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3,520,682

## PHOTOGRAPHIC MOUNTING PROCESS AND PRODUCT

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No Drawing. Filed June 21, 1966, Ser. No. 559,069

Int. Cl. G03c 11/12, 5/54

U.S. Cl. 96-27

12 Claims

### ABSTRACT OF THE DISCLOSURE

This invention provides a process comprising (1) immersing in a solution selected from the group consisting of (A) liquid lower dialkyl ketone and (B) gum arabic, water and hydroxy benzene, an element comprising a support and thereon a diffusion transfer image-containing layer which had been formed by contacting a latent image with an image receiving layer thereby forming said diffusion transfer image, and (2) removing said support from said layer including said diffusion transfer image. The invention also provides uses for said layer including said diffusion transfer image. The invention further provides novel compositions for removing said layer from said support.

This invention relates to a process of removing a photographic layer bearing an image formed by the diffusion transfer process from its supporting member. The invention also relates to novel photographs that glow in the dark.

In the past it has not been possible to remove diffusion transfer images intact from their support layers. It has been the practice to leave diffusion transfer images on the support materials on which they have been formed, without attempting to remove them from their support thereby preventing transposition to a different support material, such as a transparent material, porcelain, enamelled metal, etc.

It is an object of this invention to provide a process for removing a diffusion transfer image from its support, while retaining desired dimensional stability. It is also an object of the present invention to provide photos that glow in the dark. Other objects and advantages of the present invention are in part obvious or will be apparent from the following description.

In accordance with certain of its aspects, this invention is directed to a process which comprises (1) immersing in a solution selected from the group consisting of (A) liquid lower dialkyl ketones, and (B) gum arabic, water and a hydroxybenzene, a diffusion transfer image on a support, and (2) removing said support from said layer containing said diffusion transfer image.

In accordance with this invention the diffusion transfer image which is removed from its support may be a black-and-white image or a color image. Such images may be produced in accordance with processes known to the art, particularly those assigned to the Polaroid Corporation, such as Nos. 2,698,237; 2,698,244; 2,968,554; 3,192,044; and 3,246,985.

Thus a black-and-white diffusion transfer image used in this invention has been produced by providing a photosensitive element comprising a white paper support and coated thereon a photosensitive emulsion, such as the extremely high panchromatic Eastman Kodak Super XXPan, the relatively high speed Eastman Kodak Verichrome, or Ansco Triple S Pan. The photosensitive emulsion is exposed in an imagewise manner to produce a latent image and indirectly contacted with an image receiving element comprising a support of material such as a cellulose, a mixed cellulose ester or a baryta paper and an image re-

ceiving layer comprising a silver precipitating agent such as metallic sulfides and selenides including sulfo-selenides, polysulfides, and polyselenides. Typical precipitating agents include the "heavy metal" sulfides of zinc, chromium, gallium, iron, cadmium, cobalt, nickel, lead, antimony, bismuth, silver, cerium arsenic and copper and "heavy metal" selenides of lead zinc, antimony and nickel. Preferably, the precipitating agents are the sulfides of zinc, cadmium and lead which have a solubility product in water at 20° C. of between 10<sup>-23</sup> and 10<sup>-30</sup>. The precipitating agents may also be such heavy metals as silver, gold, platinum, palladium and mercury, and preferably the noble metals in the form of colloidal particles. Simple inorganic and readily reducible salts of heavy metals such as silver nitrate, gold chloride and gold nitrates may also be used as precipitating agents. Other precipitating agents include thio compounds such as lead thiooxamate, zinc thiooxamate, potassium thiooxalate and the lead and nickel complexes thereof. Even relatively soluble sulfides and selenides such as those of sodium, potassium, cesium, calcium, barium and magnesium give acceptable pictorial quality. The pictorial quality of the image receiving layer is improved by including as a dispersing medium a matrix for the precipitating agent of particles of colloidal silica material.

The exposed emulsion bearing a latent image is indirectly contacted with the image receiving layer through a processing agent. The processing agent contains components for developing the latent image. A typical processing layer contains 1860 parts by weight water, 117 parts by weight sodium carboxymethyl cellulose, 78 parts by weight sodium sulfite, 74.6 parts by weight sodium hydroxide, 14.5 parts by weight sodium thiosulfate, 38.5 parts by weight citric acid and 52 parts by weight hydroquinone. The components of the processing agent are preferably present in an elongated rupturable container which is at least about equal in size to the transverse dimension of the area of the photosensitive element. One or more of such containers may be present.

To form the diffusion transfer image the container enclosing the processing agent is broken by pressure, the processing agent permeates the emulsion and develops the latent image in the emulsion layer. Almost simultaneously with this development, the complex-forming component of the processing agent, such as sodium thiosulfate, forms a soluble silver complex with the undeveloped silver halide of the emulsion. This soluble silver complex is transported to and diffused into the image receiving layer where it reacts with the silver precipitating agent, typically 280 parts by weight of a 1% aqueous solution of sodium sulfide dispersed in 30 parts by weight of silica aerogel and 92 parts by weight of a solution formed from 30 parts by weight of cadmium acetate, 1 part by weight of neutral lead acetate and 30 parts by weight of zinc nitrate in 100 parts by weight of water thereby precipitating silver from the silver complex to form a diffusion transfer image.

A color diffusion transfer image which may be used in this invention has been produced by providing a support for the image receiving layer formed of material such as a cellulose, or a mixed cellulose ester or paper which may include baryta. On this support, there is present an image receiving layer which bears a color developer which is capable of reacting with an unreacted color coupling material which diffuses from an exposed and developed multicolor photographic emulsion to the image receiving layer.

Thus, a color diffusion transfer image may be formed in a process which includes exposure of a color photographic emulsion, such as a silver halide emulsion, developing said photographic emulsion with a processing agent emitted from a pressure rupturable container which

includes developer material for the exposed silver halide, such as 1,5-dihydroxynaphthalene and contacting said exposed and developed color photographic emulsion with an image receiving element comprising a compound which is capable of reacting with unreacted developer, such as Naphthosol Fast Orange Salt GC and a support therefor thereby diffusing the unreacted silver halide developer into the image receiving layer causing reaction to form a color image receiving layer.

The image receiving layer which receives color images may contain other materials than Naphthosol Fast Orange Salt GC, which materials react with unreacted developer which diffuses from the exposed photographic emulsion. A typical material disclosed for reaction with unreacted developer materials such as 1-phenyl-3-pyrazolidone or 5-nitroimidazole is N-methoxymethyl polyhexamethylene adipamide, which is sold under the trade name Nylon Type F8 by E. I. du Pont de Nemours & Co., Wilmington, Del.

The aforescribed processes for forming layers containing diffusion transfer images on supports are largely those described in the patents specified hereinbefore.

It is preferred to clean the diffusion transfer image prior to immersing the supported image in the solution for separating image from support. A preferred image cleaning solution is naphtha. The naphtha is preferably applied to the image on absorbent cotton and the image should be dried for about 10 minutes.

The cleaned image is then immersed in a solution which removes the image-containing layer from the support in accordance with this invention. A preferred solution is a liquid lower alkyl ketone, i.e., a ketone containing a total of 3 to 11 carbon atoms, such as acetone, methyl ethyl ketone, diethyl ketone, ethyl propyl ketone, di-propyl ketone, dibutyl ketone, diamyl ketone; and most preferably acetone.

Liquid lower dialkyl ketone is preferably employed in removing color diffusion transfer images from supports. Another solution which is employed for removing diffusion transfer images, and particularly color diffusion transfer images for supports is gum arabic, water and a hydroxy benzene, preferably present in amounts of 8-15 parts by weight, e.g., 11 parts of gum arabic, 75-100 parts by weight, e.g., 85 parts of water and 0.03-0.1 part by weight, e.g., 0.05 part of hydroxy benzene. Typical hydroxy benzenes include phenol, resorcinol, phloroglucinol, hydroxy toluene, etc. The preferred hydroxy benzene is phenol (carbolic acid). This type of solution is particularly effective in providing an image which when removed from its support has sufficient flexibility to be applied to rounded and concave surfaces such as lamp bases.

In accordance with this invention liquid lower dialkyl ketone, such as acetone, may be admixed with other components to effectively provide a solution for separating black-and-white and color diffusion transfer images from their supports. Thus 5-10 parts by weight, e.g. 8.4 parts of an alkyl cellulose, 10-20 parts by weight, e.g. 16.5 parts of a carboxylic acid ester, 20-30 parts by weight, e.g. 25.3 parts of an alkyl ester of a hydroxy carboxylic acid and 10-20 parts by weight, e.g. 15.8 parts, of an aromatic hydrocarbon may be added to 40-60 parts by weight, e.g. 48.2 parts, of a liquid lower alkyl ketone. Typical alkyl celluloses include, methyl cellulose, ethyl cellulose, propyl cellulose, butyl cellulose, etc., preferably ethyl cellulose. Typical carboxylic acid esters include methyl acetate, ethyl acetate, methyl propionate, butyl butyrate, etc., preferably ethyl acetate. Typical alkyl esters of hydroxy carboxylic acids include the alkyl lactates, such as methyl lactate, ethyl lactate, propyl lactate, etc., preferably ethyl lactate. Typical aromatic hydrocarbons include benzene, toluene, xylene, trimethyl benzene, ethyl benzene, etc., toluene is preferable. The preferred lower alkyl ketone is acetone.

The diffusion transfer image-containing layer and sup-

port is immersed in solution, as aforescribed, preferably for two seconds to one hour. When using the aforescribed preferred liquid lower dialkyl ketone and cellulose containing solution, the solution may be spread on the surface of the image side of the containing layer as a thin liquid film and then allowed to dry; preferably it is dipped into said solution for several seconds, e.g., 1-3, and then hung up and allowed to dry. When dried a coating is formed on the image-layer and the image-containing layer may be separated and removed from the support. Preferably a sharp instrument such as a knife or a blade is employed to separate the image layer from the support at a corner thereof and one is then easily and completely removed from the other. If there is any sticking, cleaning with a swab of acetone facilitates the removal. These image-layers retain a high degree of dimensional stability.

Preferably, when a color diffusion transfer image-layer is to be removed from its support using an acetone solution, the rear portion of the support is removed prior to immersion in the solution. This avoids a darkening of the image attributed to a reaction between acetone and a substance found in the rearmost portion of the support. When this step is taken, the image-layer will become free of its backing while still in solution after about two minutes. When the invention is practiced in this manner it is preferable to place a small mark in a particular corner of the image, such as a small cut out, in order to indicate the face side of the image upon removal from the solution.

When the black-and-white or color diffusion transfer image-containing layer has been removed from its support it may desirably be applied to various other supporting materials or, in the case of a black-and-white or color image it may be used to prepare color separation negatives in halftone for reproduction in the graphic arts.

Thus, the image can be applied to a plate, a lantern slide, etc., by first cleaning the new support, preferably with an alcohol, such as methanol, ethanol, propanol, isopropanol, butanol, etc., preferably isopropanol. An adhesive is then applied to the new support. Typical preferred adhesives are solutions such as 8-15 parts by weight, e.g., 11 parts, of gum arabic, 75-100 parts by weight, e.g., 85 parts, of water and 0.03-0.1 part by weight, e.g., 0.05 part, of an hydroxy benzene such as phenol. Another adhesive which may be employed comprises 15-35 parts by weight, e.g., 2.3 parts of an alkyl cellulose, such as ethyl cellulose and 35-45 parts by weight, e.g., 39.6 parts of acetone.

The black-and-white image may then be dipped in water and applied to the new support firmly, as with small blotters while the edges are moistened with a wet sponge. However, water is not used with color images. After drying, the image may be silhouetted, encircled or the like and any undesired film removed with a knife. Naphtha on absorbent cotton may then be applied for cleaning. After drying again, a protective coat may be applied. A typical type of protective coat is formed by dissolving 6-10 parts by weight, e.g., 7.8 parts of Parlodion, a pure collodion in 100-200 parts by weight, e.g., 113.4 parts of a liquid lower alkyl ketone such as acetone and then adding 100-200 parts by weight, e.g., 113.4 parts of a carboxylic acid ester, such as amyl acetate. If desired this solution may be thinned with equal parts by weight of ketone and ester.

The image may be transferred to a small crystal or to a watch dial by pressing firmly with small blotters and moistening the edges. In this case, however, a particularly desirable protective coat comprises 5-10 parts by weight, e.g., 6.5 parts gum dammar and 100-200 parts by weight, e.g., 113.4 parts of carbon tetrachloride.

When it is desired to place the color image on a concave surface such as a lamp base, the solution employed to remove the image-containing layer from its initial support is preferably 11 parts by weight of gum arabic, 85 parts by weight of water and 0.05 part by weight of

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phenols and the period of immersion is about one-half hour. The concave surface is cleaned with alcohol and the image after separation from its initial support and removal from the solution, is applied directly to the concave surface, held down firmly with a plastic film to prevent wrinkles and then blotted and dried at room temperature. The image can be silhouetted or encircled and the unwanted part removed with a knife. Naphtha on absorbent cotton may be applied for cleaning. After drying, a protective coat, such as a gum dammar spray solution, is applied.

Black-and-white diffusion transfer images can be applied to enameled metal surfaces or to ceramic surfaces or glass. Thus, in application of the image to enameled metal, a metal from various metallic compounds of gold, platinum, lead, palladium, etc., is plated on the image prior to treatment with the diketone solution and separation from its support, and fired to form a glaze. Red color is produced when gold compound is used; black when palladium compound is used; and yellow when lead compound is used. Gold and palladium compounds are also used together for black color.

Prior to plating of the metallic compounds and firing, the image is cleaned with naphtha on absorbent cotton and dried for a few minutes. It is immersed in 14.8 parts by weight of a solution of 3.9 parts by weight of thiourea in 226.8 parts by weight of water, then 14.8 parts by weight of a solution of 1.0 part by weight of gold chloride in 226.8 parts by weight of water and then 9.3 parts by weight of a solution of 3.2 parts by weight of citric acid in 226.8 parts by weight of water for red plating.

For black image plating the above red plating procedure is practiced followed by a 10-second water rinse and immersion in an aqueous solution of hydrochloric acid and palladous chloride.

The image, plated as aforescribed with the color desired, is separated from its support by immersion in a solution in accordance with this invention, such as 8.4 parts by weight of ethyl cellulose, 48.2 parts by weight of acetone, 16.5 parts by weight of ethyl acetate, 25.3 parts by weight of ethyl lactate and 15.8 parts by weight of toluene, application of a knife to a corner of plated image and support and removal of one from the other.

After drying and dipping in water the plated image is applied firmly with small blotters to the enameled metal surface which has been cleaned first with gum turpentine and then with denatured alcohol to which an adhesive of gum arabic, water and phenol has been applied such as with a hair brush. The element is dried, placed in a kiln for about 1½–2 minutes which has been preheated to about 650° C.–750° C., say e.g., 676.7° C., removed and cooled on asbestos.

The image may be first plated and then separated from its support in a similar manner when it is desired to apply the image to ceramic material such as glass or porcelain or china. When the image is applied to ceramics bearing an adhesive, the kiln into which the element is placed is not preheated, but the image is rather placed in the kiln prior to heating. With glasses, heat is applied to about 475° C.–525° C., e.g., 482.2° C. with the kiln door ajar, e.g., 0.6 cm. The door is then closed and the temperature raised to about 550° C.–600° C., e.g., 557.2° C. Higher temperatures, e.g., about 850° C.–870° C. are used with porcelain or china. The kiln is then cooled and the ceramic image removed when the kiln has reached ambient temperature.

In accordance with an aspect of this invention the support to which the diffusion transfer image is applied can be a transparent plastic support, such as polyethylene terephthalate. Luminescent material is then applied directly to the other surface of the image, thereby permitting the image to be viewed through the transparent support and to glow in the dark after having been exposed to artificial or natural light. Such exposure may be for even very short times, say 3–10 seconds. The luminescent material may

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also be applied directly on, or directly against the back of the image layer, presenting a photo that glows in the dark after exposure to light.

Thus, for instance, a black-and-white diffusion transfer image is cleaned with naphtha, dried and removed from its support after immersion in a solution of ethyl cellulose, ethyl acetate, ethyl lactate and toluene in acetone. The image is then dipped in water and applied firmly with small blotters, while moistening the edge on a wet sponge, to a transparent support, e.g., polyethylene terephthalate, which has been cleaned with isopropanol and bears an adhesive of gum arabic, water and phenol. After drying, the back of the image layer is coated with a luminescent material such as the paint and film sold under the trademark "Spot-Lite" by Canrad Sales Corporation. Alternatively, the image-containing layer may be applied directly on a luminescent material support. The image would then preferably be coated, e.g., with Parlodion, for protection.

The invention will be further illustrated but is not intended to be limited by the following examples. All parts, except where otherwise stated, are by weight.

#### EXAMPLE 1

A black-and-white diffusion transfer element including an image affixed to a support is cleaned by wiping the image surface with naphtha on a wad of absorbent cotton and dried for 5 minutes. The element is then dipped into the following solution for 1–3 seconds and then suspended and allowed to dry.

| Parts: | Components       |
|--------|------------------|
| 8.4    | Ethyl cellulose. |
| 16.5   | Ethyl acetate.   |
| 25.3   | Ethyl lactate.   |
| 15.8   | Toluene.         |
| 48.2   | Acetone.         |

After one hour the portion of the dried image of interest is squared up by pressing a knife edge through the image layer into the support, and then peeling the image layer away from the support.

#### EXAMPLE 2

A color diffusion transfer element including an image and a baryta paper support is cleaned by wiping with naphtha on a wad of absorbent cotton and dried. The image layer and front half of the paper support is squared up with a knife and removed from the rear half of the paper support. A small piece, 0.16 cm., is cut from the upper right corner of the element to indicate the face side of the image. The element is then immersed in acetone and the liquid is agitated. After 2 minutes, the color image layer detaches itself completely from the paper backing. The image is removed from the liquid, the cut in the upper right hand corner clearly indicating the face side. Any support material still affixed to the image layer is easily removed with acetone.

#### EXAMPLE 3

A color diffusion transfer element including an image and a baryta paper support is obtained. A small piece of the element is cut to indicate the face side of the image. The element is then immersed in the following solution:

| Parts: | Components  |
|--------|-------------|
| 11     | Gum arabic. |
| 85     | Water.      |
| 0.05   | Phenol.     |

After one-half hour, the color image detaches itself completely from the paper backing. The image is removed from the liquid, the cut in the element clearly indicating the face side.

#### EXAMPLE 4

The image freed of its support in accordance with Example 1 is pressed with small blotters firmly onto the

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surface of a crystal which has been cleaned with isopropanol and to which an adhesive of 11 parts of gum arabic, 85 parts of water and 0.05 part (4 drops) of phenol, has been applied. The element is then cleaned with a cotton wad of naphtha and the image protected with a solution of 6.5 parts of gum damar and 113.4 parts of carbon tetrachloride.

## EXAMPLE 5

A black-and-white diffusion transfer element including an image and a support is obtained and cleaned with a cotton wad of naphtha.

The element is then immersed in the following red plating solution: 14.8 parts of a solution of 3.9 parts of thiourea in 226.8 parts of distilled water; 14.8 parts of a solution of 1.0 part of gold chloride in 226.8 parts of distilled water; 9.3 parts of a solution of 3.2 parts of citric acid in 226.8 parts of distilled water.

After agitating for one-half hour the element is rinsed with water for 10 seconds and immersed for 3 minutes in the following black plating solution: 28.4 parts of a 5% palladium chloride solution in 85 additional parts of hydrochloric acid acidified distilled water.

The element is then washed for 3 minutes, dried and immersed in the following solution:

| Parts:     | Components       |
|------------|------------------|
| 8.4 -----  | Ethyl cellulose. |
| 16.5 ----- | Ethyl acetate.   |
| 25.3 ----- | Ethyl lactone.   |
| 15.8 ----- | Toluene.         |
| 48.2 ----- | Acetone.         |

After 1 hour the element is removed from the solution and the image separated from the support in the manner set forth in Example 1.

An enameled metal surface is cleaned first with gum turpentine and then with denatured alcohol. An adhesive of 11 parts of gum arabic, 85 parts of water and 0.05 part of phenol is applied to the enamel surface with a camel hair brush.

The image freed of its support is placed in firm contact with the enameled metal surface with small blotters, the sides moistened with a sponge and dried.

The enameled surface and the image are placed in a kiln preheated to 676.7° C. for 2 minutes, removed and cooled on asbestos. The resultant image is fused to the enamel surface.

## EXAMPLE 6

The procedure of Example 5 is followed except that a white glass (milky glass) is used in place of the enameled metal surface and after the image is firmly placed on the glass, moistened and dried, the glass and image are placed in an unheated kiln which is then heated to 482.2° C. with the door ajar 0.6 cm. The door is then closed and the temperature raised to 557.2° C. The kiln is then cooled and a ceramic image removed after complete cooling.

## EXAMPLE 7

The image layer freed of its support in accordance with Example 1 is pressed firmly onto a transparent plastic surface which has been cleaned with isopropyl alcohol and to which an adhesive of 11 parts of gum arabic, 85 parts of water, and .05 part of phenol has been applied. The other surface of the image layer is then coated with a luminous paint in liquid form trademarked "Spot-Lite" and sold by Canrad Sales Corporation. The element is exposed to light for 10 seconds and placed in the dark. A luminous image is observed.

## EXAMPLE 8

The image freed of its support in accordance with Example 3 is pressed face up onto a concave lamp base which has been cleaned with isopropyl alcohol, being careful to eliminate wrinkles by placing over a piece of plas-

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tic film then using blotters. After drying, the image is then cleaned with a cotton wad of naphtha, working from the center out. Trimming of the image with a knife point to remove undesired sections is preferably carried out before cleaning. The image is then protected by application of a spray of the gum dammar solution disclosed in column 4, lines 65-70.

It will be understood further that other changes may be made in the details of this invention without departing from the spirit thereof, especially as defined in the following claims.

What is claimed is:

1. A process comprising (1) immersing in a composition selected from the group consisting of (A) liquid lower dialkyl ketone and (B) gum arabic, water and hydroxy benzene, an element comprising a support and thereon a diffusion transfer image-containing layer which had been formed by contacting a latent image with an image receiving layer thereby forming said diffusion transfer image, and (2) removing said support from said layer including said diffusion transfer image.

2. The process of claim 1 wherein said composition is a liquid lower dialkyl ketone.

3. The process of claim 2 wherein said ketone is acetone.

4. The process of claim 2 wherein said ketone solution also includes an alkyl cellulose, a carboxylic acid ester, an alkyl ester of a hydroxy carboxylic acid and an aromatic hydrocarbon wherein said immersion was for several seconds followed by drying, and wherein said image-containing layer is removed by inserting a sharp edged instrument through the image-containing layer into the support layer, and then peeling the two layers apart.

5. The process of claim 4 wherein said acetone solution comprises 5-10 parts by weight of ethyl cellulose, 10-20 parts by weight of ethyl acetate, 20-30 parts by weight of ethyl lactate, 10-20 parts by weight of toluene and 40-60 parts by weight of acetone.

6. The process of claim 1 wherein said composition comprises 8-15 parts by weight of gum arabic, 75-100 parts by weight of water and 0.03-0.1 part by weight of phenol.

7. The process of claim 6 wherein said element is immersed in said composition for about one-half hour until the image-containing layer becomes detached from said support.

8. The process of claim 1 wherein said diffusion transfer image is a color image and the rear portion of said support is separated from the remainder of said support prior to the immersion.

9. The process of claim 1 wherein said diffusion transfer image-containing layer is applied to a new support subsequent to its separation from the original support.

10. The process of claim 4 wherein said diffusion transfer image-containing layer is a black-and-white image, and a metal is plated on said diffusion transfer image layer prior to said immersion; and wherein, subsequent to the separation, said image-containing layer including said diffusion transfer image is applied to an enameled metal surface, heated for about 1½-2 minutes in a preheated closed kiln at about 650-750° C. and removed from the hot kiln.

11. The process of claim 4 wherein said diffusion transfer image-containing layer is a black-and-white image, and a metal selected from the group consisting of gold, lead and palladium is plated on said diffusion transfer image layer prior to said immersion; and wherein, subsequent to the separation, said image-containing layer including said diffusion transfer image is applied to a glass material, placed in a previously unheated kiln, heated to about 475° C.-525° C. with the door ajar, and then heated to about 550° C.-600° C. in the kiln with the door closed and then cooled in the kiln.

12. The process of claim 1 wherein a luminescent backing material is affixed to said diffusion transfer image-con-

taining layer to form a photographic image that glows in the dark after being subjected to light.

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