene coating, was sized with a gelatin solution of sodium sulfate to provide an internal antistat, and an external antistat layer is coated from an aqueous dispersion of hydroxyethyl cellulose (1.3 lbs.), colloidal silica (30%, 140 lbs.) and the compound p-(1,1,3,3-tetramethylbutyl) phenoxyethoxyethyl sodium sulfonate (12 lbs.) on the wire side of the polyethylene coated paper, the polyethylene preferably having been first activated with corona discharge to improve adhesion of the antistat layer. As a result of the delayed coating of the emulsion layers on the corona activated polyethylene surface, the blue sensitive emulsion layer 12 has low fog and the green sensitive emulsion layer 14 has good speed and contains no mottle. Emulsion adhesion to the polyethylene surface is good.

When the same polyethylene coated paper containing the internal and external antistat materials is corona activated and coated immediately thereafter with the blue sensitive emulsion layer 12, it has substantial fog and emulsion layer 14 had objectionable mottle and lower green speed.

EXAMPLE 40

Photographic paper is extrusion coated on each surface with about 8 lbs./1,000 sq. ft. of polyethylene terephthalate. Corona activation of one polyethylene terephthalate surface with 400 Hz. corona as in Example 2 followed about 24 hours later by the coating of layers 12-17 as described in Example 2 can be expected to give an acceptable level of fog in layer 12, and low mottle in layer 14. However, if the corona activated polyethylene terephthalate surface is immediately coated with layers 12-17, undesirable fog can be expected in layer 12 and mottle in layer 14.

EXAMPLE 5

The process of Example 2 is carried out except opaque pigmented polyethylene sheeting is used in place of the polyethylene coated paper. As a result of the delayed emulsion coating of the activated polyethylene surface, emulsion fog and mottle can be expected to be acceptable. The adhesion of the emulsion layers is also good.

EXAMPLE 6

A medium speed gelatin-silver bromiodide emulsion is coated at 125 f.p.m. upon a polyethylene coated paper such as described in Example 3 just after 400 Hz. corona activation of the polyethylene surface at 125 f.p.m. After exposure and development in a hydroquinone type developer objectionable fog is found in the emulsion layer.

When the emulsion layer is coated more than 30 minutes after activation of the polyethylene surface, the emulsion layer has a low level of fog.

In the manner of the above examples the other polymer films and polymer coated papers such as polypropylene coated paper, are corona activated and subjected to delayed emulsion coating of the activated polymer surfaces, to reduce the tendency for emulsion fog and mottle to develop.

Herein "corona activated" and "activating corona discharge," etc. means that the polymeric surface is treated with the corona discharge under the conditions and with the apparatus described above such that adequate adhesion of the photographic layer to the polymeric surface is obtained. Adhesion of hydrophilic layers to the activated polymer surface can be determined before coating the layer by measuring the contact angle of the activated surface. "Contact angle" in degrees is a measure of the layer adhesion to the activated polymer surface. Thus, in the case of polyethylene surfaces, if the corona activated surface has a contact angle of less than about 76°, for example 40° to 75°, dry and wet adhesion of hydrophilic layers such as gelatin layers is usually adequate. In the case of polypropylene surfaces an angle less than about

54° is desirable. Contact angle is the angle of contact of a drop of distilled water placed on a level surface of the activated polymer layer. The contact angle is obtained by projecting the image of the drop onto a suitable screen using a contour projector and measuring the angle of a line tangent to the drop image at the point the edge of the drop touches the sample.

The invention has been described in detail with particular reference to 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. In a process for preparing a photographic element wherein a polymeric surface of a support is activated with corona discharge to effect adhesion of differently sensitized photographic silver halide emulsion layers to the polymer surface, the improvement for obtaining greater uniformity of coating and stability of the emulsion layers, comprising activating the polymeric surface with a corona discharge producing a surface tending to fog a silver halide emulsion layer immediately coated thereon, and at least about 30 minutes later but before the adhesive property of the polymeric surface has substantially decreased, coating at least one of the emulsion layers on the activated polymeric surface.

2. The process of claim 1 wherein the support is paper having polymeric surfaces on each side and the emulsion layers are gelatin-silver halide emulsion layers which contain color-forming coupler compounds.

3. The process of claim 2 wherein the polymeric surface on at least one side of the paper is polyethylene.

4. The process of claim 2 wherein the polymeric surface on at least one surface of the paper is polyethylene, polypropylene, an ethylene copolymer or a linear polyester.

5. The process of claim 2 wherein the emulsion layers are coated simultaneously onto the corona activated polymer surface.

6. The process of claim 2 wherein one of the emulsion layers is coated just after corona activation of the polymer surface and the remaining emulsion layers are coated thereon at least about 30 minutes after the corona activation.

7. The process of claim 1 wherein the sensitive layer is a gelatin-silver halide emulsion layer.

8. The light-sensitive element prepared according to claim 2.

9. The light-sensitive element prepared according to claim 5.

10. The light-sensitive element prepared according to claim 6.

11. The process of claim 1 wherein the current frequency of the corona discharge is greater than about 200 Hz.

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