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(54) Title: WET ETCHING PATTERNING COMPOSITIONS AND METHODS

(57) Abstract: A method comprising etching a film comprising electrically conductive structures according to a pattern using an aqueous etching solution to provide an etched region having a first conductivity and an unetched region having a second conductivity, the second conductivity being greater than the first conductivity, wherein the aqueous etching solution either comprises 25 to 65% by weight of phosphoric acid and 1 to 18% by weight of nitric acid, or the aqueous etching solution comprises 65 to 75% by weight of nitric acid.

WET ETCHING PATTERNING COMPOSITIONS AND METHODS

CROSS REFERENCE TO RELATED APPLICATIONS

This application claims the benefit of U.S. Provisional Patent
5 Application No. 61/876,417, filed September 11, 2013, entitled “WET ETCHING
PATTERNING COMPOSITIONS AND METHODS,” which is hereby
incorporated by reference in its entirety.

BACKGROUND

10 Electrically conductive films are used in electronic applications,
such as touch screen sensors for portable electronic devices. Electrically
conductive films comprising metal nanowires, such as, for example, silver
nanowires, are particularly well suited for such applications because of their high
conductivity, high optical transparency, and flexibility.

15 For many applications, such as, for example, capacitive touch
screen devices, films are patterned to provide regions of different conductivities.
The presence of a finger may be detected by circuitry attached to the patterned
film. Non-conductive regions can be formed by chemically etching the film. See
for example, Allemand et al., U.S. Patent No. 8,174,667, and Winoto et al., U.S.
20 Patent Publication 2011/0253668, which are hereby incorporated by reference in
their entirety.

SUMMARY

In some embodiments, a method may comprise etching a film
25 comprising electrically conductive structures according to a pattern using an
aqueous etching solution to provide an etched region having a first conductivity
and an unetched region having a second conductivity, the second conductivity
being greater than the first conductivity, where the aqueous etching solution may
comprise 25 to 65% by weight of phosphoric acid and 1 to 18% by weight of
30 nitric acid. Such a film may, in some cases, be electrically conductive prior to
etching.

In some embodiments, the aqueous etching solution may comprise 40 to 60% by weight of phosphoric acid, 10 to 15% by weight of nitric acid, and 0.005-0.05% by weight surfactant. In some embodiments, the aqueous etching solution may comprise 45% by weight of phosphoric acid, 15% by weight of nitric acid, and 0.01% by weight surfactant. In some embodiments, the aqueous etching solution may comprise 50% by weight of phosphoric acid, 10% by weight of nitric acid, and 0.01% by weight surfactant. In some embodiments, the aqueous etching solution may comprise 63.5% by weight of phosphoric acid and 14.5% by weight of nitric acid. In some embodiments, the aqueous etching solution may comprise 40% by weight of phosphoric acid, 18% by weight of nitric acid, and 0.01% by weight surfactant. In some embodiments, the aqueous etching solution may comprise 60% by weight of phosphoric acid, 10% by weight of nitric acid, and 0.01% by weight surfactant. In some embodiments, the aqueous etching solution may comprise 6 to 12% by weight of hydrochloric acid, 25 to 35% by weight of phosphoric acid, and 0 to 1.5% by weight of nitric acid. In some embodiments, the aqueous etching solution may comprise 7% by weight of hydrochloric acid, 34% by weight of phosphoric acid, and 1% by weight of nitric acid. In some embodiments, the aqueous etching solution may comprise 10% by weight of hydrochloric acid, 26% by weight of phosphoric acid, and 1% by weight of nitric acid.

In some embodiments, prior to etching, the film may exhibit a preexisting set of optical properties, and after etching, the etched region may exhibit a first consequent set of optical properties, and the unetched region may exhibit a second consequent set of optical properties, and the preexisting set of optical properties and the first consequent set of optical properties may be substantially identical. In some embodiments, the preexisting set of optical properties may comprise a preexisting total light transmission and the first consequent set of optical properties may comprise a first consequent total light transmission that may be substantially identical to the preexisting total light transmission. In some embodiments, the preexisting set of optical properties may comprise a preexisting haze and the first consequent set of optical properties may comprise a first consequent haze that may be substantially identical to the

preexisting haze. In some embodiments, the preexisting set of optical properties may comprise a preexisting L^* value and the first consequent set of optical properties may comprise a first consequent L^* value that may be substantially identical to the preexisting L^* value. In some embodiments, the preexisting set of optical properties may comprise a preexisting a^* value and the first consequent set of optical properties may comprise a first consequent a^* value that may be substantially identical to the preexisting a^* value. In some embodiments, the preexisting set of optical properties may comprise a preexisting b^* value and the first consequent set of optical properties may comprise a first consequent b^* value that may be substantially identical to the preexisting b^* value.

In some embodiments, the first consequent set of optical properties and the second consequent set of optical properties may be substantially identical. In some embodiments, the first consequent set of optical properties may comprise a first consequent total light transmission and the second consequent set of optical properties may comprise a second consequent total light transmission that may be substantially identical to the first consequent total light transmission. In some embodiments, the first consequent set of optical properties may comprise a first consequent haze and the second consequent set of optical properties may comprise a second consequent haze that may be substantially identical to the first consequent haze. In some embodiments, the first consequent set of optical properties may comprise a first consequent L^* value and the second consequent set of optical properties may comprise a second consequent L^* value that may be substantially identical to the first consequent L^* value. In some embodiments, the first consequent set of optical properties may comprise a first consequent a^* value and the second consequent set of optical properties may comprise a second consequent a^* value that may be substantially identical to the first consequent a^* value. In some embodiments, the first consequent set of optical properties may comprise a first consequent b^* value and the second consequent set of optical properties may comprise a second consequent b^* value that may be substantially identical to the first consequent b^* value.

In some embodiments, after etching, the pattern may be invisible to the unaided eye. In some embodiments, the absolute value of the difference

between the preexisting haze and the first consequent haze may be less than about 0.1. In some embodiments, the absolute value of the difference between the first consequent haze and the second consequent haze may be less than about 0.1. In some embodiments, the absolute value of the difference between the preexisting b* value and the first consequent b* may be less than about 0.1. In some
5 embodiments, the absolute value of the difference between the first consequent b* value and the second consequent b* value may be less than about 0.1. In some embodiments, the absolute value of the difference between the preexisting haze and the first consequent haze may be less than about 0.1, and the absolute value of
10 the difference between the first consequent haze and the second consequent haze may be less than about 0.1. In some embodiments, the absolute value of the difference between the preexisting b* value and the first consequent b* may be less than about 0.1, and the absolute value of the difference between the first consequent b* value and the second consequent b* value may be less than about
15 0.1. In some embodiments, the electrically conductive structures may comprise silver nanowires.

In some embodiments, the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising 42.5% by weight of phosphoric acid, 15% by weight of nitric acid, and 0.01% by weight of the
20 surfactant. In some embodiments, the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising 45% by weight of phosphoric acid, 13.75% by weight of nitric acid, and 0.01% by weight of the surfactant. In some embodiments, the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising 45% by weight of phosphoric acid, 12.5% by
25 weight of nitric acid, and 0.01% by weight of the surfactant. In some embodiments, the aqueous solution comprises a surfactant. In some embodiments, the aqueous solution comprises an anionic surfactant. In some embodiments, the aqueous solution comprises a surfactant, the surfactant comprising decyl(sulfophenoxy)benzenesulfonic acid, disodium salt, and
30 oxybis(decylbenzenesulfonic acid). In some embodiments, prior to etching the film, a mask is disposed onto the film, and after etching the film, the mask is removed from the film by being dissolved in a solution. In some embodiments,

prior to etching the film, a mask is disposed onto the film, and after etching the film, the mask is peeled from the film.

In some embodiments, a method may comprise etching a film comprising electrically conductive structures according to a pattern using an aqueous etching solution to provide an etched region having a first conductivity and an unetched region having a second conductivity, the second conductivity being greater than the first conductivity, where the aqueous etching solution may comprise 65 to 75% by weight of nitric acid. In some embodiments, the aqueous etching solution may comprise 70% by weight of nitric acid. Such a film may, in some cases, be electrically conductive prior to etching.

DESCRIPTION

All publications, patents, and patent documents referred to in this document are hereby incorporated by reference in their entirety, as though individually incorporated by reference.

U.S. Provisional Patent Application No. 61/876,417, filed September 11, 2013, entitled "WET ETCHING PATTERNING COMPOSITIONS AND METHODS," which hereby incorporated by reference in its entirety.

20

Introduction

Applicants have discovered a method of patterning an electrically conductive film employing a composition comprising phosphoric acid and nitric acid. The composition may produce a patterned film having regions of suitably low conductivity while the pattern is invisible to the unaided eye with minimal top coat damage and without requiring additional processing steps, such as additional heating or quenching.

Low and High Conductivity Regions of an Electrically Conductive Film

Electrically conductive films comprising electrically conductive structures, such as electrically conductive microstructures or electrically conductive nanostructures, are known. Microstructures and nanostructures are

30

defined according to the length of their shortest dimensions. The shortest dimension of the nanostructure is sized between about 1 nm and about 100 nm. The shortest dimension of the microstructure is sized between about 0.1 μm and about 100 μm . Conductive nanostructures may include, for example, metal nanostructures. Non-limiting examples of electrically conductive nanostructures that may be incorporated into the electrically conductive layer include nanowires, nanotubes, metal meshes, graphenes, and oxides, such indium tin oxide. Such electrically conductive nanostructures may comprise metals, such as silver. For example, the electrically conductive nanostructures may be silver nanowires.

5

10 Examples of transparent conductive films comprising silver nanowires and methods for preparing them are disclosed in US patent application publication 2012/0107600, entitled "TRANSPARENT CONDUCTIVE FILM COMPRISING CELLULOSE ESTERS," which is hereby incorporated by reference in its entirety.

15 Such electrically conductive films may exhibit surface resistivities of about 100 ohms/sq or lower, such as, for example, 60 ohms/sq, prior to patterning. The electrically conductive films may be patterned to introduce low conductivity regions, leaving the remaining regions as high conductivity regions. In some embodiments, the low conductivity regions may have substantially no

20 conductivity. In such cases, an Eddy Current meter may register a conductivity of 0. In some embodiments, the conductivity of the high conductivity regions after patterning may be within 10% (e.g. 5%, 1%) of the conductivity of the film prior to patterning.

 Patterning methods are known in the art, such as chemical etching

25 by screen printed mask, chemical etching by photolithography, chemical etching by screen printed etchant, or direct laser patterning. Such methods may include disposing a mask prior to etching a film, and the mask may be removed by being dissolved in a solution or peeling the mask from the film. In some cases, the fact that patterning has been performed may be obscured by making the high

30 conductivity regions and low conductivity regions have similar optical properties, rendering the patterned film suitable for various end-use applications.

Maintenance of the film's optical properties during the etching process can be important.

Etchant Composition

5 Etchants may comprise one or more polar solvents, at least one acid, and optionally, at least one metal halide, at least one surfactant, or at least one polymer. In some embodiments, an acid etching solution may be used. An acidic etchant may comprise at least one acid. Non-limiting examples of acids include nitric acid, phosphoric acid, and hydrochloric acid. The acidic etchant
10 may comprise an acid or a combination of acids with or without a binder. In some cases, the acidic etchant may etch the conductive nanostructures, such as silver nanowires, such that the silver nanowires are structurally compromised. In such cases, the etched silver nanowires may be no longer conductive or may become less conductive than the unetched silver nanowires.

15 In some embodiments, an aqueous etching solution may comprise 25 to 65% by weight of phosphoric acid and 1 to 18% by weight of nitric acid. In some embodiments, the aqueous etching solution may comprise 40 to 60% by weight of phosphoric acid, 10 to 15% by weight of nitric acid, and 0.005 to 0.05% by weight surfactant. In some embodiments, the aqueous etching solution may
20 comprise 42.5% by weight of phosphoric acid, 15% by weight of nitric acid, and 0.01% by weight of a surfactant. In some embodiments, the aqueous etching solution may comprise 45% by weight of phosphoric acid, 13.75% by weight of nitric acid, and 0.01% by weight of a surfactant. In some embodiments, the aqueous etching solution may comprise 45% by weight of phosphoric acid, 12.5%
25 by weight of nitric acid, and 0.01% by weight of a surfactant. In some embodiments, the aqueous etching solution may comprise 45% by weight of phosphoric acid, 15% by weight of nitric acid, and 0.01% by weight of a surfactant. In some embodiments, the aqueous etching solution may comprise 50% by weight of phosphoric acid, 10% by weight of nitric acid, and 0.01% by
30 weight of a surfactant. In some embodiments, the aqueous etching solution may comprise 55% by weight of phosphoric acid and 15% by weight of nitric acid. In some embodiments, the aqueous etching solution may comprise 55% by volume

of phosphoric acid, 15% by volume of nitric acid, and 20% by volume of water. In such cases, the aqueous etching solution may comprise about 63.5% by weight of phosphoric acid, 14.5% by weight of nitric acid, and 13.7% by weight of water. In some embodiments, the aqueous etching solution may comprise 40% by weight
5 of phosphoric acid, 18% by weight of nitric acid, and 0.01% by weight surfactant. In some embodiments, the aqueous etching solution may comprise 60% by weight of phosphoric acid, 10% by weight of nitric acid, and 0.01% by weight surfactant. In some embodiments, the aqueous etching solution may comprise 6 to 12% by weight of hydrochloric acid, 25 to 35% by weight of phosphoric acid, and 0 to
10 1.5% by weight of nitric acid. In some embodiments, the aqueous etching solution may comprise 7% by weight of hydrochloric acid, 34% by weight of phosphoric acid, and 1% by weight of nitric acid. In some embodiments, the aqueous etching solution may comprise 10% by weight of hydrochloric acid, 26% by weight of phosphoric acid, and 1% by weight of nitric acid. In some
15 embodiments, the surfactant may be an anionic surfactant, such as, for example, DOWFAX™ 3B2, available from The Dow Chemical Company.

Optical Properties

In some embodiments, prior to etching the electrically conductive
20 film, the film may exhibit a preexisting set of optical properties, and after etching the film in at least one region, the etched region may exhibit a first consequent set of optical properties that are substantially identical to the preexisting set of optical properties. In some embodiments, after etching the film in at least one region, an unetched region may exhibit a second consequent set of optical properties that are
25 substantially identical to the first consequent set of optical properties. For the purpose of this application, the term “substantially identical” indicates differences that are not discernible to the unaided eye. In some embodiments, the absolute value of the differences between the preexisting set of optical properties and the first consequent set of optical properties or the first consequent set of optical
30 properties and the second consequent set of optical properties may be less than 0.3 (e.g. less than 0.2, less than 0.1) for optical differences that remain indiscernible to the unaided eye. In such cases, the pattern may be invisible.

Such a preexisting set of optical properties may, for example, comprise one or more of a preexisting total light transmission, a preexisting haze, a preexisting L^* value, a preexisting a^* value, or a preexisting b^* value. Such a first consequent set of optical properties may, for example, comprise one or more
5 of a first consequent total light transmission, a first consequent haze, a first consequent L^* value, a first consequent a^* value, or a first consequent b^* value. Such a second consequent set of optical properties may, for example, comprise one or more of a second consequent total light transmission, a second consequent haze, a second consequent L^* value, a second consequent a^* value, or a second
10 consequent b^* value. In some embodiments, the absolute value of the differences between the preexisting set b^* value and the first consequent b^* or the first consequent b^* value and the second consequent b^* value may be less than 0.3 (e.g. less than 0.2, less than 0.1) for optical differences that remain indiscernible or a pattern invisible to the unaided eye. In some embodiments, the absolute value
15 of the differences between the preexisting haze and the first consequent haze or the first consequent haze and the second consequent haze may be less than 0.3 (e.g. less than 0.2, less than 0.1) for optical differences that remain indiscernible or a pattern invisible to the unaided eye. In some embodiments, a combination of the absolute value of the differences between the preexisting set b^* value and the
20 first consequent b^* or the first consequent b^* value and the second consequent b^* value may be less than 0.3 (e.g. less than 0.2, less than 0.1) and the absolute value of the differences between the preexisting haze and the first consequent haze or the first consequent haze and the second consequent haze may be less than 0.3 (e.g. less than 0.2, less than 0.1) for optical differences that remain indiscernible
25 or a pattern invisible to the unaided eye.

EXEMPLARY EMBODIMENTS

U.S. Provisional Patent Application No. 61/876,417, filed September 11, 2013, entitled "WET ETCHING PATTERNING
30 COMPOSITIONS AND METHODS," which is hereby incorporated by reference in its entirety, disclosed the following 40 non-limiting exemplary embodiments:
A. A method comprising:

etching a film comprising electrically conductive structures according to a pattern using an aqueous etching solution to provide an etched region having a first conductivity and an unetched region having a second conductivity, the second conductivity being greater than the first conductivity,

5 wherein the aqueous etching solution comprises 25 to 65% by weight of phosphoric acid and 1 to 18% by weight of nitric acid.

B. The method according to embodiment A, wherein the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising 40 to 60% by weight of phosphoric acid, 10 to 15% by weight of nitric acid, and 0.005-0.05%
10 by weight of the surfactant.

C. The method according to either of embodiments A or B, wherein the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising 45% by weight of phosphoric acid, 15% by weight of nitric acid, and 0.01% by weight of the surfactant.

15 D. The method according to either of embodiments A or B, wherein the aqueous etching solution comprise a surfactant, the aqueous etching solution comprising 50% by weight of phosphoric acid, 10% by weight of nitric acid, and 0.01% by weight of the surfactant.

E. The method according to either of embodiments A or B, wherein the aqueous
20 etching solution comprises 63.5% by weight of phosphoric acid and 14.5% by weight of nitric acid.

F. The method according to any of either of embodiments A or B, wherein the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising 40% by weight of phosphoric acid, 18% by weight of nitric acid, and
25 0.01% by weight of the surfactant.

G. The method according to either of embodiments A or B, wherein the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising 60% by weight of phosphoric acid, 10% by weight of nitric acid, and 0.01% by weight of the surfactant.

30 H. The method according to embodiment A, wherein the aqueous etching solution further comprises hydrochloric acid, the aqueous etching solution comprising 6 to 12% by weight of hydrochloric acid, 25 to 35% by weight of phosphoric acid, and

0 to 1.5% by weight of nitric acid.

J. The method according to either of embodiments A and H, wherein the aqueous etching solution further comprises hydrochloric acid, the aqueous etching solution comprising 7% by weight of hydrochloric acid, 34% by weight of phosphoric acid, and 1% by weight of nitric acid.

K. The method according to either of embodiments A and H, wherein the aqueous etching solution further comprises hydrochloric acid, the aqueous etching solution comprising 10% by weight of hydrochloric acid, 26% by weight of phosphoric acid, and 1% by weight of nitric acid.

L. The method according to any of embodiments A-K, wherein prior to etching, the film exhibited a preexisting set of optical properties, and wherein after etching, the etched region exhibited a first consequent set of optical properties, and the unetched region exhibited a second consequent set of optical properties, and

further wherein the preexisting set of optical properties and the first consequent set of optical properties are substantially identical.

M. The method according to embodiment L, wherein the preexisting set of optical properties comprises a preexisting total light transmission and the first consequent set of optical properties comprises a first consequent total light transmission that is substantially identical to the preexisting total light transmission.

N. The method according to embodiment L, wherein the preexisting set of optical properties comprises a preexisting haze and the first consequent set of optical properties comprises a first consequent haze that is substantially identical to the preexisting haze.

P. The method according to embodiment L, wherein the preexisting set of optical properties comprises a preexisting L^* value and the first consequent set of optical properties comprises a first consequent L^* value that is substantially identical to the preexisting L^* value.

Q. The method according to embodiment L, wherein the preexisting set of optical properties comprises a preexisting a^* value and the first consequent set of optical properties comprises a first consequent a^* value that is substantially identical to the preexisting a^* value.

- R. The method according to embodiment L, wherein the preexisting set of optical properties comprises a preexisting b^* value and the first consequent set of optical properties comprises a first consequent b^* value that is substantially identical to the preexisting b^* value.
- 5 S. The method according to embodiment L, wherein the first consequent set of optical properties and the second consequent set of optical properties are substantially identical.
- T. The method according to embodiment 17, wherein the first consequent set of optical properties comprises a first consequent total light transmission and the
10 second consequent set of optical properties comprises a second consequent total light transmission that is substantially identical to the first consequent total light transmission.
- U. The method according to embodiment 17, wherein the first consequent set of optical properties comprises a first consequent haze and the second consequent set
15 of optical properties comprises a second consequent haze that is substantially identical to the first consequent haze.
- V. The method according to embodiment 17, wherein the first consequent set of optical properties comprises a first consequent L^* value and the second
20 consequent set of optical properties comprises a second consequent L^* value that is substantially identical to the first consequent L^* value.
- W. The method according to embodiment 17, wherein the first consequent set of optical properties comprises a first consequent a^* value and the second
consequent set of optical properties comprises a second consequent a^* value that is substantially identical to the first consequent a^* value.
- 25 X. The method according to embodiment 17, wherein the first consequent set of optical properties comprises a first consequent b^* value and the second consequent set of optical properties comprises a second consequent b^* value that is substantially identical to the first consequent b^* value.
- Y. The method according to any of embodiments A-X, wherein after etching, the
30 pattern is invisible to the unaided eye.
- Z. The method according to embodiment N, wherein the absolute value of the difference between the preexisting haze and the first consequent haze is less than

about 0.1.

AA. The method according to embodiment U, wherein the absolute value of the difference between the first consequent haze and the second consequent haze is less than about 0.1.

5 AB. The method according to embodiment R, wherein the absolute value of the difference between the preexisting b^* value and the first consequent b^* is less than about 0.1.

AC. The method according to embodiment X, wherein the absolute value of the difference between the first consequent b^* value and the second consequent b^*
10 value is less than about 0.1.

AD. The method according to either of embodiments N or U,
wherein the absolute value of the difference between the preexisting haze and the first consequent haze is less than about 0.1, and
wherein the absolute value of the difference between the first consequent
15 haze and the second consequent haze is less than about 0.1.

AE. The method according to either of embodiments R or X,
wherein the absolute value of the difference between the preexisting b^* value and the first consequent b^* is less than about 0.1, and
wherein the absolute value of the difference between the first consequent
20 b^* value and the second consequent b^* value is less than about 0.1.

AF. The method according to any of embodiments A-AE, wherein the electrically conductive structures comprise silver nanowires.

AG. The method according to either of embodiments A or B, wherein the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising
25 42.5% by weight of phosphoric acid, 15% by weight of nitric acid, and 0.01% by weight of the surfactant.

AH. The method according to either of embodiments A or B, wherein the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising 45% by weight of phosphoric acid, 13.75% by weight of nitric acid,
30 and 0.01% by weight of the surfactant.

AJ. The method according to either of embodiments A or B, wherein the aqueous etching solution comprises a surfactant, the aqueous etching solution comprising

45% by weight of phosphoric acid, 12.5% by weight of nitric acid, and 0.01% by weight of the surfactant.

AK. The method according to any of embodiments A-AJ, wherein the aqueous solution comprises a surfactant.

5 AL. The method according to any of embodiments A-AK, wherein the aqueous solution comprises an anionic surfactant.

AM. The method according to any of embodiments A-AL, wherein the aqueous solution comprises a surfactant, the surfactant comprising
decyl(sulfophenoxy)benzenesulfonic acid, disodium salt, and
10 oxybis(decylbenzenesulfonic acid).

AN. The method according to any of embodiments A-AM, wherein prior to etching the film, a mask is disposed onto the film, and wherein after etching the film, the mask is removed from the film by being dissolved in a solution.

15 AP. The method according to any of embodiments A-AN, wherein prior to etching the film, a mask is disposed onto the film, and wherein after etching the film, the mask is peeled from the film.

AQ. A method comprising:

20 etching a film comprising electrically conductive structures according to a pattern using an aqueous etching solution to provide an etched region having a first conductivity and an unetched region having a second conductivity, the second conductivity being greater than the first conductivity,

wherein the aqueous etching solution comprises 65 to 75% by weight of nitric acid.

25 AR. The method according to embodiment AQ, wherein the aqueous etching solution comprises 70% by weight of nitric acid.

EXAMPLES

Methods

30 Electrical conductivity of films was measured using a 707 Conductance Monitor (Delcom Instruments Inc.).

Percent transmittance and percent haze of films were measured according to the ASTM D-1003 test method using a BYK-Gardner HAZE-GARD PLUS hazemeter.

CIE L*, a*, and b* color metrics of films were measured using a
5 HunterLab ULTRASCAN[®] VIS colorimeter.

Screen printing was performed using a Model F1-12 precision screen printer (Systematic Automation, Farmington, CT) equipped with a 305 mesh screen, which is a rectangular or a square block of material.

10 **Materials**

All materials (e.g. phosphoric acid, nitric acid, hydrochloric acid, etc.) used in the following examples are readily available from standard commercial sources, such as Sigma-Aldrich Co. LLC. (St. Louis, Missouri) unless otherwise specified.

15 DOWFAX[™] 3B2 anionic surfactant is an aqueous solution comprising less than or equal to 38 % decyl(sulfophenoxy)benzenesulfonic acid, disodium salt, and less than or equal to 8 % oxybis(decylbenzenesulfonic acid), disodium salt (Dow Chemical).

FLEXX[™] 100 is a transparent conductive film comprising silver
20 nanowires (Carestream Health, Inc., Rochester, NY).

GC-UV-60 ink is a screen printable UV curable resist (Green Cure Technology Corporation, Ltd., Taiwan).

LP400-BL8 ink is a screen printable UV curable resist (Green Cure Technology Corporation, Ltd., Taiwan).

25 HOYO MI-B077 is a screen printable and thermal curable mask available from Hoyo-Chemical. It is reported to be a peelable mask that does not require a stripping agent.

Analysis

30 Samples of film having etched regions with 0 conductivity readings were selected for further calculations of changes in percent haze and CIE L*, a*, b* and visual observation of invisibility of the pattern. Films visually observed to

be invisible tended to have changes in percent haze and CIE L*, a*, and b* of less than 0.1.

Example 1

5 Samples of FLEXX™ 100 transparent conductive film (Lot 244-3133, Carestream Health, Inc., Rochester, NY), which had a resistivity of 100 Ohms, were annealed at 150°C for 30 minutes. Initial measurements of electrical conductivity and percent haze were recorded. Masks were disposed onto the samples by being screen printed with a GC-UV-60A ink and cured with a UV
10 lamp at about 230 mJ/cm² with one pass at a rate of 30 ft/m. The samples were dipped into a various etchant compositions (varying wt% of H₃PO₄, HNO₃, HCl, or H₂O) at a selected temperatures (room temperature of about 21°C, 40°C, or 45°C) for selected times (60, 120, or 180 seconds). To remove excess etchant, the samples were washed with distilled water. The masks were removed by dipping
15 the sample into a 35°C 3% (w/v) aqueous solution of sodium hydroxide for 71 seconds. The samples were washed with distilled water and oven dried at 230°F for 2 to 3 minutes. Final measurements of electrical conductivity and percent haze were recorded. Table 1 shows the electrical conductivity and haze of the etched region after stripping. (RT indicates “room temperature,” while NC denotes “not
20 checked.”

Table 1

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /HCl/ H ₂ O	Etchant Temp (°C)	Etching Time (s)	Eddy Current (mmhos)		Delta Haze
				initial	etched	
1-1	0/1/21/78	RT	60	11.02	6.56	-0.06
1-2	0/1/21/79	RT	60	11.73	6.98	-0.11
1-3	0/1/21/80	RT	120	12.53	0.52	-0.18
1-4	0/1/21/81	RT	120	11.6	0.61	-0.2
1-5	0/1/21/82	RT	180	12.18	0	-0.3
1-6	0/1/21/83	RT	180	12.08	0	-0.31
1-7	34/1/7/58	RT	60	12.85	11.1	NC
1-8	34/1/7/59	RT	60	12.47	12.33	NC
1-9	34/1/7/60	RT	120	10.65	10.59	NC
1-10	34/1/7/61	RT	120	11.26	10.54	NC
1-11	34/1/7/62	RT	180	10.83	9.14	NC
1-12	34/1/7/63	RT	180	11.95	12.14	NC
1-13	26/1/10/63	RT	60	12.9	8.92	NC
1-14	26/1/10/64	RT	60	12.4	11	NC
1-15	26/1/10/65	RT	120	11.31	10.59	NC
1-16	26/1/10/66	RT	120	11.43	10.71	NC
1-17	26/1/10/67	RT	180	9.67	9.98	NC
1-18	26/1/10/68	RT	180	9.01	8.54	NC
1-19	0/1/21/78	40	60	11.4	0	-0.51
1-20	0/1/21/79	40	60	11.37	0	-0.51
1-21	0/1/21/80	40	120	12.11	0	-0.52
1-22	0/1/21/81	40	120	11.04	0	-0.61
1-23	0/1/21/82	40	180	11.28	0	-0.57
1-24	0/1/21/83	40	180	11.91	0	-0.54
1-25	34/1/7/58	40	60	12.11	11.22	NC
1-26	34/1/7/59	40	60	10.66	11.4	NC
1-27	34/1/7/60	40	120	12.06	2.94	-0.11
1-28	34/1/7/61	40	120	12.26	2.68	-0.17
1-29	34/1/7/62	40	180	12.38	0	0.01
1-30	34/1/7/63	40	180	11.72	0	-0.12
1-31	26/1/10/63	40	60	12.31	6.13	-0.06
1-32	26/1/10/64	40	60	10.53	6.27	-0.02
1-33	26/1/10/65	40	120	11.29	0	0.06
1-34	26/1/10/66	40	120	12.03	0	-0.21
1-35	26/1/10/67	40	180	11.8	0	-0.24
1-36	26/1/10/68	40	180	11.92	0	-0.29

Table 1 (cont'd)

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /HCl/ H ₂ O	Etchant Temp (°C)	Etching Time (s)	Eddy Current (mmhos)		Delta Haze
				initia l	etched	
1-37	34/1/7/58	45	60	11.56	2.43	-0.1
1-38	34/1/7/59	45	60	12.55	3.76	-0.27
1-39	34/1/7/60	45	120	10.7	0	-0.28
1-40	34/1/7/61	45	120	11.61	0	-0.33
1-41	34/1/7/62	45	180	11.68	0	-0.33
1-42	34/1/7/63	45	180	11.82	0	-0.49

Example 2

Samples of FLEXX™ 100 transparent conductive film (Lot 308-3112, Carestream Health, Inc., Rochester, NY), which had a resistivity of 100 Ohms, were annealed at 150°C for 30 minutes. Initial measurements of electrical conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded. Masks were disposed onto the samples by being screen printed with an LP400-BL8 ink and cured at with a UV lamp at about 230 mJ/cm² with one pass at a rate of 20 ft/m. The masked samples were dipped into various etchant compositions (varying wt% of H₃PO₄, HNO₃, HCl, or H₂O) at selected temperatures (room temperature of about 21°C or 35°C) for selected times (30, 60, 120, or 180 seconds). To remove excess etchant, the samples were washed with distilled water and dried with compressed air using a blower. Final measurements of conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded for the etched areas. Table 2 shows the electrical conductivity, change in percent haze, and change in b* of the etched region after etching. (RT indicates “room temperature.”)

Table 2

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /HCl/H ₂ O	Etching Temp (°C)	Etching Time (s)	Eddy Current (mmhos)		Delta Haze	Delta b*
				initial	etched		
2-1	0/18/0/0.01	RT	30	11.78	11.9	-0.01	-0.08
2-2	0/18/0/0.01	RT	60	12.09	12.2	0	-0.04
2-3	0/18/0/0.01	RT	90	12.18	12.2	0.19	-0.06
2-4	0/18/0/0.01	RT	120	9.73	9.92	-0.02	-0.07
2-5	40/18/0/0.01	RT	30	9.64	10.1	-0.02	-0.26
2-6	40/18/0/0.01	RT	60	10.21	9.64	-0.07	-0.16
2-7	40/18/0/0.01	RT	90	12.12	2.16	-0.01	-0.07
2-8	40/18/0/0.01	RT	120	11.87	0.25	-0.01	-0.02
2-9	0/0/15/0.01	RT	30	11.69	11.8	0.06	-0.06
2-10	0/0/15/0.01	RT	60	11.66	12.1	-0.05	0.11
2-11	0/0/15/0.01	RT	90	11.7	12	0.02	-0.14
2-12	0/0/15/0.01	RT	120	11.3	11.5	-0.01	0.02
2-13	40/0/15/0.01	RT	30	11.68	12.7	-0.01	-0.06
2-14	40/0/15/0.01	RT	60	11.82	12.6	-0.13	-0.16
2-15	40/0/15/0.01	RT	90	11.73	12.9	-0.04	-0.17
2-16	40/0/15/0.01	RT	120	12.2	13.7	0.03	-0.19
2-17	70/0/0/0.01	RT	30	10.11	10.28	0.08	-0.04
2-18	70/0/0/0.01	RT	60	10.14	10.75	0.06	-0.03
2-19	70/0/0/0.01	RT	90	10.21	10.69	0.09	0
2-20	70/0/0/0.01	RT	120	11.61	12.27	0.04	-0.05
2-21	0/70/0/0.01	RT	30	11.7	0	-0.19	0.11
2-22	0/70/0/0.01	RT	60	11.34	0	-0.31	-0.34

Table 2 (cont'd)

Sample	Etchant Composition (wt%) HCl/HNO ₃ / H ₃ PO ₄ /H ₂ O	Etchant Temp (°C)	Etching Time (s)	Eddy Current (mmhos)		Delta Haze	Delta b*
				initial	etched		
2-23	0/70/0/0.01	RT	90	10.43	0	0.01	-0.45
2-24	0/70/0/0.01	RT	120	10.41	0	0.5	-0.68
2-25	0/0/37/0.01	RT	30	10.23	11.49	0.14	-0.46
2-26	0/0/37/0.01	RT	60	10.01	11.49	0.26	-0.34
2-27	0/0/37/0.01	RT	90	10.04	11.46	0.05	-0.28
2-28	0/0/37/0.01	RT	120	10.01	11.44	0.23	-0.11
2-29	0/18/0/0.01	35	30	11.92	12.1	-0.01	-0.17
2-30	0/18/0/0.01	35	60	11.65	11.8	-0.03	-0.17
2-31	0/18/0/0.01	35	90	12.5	12.9	0.01	-0.07
2-32	0/18/0/0.01	35	120	12.23	12.1	0.03	-0.1
2-33	40/18/0/0.01	35	30	11.9	0	-0.03	0
2-34	40/18/0/0.01	35	60	10.07	0	-0.08	-0.19
2-35	40/18/0/0.01	35	90	10.13	0	-0.01	-0.16
2-36	40/18/0/0.01	35	120	10.24	0	-0.05	-0.21
2-37	0/0/15/0.01	35	30	9.86	10	0.05	-0.1
2-38	0/0/15/0.01	35	60	10.24	10.7	-0.04	-0.2
2-39	0/0/15/0.01	35	90	10.39	10.9	0	-0.21
2-40	0/0/15/0.01	35	120	10.04	10.9	-0.03	-0.13
2-41	40/0/15/0.01	35	30	10.76	11.6	0.03	-0.07
2-42	40/0/15/0.01	35	60	10.59	11.8	0.01	-0.26
2-43	40/0/15/0.01	35	90	11.72	13.4	0.06	-0.18
2-44	40/0/15/0.01	35	120	11.35	12.7	0.07	-0.12

Example 3

Samples of FLEXX™ 100 transparent conductive film (Lot 308-3112, Carestream Health, Inc., Rochester, NY), which had a resistivity of 100 Ohms, were annealed at 150°C for 30 minutes. Initial measurements of electrical conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded. Masks were screen printed onto the samples and cured with a UV lamp at 230 mJ/cm² with one pass at a rate of 20 ft/m. The masked samples were dipped into a selected etchant composition (varying wt% of H₃PO₄, HNO₃, HCl or 3B2, or H₂O) at selected temperatures (room temperature of about 21°C, 35°C, or 40°C) for selected times (30, 60, 90, or 120 seconds). To remove excess etchant, the samples were washed with distilled water and dried with compressed air using a blower. The masks were removed by dipping the samples into an aqueous solution of 5% (w/v) sodium hydroxide. The samples were washed with distilled water and air dried. Final measurements of conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded for the etched and masked areas. Tables 3A and 3B show the electrical conductivity, change in percent haze, and change in b* of the etched regions after the mask were removed and of the masked regions after etching. (RT indicates “room temperature.”)

Table 3A

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /HCl/H ₂ O	Etchant Temp (°C)	Etching Time (s)	Electrical Conductivity (mmhos)		Delta Haze etched	Delta b* etched	Delta Haze masked	Delta b* masked
				initial	etched				
3-1	40/15/0/0.01	RT	30	9.14	9.09	0.34	-0.08	0.13	-0.11
3-2	40/15/0/0.01	RT	60	9.04	8.61	0.47	-0.06	0.12	0.05
3-3	40/15/0/0.01	RT	90	9.8	9.14	0.41	-0.14	0.14	-0.05
3-4	40/15/0/0.01	RT	120	10.27	8.33	0.42	-0.11	0.2	-0.23
3-5	40/10/0/0.01	RT	30	9.42	9.3	0.73	-0.14	0.47	-0.14
3-6	40/10/0/0.01	RT	60	8.69	8.37	0.19	-0.11	0.06	0.16
3-7	40/10/0/0.01	RT	90	8.12	7.91	0.2	-0.15	0.06	-0.35
3-8	40/10/0/0.01	RT	120	7.98	7.88	0.23	-0.06	0.05	-0.32
3-9	60/18/0/0.01	RT	30	8.34	0	0.21	0.17	0.17	-0.05
3-10	60/18/0/0.01	RT	60	11.22	0	0.75	0.33	0.32	-0.08
3-11	60/18/0/0.01	RT	90	11.16	0	0.07	-0.01	-0.01	0.13
3-12	60/18/0/0.01	RT	120	11.45	0	-0.38	-0.18	-0.13	0.34
3-13	60/15/0/0.01	RT	30	10.07	0.7	0.25	0.37	0.31	-0.1
3-14	60/15/0/0.01	RT	60	9.8	0	0.37	0.3	0.16	-0.26
3-15	60/15/0/0.01	RT	90	10.16	0	-0.08	0.31	0.17	-0.14
3-16	60/15/0/0.01	RT	120	10.87	0	-0.3	0.21	0.27	-0.07
3-17	60/10/0/0.01	RT	30	10.95	0	0.14	0.06	0.18	-0.07
3-18	60/10/0/0.01	RT	60	11.42	0	0.19	0.11	0.22	-0.07
3-19	60/10/0/0.01	RT	90	10.8	0	0.11	0.1	0.02	-0.12
3-20	60/10/0/0.01	RT	120	10.54	0	-0.02	0.21	-0.01	-0.09
3-21	40/15/0/0.01	40	30	10.55	0	0.29	-0.08	0	0.12

Table 3A (cont'd)

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /HCl/H ₂ O	Etchant Temp (°C)	Etching Time (s)	Electrical Conductivity (mmhos)		Delta Haze etched	Delta b* etched	Delta Haze masked	Delta b* masked
				initial	etched				
3-22	40/15/0/0.01	40	60	9.66	0.24	0.15	-0.07	0	-0.02
3-23	40/15/0/0.01	40	90	9.39	0	0.06	-0.05	0.07	-0.09
3-24	40/15/0/0.01	40	120	10.11	0	0.34	-0.01	0.12	0.02
3-25	40/10/0/0.01	40	30	10.74	10.39	0.23	-0.14	0.06	-0.16
3-26	40/10/0/0.01	40	60	9.76	9.6	0.1	-0.17	0.08	-0.1
3-27	40/10/0/0.01	40	90	9.55	9.06	0.18	-0.11	0.34	0
3-28	40/10/0/0.01	40	120	9.34	8.62	0.45	-0.16	0.08	-0.13
3-29	60/18/0/0.01	40	30	9.08	0	-0.69	-0.29	-0.1	-0.08
3-30	60/18/0/0.01	40	60	9.24	0	-0.56	-0.63	-0.57	-0.31
3-31	60/18/0/0.01	40	90	8.26	0	-0.67	-0.64	-0.72	-0.6
3-32	60/18/0/0.01	40	120	8.31	0	-0.72	-0.51	-0.78	-0.72
3-33	60/15/0/0.01	40	30	10.12	0	0	0.58	0.42	-0.27
3-34	60/15/0/0.01	40	60	10.02	0	0	-0.12	-0.57	-0.67
3-35	60/15/0/0.01	40	90	10.242	0	0	0.2	-0.85	-0.82
3-36	60/15/0/0.01	40	120	11.07	0	0	-0.05	-0.78	-0.81
3-37	60/10/0/0.01	40	30	8.36	0	-0.19	0.15	-0.11	-0.26
3-38	60/10/0/0.01	40	60	10.26	0	-0.23	0.06	-0.07	-0.06
3-39	60/10/0/0.01	40	90	9.87	0	-0.39	0	-0.01	0.05
3-40	60/10/0/0.01	40	120	9.74	0	-0.49	-0.18	-0.14	0.04

Table 3B

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /3B2/H ₂ O	Etchant Temp (°C)	Etching Time (s)	Electrical Conductivity (mmhos)		Delta Haze etched	Delta b* etched	Delta Haze masked	Delta b* masked
				initial	etched				
3-41	40/15/0.01/44.99	35	30	10.31	9.86	-0.06	-0.07	0.93	0.14
3-42	40/15/0.01/44.99	35	60	11.07	9.62	-0.01	-0.07	0.23	-0.06
3-43	40/15/0.01/44.99	35	90	9.83	0.71	0.12	0.02	1.58	-0.19
3-44	40/15/0.01/44.99	35	120	10.25	1.29	0.03	0	0.26	-0.16
3-45	45/15/0.01/39.99	35	30	10.42	0	-0.13	-0.03	-0.09	-0.12
3-46	45/15/0.01/39.99	35	60	10.9	0	0.36	-0.07	0.21	-0.05
3-47	45/15/0.01/39.99	35	90	9.1	0	0.14	-0.1	-0.05	0.05
3-48	45/15/0.01/39.99	35	120	8.95	0	0.05	-0.14	0.08	-0.25
3-49	50/15/0.01/34.99	35	30	9.1	0	-0.06	-0.05	0.11	0.11
3-50	50/15/0.01/34.99	35	60	8.81	0	-0.01	-0.13	0.18	0.01
3-51	50/15/0.01/34.99	35	90	9.34	0	0.01	0.03	0.36	0.26
3-52	50/15/0.01/34.99	35	120	8.43	0	0.01	-0.2	0.2	0.06
3-53	45/10/0.01/44.99	35	30	10.05	9.88	0.01	-0.11	0.31	-0.13
3-54	45/10/0.01/44.99	35	60	10.41	10.04	-0.01	-0.14	1.56	-0.131
3-55	45/10/0.01/44.99	35	90	10.49	10.05	0.02	-0.07	-0.04	0.01
3-56	45/10/0.01/44.99	35	120	10.25	9.88	0.01	-0.11	0.1	-0.08
3-57	50/10/0.01/39.99	35	30	10.09	9.94	0.13	-0.13	0.25	-0.1
3-58	50/10/0.01/39.99	35	60	10.25	8.47	-0.01	-0.11	0.29	-0.09
3-59	50/10/0.01/39.99	35	90	11.81	1.3	-0.03	0	0.13	0.12
3-60	50/10/0.01/39.99	35	120	9.19	0	0.1	-0.14	0.18	-0.14

Table 3B (cont'd)

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /3B2/H ₂ O	Etchant Temp °C	Etching Time (s)	Electrical Conductivity (mmhos)		Delta Haze		Delta b*		Delta Haze		Delta b*	
				initial	etched	etched	etched	etched	masked	masked	masked		
3-61	55/10/0.01/34.99	35	30	10.93	0	0.08	0.09	0.05	-0.25	0.05	-0.37		
3-62	55/10/0.01/34.99	35	60	9.84	0	-0.04	0.13	0.06	-0.37	0.06	-0.37		
3-63	55/10/0.01/34.99	35	90	11.14	0	-0.16	0.14	0.65	-0.04	0.65	-0.04		
3-64	55/10/0.01/34.99	35	120	9.38	0	-0.33	0.06	-0.04	-0.27	-0.04	-0.27		
3-65	40/15/0.01/44.99	40	30	10.11	9.16	0.12	-0.15	0.56	-0.14	0.56	-0.14		
3-66	40/15/0.01/44.99	40	60	9.85	0.81	0.63	-0.12	0.67	-0.14	0.67	-0.14		
3-67	40/15/0.01/44.99	40	90	10.74	0	0.37	-0.19	0.19	-0.14	0.19	-0.14		
3-68	40/15/0.01/44.99	40	120	10.29	0	0.03	-0.07	0.32	-0.07	0.32	-0.07		
3-69	45/15/0.01/39.99	40	30	10.42	0	0.3	0.03	0.04	-0.25	0.04	-0.25		
3-70	45/15/0.01/39.99	40	60	10.17	0	0.48	-0.06	1.22	-0.16	1.22	-0.16		
3-71	45/15/0.01/39.99	40	90	8.57	0	-0.01	-0.03	0.23	-0.35	0.23	-0.35		
3-72	45/15/0.01/39.99	40	120	10.06	0	0.01	-0.11	0.28	-0.18	0.28	-0.18		
3-73	50/15/0.01/34.99	40	30	9.21	0	-0.21	0.13	0.94	-0.37	0.94	-0.37		
3-74	50/15/0.01/34.99	40	60	10.61	0	-0.55	0.11	0.46	-0.32	0.46	-0.32		
3-75	50/15/0.01/34.99	40	90	9.25	0	-0.68	-0.02	-0.08	-0.34	-0.08	-0.34		
3-76	50/15/0.01/34.99	40	120	10.51	0	-0.66	-0.58	-0.05	0.4	-0.05	0.4		
3-77	45/10/0.01/44.99	40	30	10.07	9.44	0.19	-0.16	0.08	-0.13	0.08	-0.13		
3-78	45/10/0.01/44.99	40	60	9.27	8.93	0.02	-0.11	0.11	-0.24	0.11	-0.24		
3-79	45/10/0.01/44.99	40	90	10.01	9.39	0.08	-0.15	0.91	0.04	0.91	0.04		
3-80	45/10/0.01/44.99	40	120	9.61	9.11	0.04	-0.15	0.07	-0.17	0.07	-0.17		

Table 3B (cont'd)

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /3B2/H ₂ O	Etchant Temp °C	Etching Time (s)	Electrical Conductivity (mmhos)		Delta Haze etched	Delta b* etched	Delta Haze masked	Delta b* masked
				initial	etched				
3-81	50/10/0.01/39.99	40	30	9.94	8.63	0.01	-0.17	-0.11	0
3-82	50/10/0.01/39.99	40	60	9.97	1.07	0.04	-0.1	0.02	-0.12
3-83	50/10/0.01/39.99	40	90	9.41	0	0.01	-0.06	0.11	-0.18
3-84	50/10/0.01/39.99	40	120	8.3	0	0.02	-0.06	0.19	-0.3
3-85	55/10/0.01/34.99	40	30	8.49	0	0.5	0.1	0.27	-0.38
3-86	55/10/0.01/34.99	40	60	9.35	0	-0.06	0	0.32	-0.3
3-87	55/10/0.01/34.99	40	90	8.51	0	-0.03	-0.01	-0.15	-0.23
3-88	55/10/0.01/34.99	40	120	9.36	0	-0.03	0.07	0.03	-0.34

Example 4

Several samples of FLEXX™ 100 transparent conductive film (Lots 304-3114, 308-3111, 308-3112, 322-3120, 318-3127, 313-3120, 326-3107, 304-3114, Carestream Health, Inc., Rochester, NY) were subjected to a similar process as described in Example 3, except that additional measurements of conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded for the etched region prior to stripping the mask from the film and visual observations of invisibility of the stripped film were recorded. Table 4 shows the electrical conductivity, change in percent haze, change in b* of the etched region before and after the mask was removed and of the masked region after etching. (EC denotes “electrical conductivity” in mmhos.)

Table 4

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /3B2/H ₂ O	Etching Temp (°C)	Etching Time (s)	Initial EC	Before Strip			After Strip			ΔHaze etched (After Strip - Before Strip)	
					EC etched	ΔHaze	Δb*	ΔHaze etched	Δb* etched	ΔHaze masked		Δb* masked
4-1	45/15/0.01/39.99	35	30	10.06	7.85	0.23	-0.01	-0.7	0.02	0.29	0	0.09
4-2	45/15/0.01/39.99	35	60	10.09	1.42	0.52	-0.03	-0.6	0.01	0.18	-0.08	-0.16
4-3	45/15/0.01/39.99	35	90	10.72	0	0.09	-0.01	-0.8	-0.03	0.45	-0.07	0.4
4-4	45/15/0.01/39.99	35	120	10.24	0	-0.05	0.04	-0.4	0.06	-0.04	0.08	0.13
4-5	45/15/0.01/39.99	35	30	11.42	0	0.17	0.05	-0.9	-0.05	0.27	-0.1	-0.07
4-6	45/15/0.01/39.99	35	60	10.46	0	0.05	-0.04	-0.6	-0.11	-0.09	0.04	-0.02
4-7	45/15/0.01/39.99	35	90	11.66	0	0.07	0.05	-0.8	0.04	0.46	-0.03	0.46
4-8	45/15/0.01/39.99	35	120	9.33	0	0.03	-0.04	-0.6	0	0.18	0.08	0.34
4-9	45/15/0.01/39.99	35	30	9.96	0	0.05	0	-0.7	-0.08	0.13	-0.16	0.07
4-10	45/15/0.01/39.99	35	60	9.7	0	-0.2	-0.12	-0.7	-0.05	0.06	-0.24	0.33
4-11	45/15/0.01/39.99	35	90	9.56	0	0	-0.04	-0.5	-0.03	0.16	-0.13	0.11
4-12	45/15/0.01/39.99	35	120	11.52	0	0.06	0.09	-0.6	-0.07	0.38	0.03	0.3
4-13	45/15/0.01/39.99	35	30	8.97	0	0.15	-0.16	-0.7	-0.09	0.43	-0.32	0.25
4-14	45/15/0.01/39.99	35	60	8.99	0	0.21	-0.11	-0.7	-0.11	0.26	-0.25	0.18
4-15	45/15/0.01/39.99	35	90	10.27	0	0.15	-0.04	-0.7	-0.08	0.21	-0.07	0.23
4-16	45/15/0.01/39.99	35	120	10.15	0	0.19	-0.11	-0.6	-0.05	0.52	-0.31	0.45
4-17	45/15/0.01/39.99	35	30	9.96	1.99	-0.04	-0.14	-0.8	0.02	-0.07	-0.1	0.07
4-18	45/15/0.01/39.99	35	60	9.56	0	0.07	-0.15	-0.8	0.07	-0.03	-0.29	0.06
4-19	45/15/0.01/39.99	35	90	10.95	0	0	-0.12	-0.9	0.04	-0.01	-0.14	0.12
4-20	45/15/0.01/39.99	35	120	9.34	0	-0.04	-0.23	-0.8	0.08	-0.13	-0.36	0.11

Table 4 (cont'd)

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /3B2/H ₂ O	Etching Temp (°C)	Etching Time (s)	Initial EC	Before Strip			After Strip				ΔHaze etched (After Strip - Before Strip)
					EC etched	ΔHaze	Δb*	ΔHaze etched	Δb* etched	ΔHaze masked	Δb* masked	
4-21	45/15/0.01/39.99	35	30	9.63	4.33	-0.13	-0.17	-0.8	0.03	-0.21	0.18	0.04
4-22	45/15/0.01/39.99	35	60	9.59	1.3	0.04	-0.09	-0.8	0.05	0	0.22	0.14
4-23	45/15/0.01/39.99	35	90	9.76	0	-0.03	0.04	-0.7	0.03	-0.22	0.31	0.02
4-24	45/15/0.01/39.99	35	120	10.13	0	0.05	-0.17	-0.8	0.01	-0.33	0.07	-0.01
4-25	45/15/0.01/39.99	35	30	8.41	8.04	0.22	-0.17	-0.7	-0.06	0.25	0.01	0.19
4-26	45/15/0.01/39.99	35	60	7.55	5.91	0.19	-0.11	-0.7	-0.12	0.37	0	0.23
4-27	45/15/0.01/39.99	35	90	7.87	6.77	0.31	-0.12	-0.6	-0.13	0.49	-0.04	0.28
4-28	45/15/0.01/39.99	35	120	7.85	0.2	0.16	-0.1	-0.7	-0.17	0.23	-0.07	0.1
4-29	45/15/0.01/39.99	40	30	10.73	3.59	0.51	-0.01	-0.8	0.07	0.41	0.01	0.27
4-30	45/15/0.01/39.99	40	60	12.13	0	0.28	0.05	-1	-0.05	0.86	0.13	0.78
4-31	45/15/0.01/39.99	40	90	10.69	0	0.18	0.04	-0.8	0	0.35	0.01	0.24
4-32	45/15/0.01/39.99	40	120	11.46	0	-0.05	0.09	-1	0.11	0.23	-0.03	0.3
4-33	45/15/0.01/39.99	40	30	9.13	0	0.22	0.01	-0.9	0.02	0.08	-0.16	0.13
4-34	45/15/0.01/39.99	40	60	9.71	0	0.27	0.02	-0.9	-0.02	-0.07	-0.17	-0.05
4-35	45/15/0.01/39.99	40	90	9.71	0	0.15	-0.05	-1	0.01	-0.01	-0.17	-0.07
4-36	45/15/0.01/39.99	40	120	9.29	0	0.23	-0.03	-0.9	-0.02	-0.12	-0.27	-0.13
4-37	45/15/0.01/39.99	40	30	9.82	0	0.05	-0.02	-0.7	-0.08	0.09	-0.19	0.11
4-38	45/15/0.01/39.99	40	60	12.45	0	0.06	0.15	-0.7	-0.11	0.32	0.04	0.25
4-39	45/15/0.01/39.99	40	90	9.61	0	0.15	-0.06	-0.6	-0.05	0.21	-0.17	0.27
4-40	45/15/0.01/39.99	40	120	11.67	0	-0.09	0.1	-0.6	-0.11	0.27	0.02	0.37

Table 4 (cont'd)

Sample	Etchant Composition (wt%) H ₃ PO ₄ /HNO ₃ /3B2/H ₂ O	Etching Temp (°C)	Etching Time (s)	Initial EC	Before Strip			After Strip				ΔHaze etched (After Strip - Before Strip)
					EC etched	ΔHaze	Δb*	ΔHaze etched	Δb* etched	ΔHaze masked	Δb* masked	
4-41	45/15/0.01/39.99	40	30	10.3	0	0	-0.1	-0.7	-0.06	0.33	-0.21	0.35
4-42	45/15/0.01/39.99	40	60	9.89	0	0.11	-0.11	-0.8	-0.08	0.11	-0.36	0.08
4-43	45/15/0.01/39.99	40	90	10.42	0	0.07	-0.07	-0.7	-0.1	0.15	-0.15	0.15
4-44	45/15/0.01/39.99	40	120	10.05	0	0.18	-0.15	-0.7	-0.05	0.24	-0.37	0.24
4-45	45/15/0.01/39.99	40	30	8.79	0	0.1	-0.2	-0.9	0.01	0.15	-0.3	0.18
4-46	45/15/0.01/39.99	40	60	11.01	0	-0.15	-0.13	-0.6	0.13	-0.09	-0.29	0.32
4-47	45/15/0.01/39.99	40	90	8.16	0	-0.07	-0.1	-1	0.05	-0.03	-0.22	0.19
4-48	45/15/0.01/39.99	40	120	10.73	1.9	0.05	-0.29	-0.8	-0.01	0.07	-0.65	0.13
4-49	45/15/0.01/39.99	40	30	10.55	0	0.02	-0.09	-0.7	0.07	0	0.05	0.17
4-50	45/15/0.01/39.99	40	60	8.56	0	-0.07	-0.04	-0.7	0.02	-0.11	0.19	0.07
4-51	45/15/0.01/39.99	40	90	10.19	0	-0.13	-0.09	-0.6	0.05	-0.09	0.28	0.18
4-52	45/15/0.01/39.99	40	120	9.84	0	-0.01	-0.05	-0.7	0.01	-0.08	-0.21	0.19
4-53	45/15/0.01/39.99	40	30	8.74	6.61	0.16	-0.08	-0.7	-0.13	0.39	0.06	0.33
4-54	45/15/0.01/39.99	40	60	8.56	0	0.24	-0.06	-0.7	-0.1	0.18	-0.07	0.11
4-55	45/15/0.01/39.99	40	90	8.96	0.08	0.41	-0.12	-0.8	-0.01	0.37	-0.12	0.4
4-56	45/15/0.01/39.99	40	120	8.87	0	-0.2	-0.09	-1.2	-0.11	-0.06	-0.07	0.26

Example 5

Samples of FLEXX™ 100 transparent conductive film (Samples 5-1 to 5-5 and 5-16 to 5-20 from Lot 350-3102; Samples 5-6 to 5-10 and 5-21 to 5-25 from Lot 403-3104; Samples 5-11 to 5-15 and 5-26 to 5-30 from Lot 403-3105; Carestream Health, Inc., Rochester, NY), which had a resistivity of 100 Ohms, were annealed at 150°C for 30 minutes. Initial measurements of electrical conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded. Rectangular peelable masks were disposed onto the samples by screen printing a substance known as HOYO MI-B077 and cured for 20 minutes at 130°C. The masked samples were dipped into a selected etchant composition (varying wt% of H₃PO₄, HNO₃, DOWFAX™ 3B2 surfactant) at 30°C for selected times (30, 60, 90, 120, or 150 seconds). To remove excess etchant, the samples were washed with distilled water and dried with compressed air using a blower. The masks were removed by being peeled from the samples. The samples were washed with distilled water and air dried. Final measurements of conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded for the etched and masked areas. Table 5 shows the percent change in electrical conductivity, change in percent haze, and change in b* of the etched regions after the mask were removed and of the masked regions after etching.

Table 5

Sample	Etchant Composition (wt%) HPO ₄ /HNO ₃ /3B2	Etching Time (s)	% EC Change (etch)	% EC Change (mask)	ΔH (etch)	ΔH (mask)	Δb^* (etch)	Δb^* (mask)
5-1	42.5/15/0.01	30	-1.99	-5.40	0	-0.42	-0.23	-0.16
5-2	42.5/15/0.01	60	15.44	-1.79	0	-0.25	-0.01	-0.08
5-3	42.5/15/0.01	90	30.65	-2.85	0.01	-0.32	0	0.09
5-4	42.5/15/0.01	120	100.00	-0.90	-0.07	-0.36	0.29	0.12
5-5	42.5/15/0.01	150	100.00	0.64	-0.15	-0.24	0.36	-0.21
5-6	42.5/15/0.01	30	1.17	-5.39	0	0.07	0.01	0.31
5-7	42.5/15/0.01	60	13.18	-2.09	-0.02	0.01	0.05	-0.03
5-8	42.5/15/0.01	90	30.65	-5.20	-0.02	0.04	0.16	0.16
5-9	42.5/15/0.01	120	74.43	0.17	-0.01	0.12	0.19	0.1
5-10	42.5/15/0.01	150	100.00	-0.68	-0.11	-0.03	0.32	0.18
5-11	42.5/15/0.01	30	3.72	-6.88	-0.01	0.14	0.05	0.1
5-12	42.5/15/0.01	60	28.26	-5.95	-0.03	0.17	0.02	-0.11
5-13	42.5/15/0.01	90	29.33	-2.49	-0.04	0.25	0.05	0.09
5-14	42.5/15/0.01	120	56.93	0.78	-0.02	0.36	0.06	0.02
5-15	42.5/15/0.01	150	99.43	0.25	-0.09	0.23	0.27	0.07

Table 5 (cont'd)

Sample	Etchant Composition (wt%) HPO ₄ /HNO ₃ /3B2	Etching Time (s)	% EC Change (etch)	% EC Change (mask)	ΔH (etch)	ΔH (mask)	Δb^* (etch)	Δb^* (mask)
5-16	45/15/0.01	30	2.10	-4.00	-0.02	-0.44	-0.13	-0.15
5-17	45/15/0.01	60	38.55	-0.09	0	-0.33	0.02	0.12
5-18	45/15/0.01	90	99.39	1.13	-0.04	-0.24	0.18	-0.34
5-19	45/15/0.01	120	100.00	-0.09	-0.21	-0.32	0.32	0.12
5-20	45/15/0.01	150	100.00	0.45	-0.24	-0.47	0.39	-0.63
5-21	45/15/0.01	30	21.57	-1.93	-0.01	-0.02	0.04	0
5-22	45/15/0.01	60	89.32	-3.85	-0.07	0.01	0.3	0.07
5-23	45/15/0.01	90	100.00	-0.44	-0.11	-0.04	0.3	0.03
5-24	45/15/0.01	120	100.00	-2.04	-0.39	0.13	0.27	0.09
5-25	45/15/0.01	150	100.00	3.13	-0.57	0.04	0.03	0.08
5-26	45/15/0.01	30	23.71	-6.55	-0.05	0.1	0.05	0.05
5-27	45/15/0.01	60	100.00	-3.02	-0.26	0.12	0.09	-0.28
5-28	45/15/0.01	90	100.00	-3.28	-0.46	0.21	0.02	-0.14
5-29	45/15/0.01	120	100.00	0.98	-0.72	0.26	-0.48	-0.34
5-30	45/15/0.01	150	100.00	2.16	-0.69	0.22	-0.21	0.07

Example 6

Samples of FLEXX™ 100 transparent conductive film (Samples 6-1 to 6-5 and 6-16 to 6-20 from Lot 350-3102; Samples 6-6 to 6-10 and 6-21 to 6-25 from Lot 403-3104; Samples 6-11 to 6-15 and 6-26 to 6-30 from Lot 403-3105; Carestream Health, Inc., Rochester, NY), which had a resistivity of 100 Ohms, were annealed at 150°C for 30 minutes. Initial measurements of electrical conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded. Rectangular peelable masks were disposed onto the samples by screen printing a substance known as HOYO MI-B077 and cured for 20 minutes at 130°C. The masked samples were dipped into a selected etchant composition (varying wt% of H₃PO₄, HNO₃, DOWFAX™ 3B2 surfactant) at selected temperatures (30°C or 35°C) for selected times (30, 60, 90, 120, or 150 seconds). Samples 6-1 to 6-15 were etched at 30°C. Samples 6-1 to 6-15 were etched at 35°C. To remove excess etchant, the samples were washed with distilled water and dried with compressed air using a blower. The masks were removed by being peeled from the samples. The samples were washed with distilled water and air dried. Final measurements of conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded for the etched and masked areas. Table 6 shows the percent change in electrical conductivity, change in percent haze, and change in b* of the etched regions after the mask were removed and of the masked regions after etching.

Table 6

Sample	Etchant Composition (wt%) HPO4/HNO3/3B2	Etching Time (s)	% EC Change (etched)	% EC Change (masked)	ΔH (etched)	ΔH (masked)	Δb^* (etched)	Δb^* (masked)
6-1	45/13.75/0.01	30	1.80	-1.73	0	-0.25	-0.02	-0.44
6-2	45/13.75/0.01	60	29.21	-0.09	-0.01	-0.16	0.08	-0.48
6-3	45/13.75/0.01	90	49.21	-1.02	0	-0.21	0.14	-0.25
6-4	45/13.75/0.01	120	81.29	-0.08	-0.03	-0.3	0.21	-0.54
6-5	45/13.75/0.01	150	96.89	-1.65	0.03	-0.38	0.61	0.05
6-6	45/13.75/0.01	30	1.53	-4.50	0.04	0.02	-0.51	0.03
6-7	45/13.75/0.01	60	41.63	-4.32	0.01	0.08	-0.68	-0.24
6-8	45/13.75/0.01	90	91.20	-0.26	-0.04	-0.02	-0.8	-0.22
6-9	45/13.75/0.01	120	100.00	-2.09	-0.09	0.28	-0.53	-0.44
6-10	45/13.75/0.01	150	100.00	-1.21	-0.08	0.12	-0.63	-0.9
6-11	45/13.75/0.01	30	24.93	-2.74	0.02	0.29	-0.62	-0.6
6-12	45/13.75/0.01	60	100.00	-0.82	-0.13	0.3	-0.63	-0.77
6-13	45/13.75/0.01	90	100.00	-8.02	-0.17	0.34	-0.32	-0.34
6-14	45/13.75/0.01	120	100.00	-4.22	-0.23	0.39	-0.24	-0.29
6-15	45/13.75/0.01	150	100.00	0.62	-0.44	0.46	-0.03	0.17

Table 6 (cont'd)

Sample	Etchant Composition (wt%)	Etching Time (s)	% EC Change (etched)	% EC Change (masked)	ΔH (etched)	ΔH (masked)	Δb^* (etched)	Δb^* (masked)
6-16	45/13.75/0.01	30	13.00	-2.31	0.03	-0.41	-0.23	0.33
6-17	45/13.75/0.01	60	55.30	-0.85	0	-0.26	-0.25	0.17
6-18	45/13.75/0.01	90	100.00	-0.56	-0.07	-0.18	-0.07	0.37
6-19	45/13.75/0.01	120	100.00	-0.42	-0.28	0.03	0.2	-0.04
6-20	45/13.75/0.01	150	100.00	0.33	-0.36	0.22	-0.09	-0.59
6-21	45/13.75/0.01	30	11.71	-4.41	0.09	0.27	0.6	-0.42
6-22	45/13.75/0.01	60	84.14	0.72	0.06	0.17	0.6	-0.29
6-23	45/13.75/0.01	90	100.00	4.82	-0.12	0.28	0.77	-0.45
6-24	45/13.75/0.01	120	100.00	-0.17	-0.15	0.33	-0.27	-0.27
6-25	45/13.75/0.01	150	100.00	-0.79	-0.53	0.31	-0.01	-0.27
6-26	45/13.75/0.01	30	76.57	-3.00	-0.03	0.13	-0.13	-0.25
6-27	45/13.75/0.01	60	100.00	-1.15	0.01	0.2	-0.35	-0.25
6-28	45/13.75/0.01	90	100.00	3.94	-0.12	0.12	-0.26	-0.28
6-29	45/13.75/0.01	120	100.00	-0.28	-0.52	0.15	0.11	0.14
6-30	45/13.75/0.01	150	100.00	-0.56	-0.76	0.6	-0.13	0.28

Example 7

Samples of FLEXX™ 100 transparent conductive film (Samples 7-1 to 7-5 and 7-16 to 7-20 from Lot 350-3102; Samples 7-6 to 7-10 and 7-21 to 7-25 from Lot 403-3104; Samples 7-11 to 7-15 and 7-26 to 7-30 from Lot 403-3105; 5 Carestream Health, Inc., Rochester, NY), which had a resistivity of 100 Ohms, were annealed at 150°C for 30 minutes. Initial measurements of electrical conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded. Rectangular peelable masks were disposed onto the samples by screen printing a substance known as HOYO MI-B077 and cured for 10 20 minutes at 130°C. The masked samples were dipped into a selected etchant composition (varying wt% of H₃PO₄, HNO₃, DOWFAX™ 3B2 surfactant) at 35°C for selected times (30, 60, 90, 120, or 150 seconds). To remove excess etchant, the samples were washed with distilled water and dried with compressed air using a blower. The masks were removed by being peeled from the samples. 15 The samples were washed with distilled water and air dried. Final measurements of conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded for the etched and masked areas. Table 7 shows the percent change in electrical conductivity, change in percent haze, and change in b* of the etched regions after the mask was removed and of the masked regions 20 after etching.

Table 7

Sample	Etchant Composition (wt%) HPO ₄ /HNO ₃ /3B2	Etching Time (s)	% EC Change (etched)	% EC Change (masked)	ΔH (etched)	ΔH (masked)	Δb^* (etched)	Δb^* (masked)
7-1	45/12.5/0.01	30	4.29	1.08	0	-0.32	0.16	0.18
7-2	45/12.5/0.01	60	11.11	-0.78	0.02	-0.21	-0.05	-0.08
7-3	45/12.5/0.01	90	31.71	1.26	-0.05	-0.38	0.08	-0.18
7-4	45/12.5/0.01	120	62.97	0.76	-0.06	-0.41	-0.28	-0.43
7-5	45/12.5/0.01	150	100.00	-0.75	-0.05	-0.17	0.16	-0.42
7-6	45/12.5/0.01	30	2.32	-6.16	0	-0.04	0.07	0.03
7-7	45/12.5/0.01	60	11.89	-1.93	-0.01	0.03	0.02	-0.04
7-8	45/12.5/0.01	90	40.35	0.81	0	0.01	0	0.02
7-9	45/12.5/0.01	120	71.89	-1.46	0.08	0	0.15	0.07
7-10	45/12.5/0.01	150	100.00	-4.55	-0.26	0.07	0.26	0.02
7-11	45/12.5/0.01	30	29.11	-8.17	0.01	0	-0.08	-0.13
7-12	45/12.5/0.01	60	84.16	-5.77	0.08	0.12	0.13	-0.01
7-13	45/12.5/0.01	90	100.00	-5.48	-0.14	0.15	0.2	-0.18
7-14	45/12.5/0.01	120	100.00	-4.42	-0.3	0.4	-0.06	-0.26
7-15	45/12.5/0.01	150	100.00	-2.09	-0.34	0.45	-0.39	-0.69

Table 7 (cont'd)

Sample	Etchant Composition (wt%) HPO ₄ /HNO ₃ /3B2	Etching Time (s)	% EC Change (etched)	% EC Change (masked)	ΔH (etched)	ΔH (masked)	Δb^* (etched)	Δb^* (masked)
7-16	42.5/13.75/0.01	30	2.25	-3.80	0.04	-0.25	-0.21	-0.31
7-17	42.5/13.75/0.01	60	11.69	0.09	-0.03	-0.29	-0.06	0.02
7-18	42.5/13.75/0.01	90	53.51	-1.41	-0.03	-0.29	-0.1	-0.28
7-19	42.5/13.75/0.01	120	94.13	0.75	-0.07	-0.3	0.08	-0.24
7-20	42.5/13.75/0.01	150	100.00	0.00	-0.08	-0.38	0.29	-0.61
7-21	42.5/13.75/0.01	30	4.79	-5.57	0.08	-0.08	0	-0.01
7-22	42.5/13.75/0.01	60	35.29	-2.11	0.02	0	0.05	0.08
7-23	42.5/13.75/0.01	90	73.19	-0.70	-0.03	-0.1	0.14	0.02
7-24	42.5/13.75/0.01	120	80.80	0.00	-0.03	-0.09	0.37	-0.04
7-25	42.5/13.75/0.01	150	100.00	0.61	-0.17	0.11	0.04	-0.42
7-26	42.5/13.75/0.01	30	21.09	-1.95	0.04	0.13	-0.07	0.01
7-27	42.5/13.75/0.01	60	79.75	-4.79	-0.03	0.09	0.12	-0.18
7-28	42.5/13.75/0.01	90	90.98	-1.96	-0.06	0.1	0.08	-0.07
7-29	42.5/13.75/0.01	120	100.00	-3.78	-0.01	0.17	0.11	-0.33
7-30	42.5/13.75/0.01	150	100.00	-6.49	-0.36	0.12	0.15	0.03

Example 8

Samples of FLEXX™ 100 transparent conductive film (Samples 8-1 to 8-6 from Lot 416-3104; Samples 8-7 to 8-12 from Lot 416-3117; Samples 8-13 to 8-17 from Lot 416-3128; Carestream Health, Inc., Rochester, NY), which
5 had a resistivity of 100 Ohms, were annealed at 150°C for 30 minutes. Initial measurements of electrical conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded. Rectangular peelable masks were disposed onto the samples by screen printing a substance known as HOYO MI-B077 and cured for 20 minutes at 130°C. The masked samples were dipped
10 into a selected etchant composition (varying wt% of H₃PO₄, HNO₃, DOWFAX™ 3B2 surfactant) at 30°C for selected times (120, 240, 270, 300, 330, 360, 390, 420, 450, 480, 510, 540 seconds, etc.). To remove excess etchant, the samples were washed with distilled water and dried with compressed air using a blower. The masks were removed by being peeled from the samples. The samples were
15 washed with distilled water and air dried. Final measurements of conductivity, percent transmittance, percent haze, and CIE L*, a*, and b* color metrics were recorded for the etched and masked areas. Table 8 shows the percent change in electrical conductivity, change in haze, and change in b* of the etched regions after the mask were removed and of the masked regions after etching.

20

The invention has been described in detail with reference to specific embodiments, but it will be understood that variations and modifications can be effected within the spirit and scope of the invention. The presently disclosed embodiments are therefore considered in all respects to be illustrative
25 and not restrictive. The scope of the invention is indicated by the attached claims, and all changes that come within the meaning and range of equivalents thereof are intended to be embraced therein.

Table 8

Sample	Etchant Composition (wt%) HPO ₄ /HNO ₃ /3B2	Etching Time (s)	% EC Change (etched)	% EC Change (masked)	ΔH (etched)	ΔH (masked)	Δb^* (etched)	Δb^* (masked)
8-1	42.5/15/0.01	120	10.74	-3.05	-0.06	-0.07	-0.18	-0.14
8-2	42.5/15/0.01	300	26.37	-5.76	0.02	0.12	-0.04	-0.26
8-3	42.5/15/0.01	420	78.03	-1.21	-0.1	0.09	-0.03	-0.14
8-4	42.5/15/0.01	480	86.90	-2.53	-0.03	0.2	0	-0.51
8-5	42.5/15/0.01	540	100.00	-0.24	-0.28	0.18	-0.19	-0.17
8-6	42.5/15/0.01	510	100.00	-1.76	-0.06	0.26	0.06	-0.21
8-7	42.5/15/0.01	120	10.98	-6.56	0.04	-0.1	-0.23	-0.35
8-8	42.5/15/0.01	300	61.59	-4.32	-0.09	-0.02	-0.06	-0.08
8-9	42.5/15/0.01	420	100.00	-6.31	0.23	0.06	-0.07	-0.57
8-10	42.5/15/0.01	390	77.57	-1.65	-0.03	0.04	0.13	-0.12
8-11	42.5/15/0.01	450	100.00	-6.11	-0.12	0.02	0.01	-0.5
8-12	42.5/15/0.01	480	100.00	-3.02	-0.24	0	-0.25	-0.09
8-13	42.5/15/0.01	240	31.21	-1.88	0.05	0.17	-0.11	-0.45
8-14	42.5/15/0.01	300	100.00	-0.57	-0.12	0.08	-0.01	-0.28
8-15	42.5/15/0.01	270	55.86	0.38	-0.04	0.11	0.02	-0.33
8-16	42.5/15/0.01	330	100.00	-1.61	-0.22	0.23	-0.13	-0.42
8-17	42.5/15/0.01	360	98.66	0.00	0.14	0.22	0.25	-0.35

WHAT IS CLAIMED:

1. A method comprising:
etching a film comprising electrically conductive structures according to a
5 pattern using an aqueous etching solution to provide an etched region having a
first conductivity and an unetched region having a second conductivity, the second
conductivity being greater than the first conductivity,
wherein the aqueous etching solution comprises 25 to 65% by weight of
phosphoric acid and 1 to 18% by weight of nitric acid.
10
2. The method according to claim 1, wherein the aqueous solution comprises at
least one surfactant.
3. The method according to claim 2, wherein the at least one surfactant comprises
15 at least one anionic surfactant.
4. The method according to claim 2, wherein the at least one surfactant comprises
decyl(sulfophenoxy)benzenesulfonic acid, disodium salt and
oxybis(decylbenzenesulfonic acid), disodium salt.
20
5. The method according to claim 2, wherein the aqueous etching solution
comprises 40 to 60% by weight of phosphoric acid, 10 to 15% by weight of nitric
acid, and 0.005-0.05% by weight of the at least one surfactant.
- 25 6. The method according to claim 1,
wherein prior to etching, the film exhibited a preexisting set of optical
properties, and wherein after etching, the etched region exhibited a first
consequent set of optical properties, and the unetched region exhibited a second
consequent set of optical properties, and
30 further wherein the preexisting set of optical properties and the first
consequent set of optical properties are substantially identical.

7. The method according to claim 6, wherein the preexisting set of optical properties comprises a preexisting total light transmission and the first consequent set of optical properties comprises a first consequent total light transmission that is substantially identical to the preexisting total light transmission.
- 5
8. The method according to claim 6, wherein the preexisting set of optical properties comprises a preexisting haze and the first consequent set of optical properties comprises a first consequent haze that is substantially identical to the preexisting haze.
- 10
9. The method according to claim 6, wherein the preexisting set of optical properties comprises a preexisting L^* value and the first consequent set of optical properties comprises a first consequent L^* value that is substantially identical to the preexisting L^* value.
- 15
10. The method according to claim 6, wherein the preexisting set of optical properties comprises a preexisting a^* value and the first consequent set of optical properties comprises a first consequent a^* value that is substantially identical to the preexisting a^* value.
- 20
11. The method according to claim 6, wherein the preexisting set of optical properties comprises a preexisting b^* value and the first consequent set of optical properties comprises a first consequent b^* value that is substantially identical to the preexisting b^* value.
- 25
12. The method according to claim 6, wherein the first consequent set of optical properties and the second consequent set of optical properties are substantially identical.
- 30
13. The method according to claim 6, wherein after etching, the pattern is invisible to the unaided eye.

14. The method according to claim 6, wherein the electrically conductive structures comprise silver nanowires.

15. A method comprising:

- 5 etching a film comprising electrically conductive structures according to a pattern using an aqueous etching solution to provide an etched region having a first conductivity and an unetched region having a second conductivity, the second conductivity being greater than the first conductivity,
- wherein the aqueous etching solution comprises 65 to 75% by weight of
- 10 nitric acid.

INTERNATIONAL SEARCH REPORT

International application No PCT/US2014/051617

A. CLASSIFICATION OF SUBJECT MATTER INV. C09K13/06 C23F1/30 G06F3/044 H05K3/06 H01L33/42 H01L51/00 H01B1/22 ADD. According to International Patent Classification (IPC) or to both national classification and IPC				
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) C09K G06F H05K C08K H01L H01B Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) EPO-Internal				
C. DOCUMENTS CONSIDERED TO BE RELEVANT				
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.		
X	US 2006/278606 A1 (PARK HONG-SICK [KR] ET AL) 14 December 2006 (2006-12-14) claims 14,18 paragraph [0049] -----	1-14		
X	US 2003/207513 A1 (SAITOU NORIYUKI [JP] ET AL) 6 November 2003 (2003-11-06) claims 1, 9, 10 paragraph [0001] -----	1-14		
X	US 2004/242017 A1 (PARK HONG-SICK [KR] ET AL) 2 December 2004 (2004-12-02) claims 1,2,18-20 -----	1,6-14		
A	----- -/--	2-5		
<input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input checked="" type="checkbox"/> See patent family annex.				
* Special categories of cited documents : <table style="width: 100%; border: none;"> <tr> <td style="width: 50%; border: none; vertical-align: top;"> "A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed </td> <td style="width: 50%; border: none; vertical-align: top;"> "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family </td> </tr> </table>			"A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family
"A" document defining the general state of the art which is not considered to be of particular relevance "E" earlier application or patent but published on or after the international filing date "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified) "O" document referring to an oral disclosure, use, exhibition or other means "P" document published prior to the international filing date but later than the priority date claimed	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art "&" document member of the same patent family			
Date of the actual completion of the international search	Date of mailing of the international search report			
4 February 2015	10/02/2015			
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Authorized officer Domínguez Gutiérrez			

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2014/051617

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. Claims Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.

3. As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-14

A method comprising etching a film comprising electrically conductive structures according to a pattern using an aqueous etching solution to provide an etched region having a first conductivity and an unetched region having a second conductivity, the second conductivity being greater than the first conductivity, the aqueous etching solution comprises 25-65 wt% of phosphoric acid and 1-18 wt% of nitric acid.

2. claim: 15

A method comprising etching a film comprising electrically conductive structures according to a pattern using an aqueous etching solution to provide an etched region having a first conductivity and an unetched region having a second conductivity, the second conductivity being greater than the first conductivity, the aqueous etching solution comprising 65-75 wt% of nitric acid.

INTERNATIONAL SEARCH REPORT

International application No
PCT/US2014/051617

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2012/031872 A1 (SEKIGUCHI HIROKI [JP] ET AL) 9 February 2012 (2012-02-09) claims 1,6,10 paragraph [0014] examples 7,9	15
X	----- RIVKIN T ET AL: "Direct write processing for photovoltaic cells", CONFERENCE RECORD OF THE IEEE PHOTOVOLTAIC SPECIALISTS CONFERENCE 2002 INSTITUTE OF ELECTRICAL AND ELECTRONICS ENGINEERS INC. US; [IEEE PHOTOVOLTAIC SPECIALISTS CONFERENCE], CONFERENCE RECORD OF THE 29TH IEEE PHOTOVOLTAIC SPECIALISTS CONFERENCE - 200, vol. CONF. 29, 19 May 2002 (2002-05-19), pages 1326-1329, XP010666528, DOI: 10.1109/PVSC.2002.1190854 ISBN: 978-0-7803-7471-3 Spray-printed contacts to Si; page 1, column 2	15
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INTERNATIONAL SEARCH REPORT

Information on patent family members

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