

Effect of Photosensitivity in Acrylic Photoreactive Electrode Pastes on Line Width Uniformity for Large-Sized Plasma Display Panels

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ABSTRACT: Ag electrodes with line width uniformity for large-sized plasma display panels were successfully fabricated through a photolithographic process using photosensitive Ag pastes with optimized photosensitive properties. The photosensitivity of the Ag electrode pastes in the photolithographic process was investigated as a function of the types and contents of photoinitiators, the molecular weights and acid values of acrylic binders with carboxylic acid groups, and the process variables, such as the UV-light intensity and dose, with a step tablet. This study revealed that the photoinitiator was a major parameter for the photosensi-

tivity of the Ag electrode pastes. With the photosensitivity of the photosensitive Ag electrode pastes optimized by the study of the photoinitiator contents, Ag electrodes with line width uniformity were achieved with an HSP-188 photoinitiator content of 15 wt % on the basis of the reactive monomers, regardless of the variation of the light dose from 250 to 350 mJ/cm² and intensity from 15 to 25 mW/cm². © 2007 Wiley Periodicals, Inc. *J Appl Polym Sci* 107: 658–666, 2008

Key words: lithography; photopolymerization; processing; radiation

INTRODUCTION

The dramatic growth of the plasma display panel (PDP) industry has been achieved by the realization of large and thin panels and high resolution, which is needed in digital broadcasting.^{1,2} Recently, engineers and researchers engaged in the development of PDPs at universities and laboratories have been pursuing the exciting objective of large, high-resolution panels, adopting single-step methods that can produce several panels on large glass plates. As a result, there shall be mass production at low cost. As the size of PDPs increases over 60 in. and high-definition-television-grade PDPs are needed, Ag electrodes with high resolution, thin layers, and no edge curls are required. High resolution, line sharpness, and line width uniformity for address electrodes and bus electrodes of PDPs, therefore, have been key issues for materials. The function of electrodes in

PDPs is to transfer signals from circuits to panels, causing plasma discharge to display information. In addition, electrodes should define pixels in PDPs, which have the same function as a black matrix. For this purpose, the development of photosensitive materials with line width uniformity and high resolution is required.^{3–5}

Ag electrodes in PDPs have been fabricated by the photolithographic process, in which a metal halide lamp has been generally used as a UV-light source with a proximity (off-contact) type under an air atmosphere in a radiation process. The crosslinking reaction of photosensitive Ag pastes for electrodes in PDPs is as follows. UV radiation of the photosensitive Ag paste activates photoinitiators, forming free radicals. These free radicals successively react with double bonds of reactive monomers, resulting in negative types of patterns that are not developable in an alkali aqueous solution. Generally, the rate and conversion of crosslinking by UV radiation are governed by instrument variables such as the UV-light intensity, dose, and wavelength as well as material characteristics such as reactive monomers, oligomers, photoinitiators, and polymeric binders with carboxylic acid.^{6–10} However, the photocrosslinking reaction in photosensitive Ag electrode pastes is

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hampered by ineffective penetration of UV light through the pastes due to a thick layer over 10 μm thick and scattering of UV light by inorganic materials such as Ag powders and glass frit. In addition, the photosensitivity of photosensitive Ag electrode pastes is reduced with the declining efficiency of photoinitiators by oxygen under UV radiation as well as the deviation of the luminous intensity over large-sized displays, and this results in decreased sharpness of patterns and nonuniformity of line width. These drawbacks, described previously, have produced systematic image quality errors (so-called mura) caused by differences in cell sizes by nonuniformity of line width. Therefore, mura over large-sized PDPs are prevented by the minimization of difference of cell sizes by nonuniformity of line width.^{11,12}

In this study, the effects of variables such as photoinitiators and UV-light intensities and doses on the photopolymerization of photosensitive Ag electrode pastes was investigated with a step tablet.^{13–15} As a result, Ag electrodes with uniform line width in PDPs were obtained with optimized formulations. These results could be used for preventing systematic image quality errors in large-sized PDPs.

EXPERIMENTAL

Materials

Polymeric binders were prepared with comonomers such as isobutyl methacrylate (iBMA), 2-hydroxy ethyl methacrylate (HEMA), and methacrylic acid (MAA), which have excellent adhesion strength to glass substrates, high thermal stability, and alkali-developable properties. The monomers were purified to remove inhibitors and moisture and then distilled under vacuum before use. α,α' -Azobisisobutyronitrile (AIBN; Junsei Co., Kyoto, Japan), used as a free-radical initiator, was reagent-grade and was recrystallized from methanol before use. Methanol (TCI Co., Tokyo, Japan) for the polymerization was distilled under vacuum, and mercaptoacetic acid (Aldrich Co., St. Louis, MO) was used as a chain-transfer agent (CTA) for controlling the molecular weights of the polymeric binders. UV-curable monomers and oligomers such as pentaerythritol triacrylate (PETA), trimethylol propanetriacrylate (TMPTA), and trimethylolpropane ethoxytriacrylate (TMPEOTA) were obtained from SK-CYTEC Co. (Ulsan, Korea) and used as received. *n*-Octadecyltriethoxy silane (TCI Co.) was used as a dispersant for photosensitive Ag pastes. Photoinitiators such as 2,4,6-trimethylbenzoyl-diphenyl phosphine (Darocur TPO, Ciba Chemical Co., Basel, Switzerland), phenyl bis(2,4,6-trimethyl benzoyl) phosphine oxide (Irgacure 819, Ciba Chemical Co.), and benzophenone

derivatives (HSP-188, SK-CYTEC Co.) were used. Silver powder made by a wet process was purchased from Sigma–Aldrich Co.

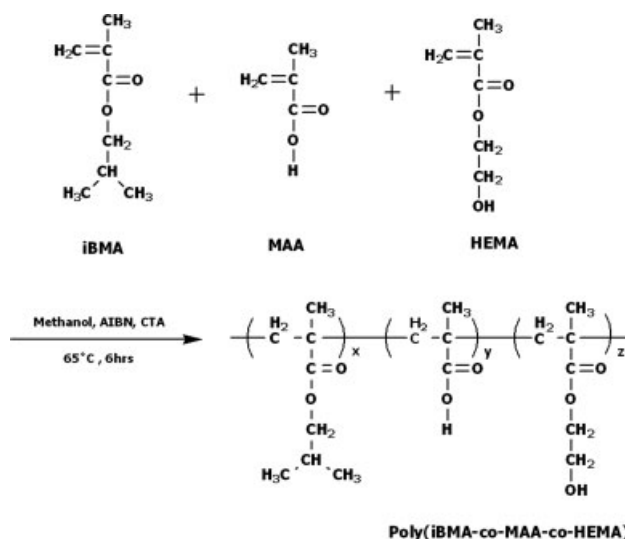
Synthesis of the polymeric binders

Comonomers such as iBMA, MAA, and HEMA were charged into a resin kettle equipped with a condenser and a stirrer. Series of polymerizations were carried out by the addition of AIBN and mercaptoacetic acid as a CTA after 65°C was reached under an N_2 atmosphere and then were continued for 6 h, resulting in polymeric binders with different molecular weights and acid values. The products were used for fabricating pastes without any further purification steps. The polymerization route to the polymeric binders is shown in Scheme 1.

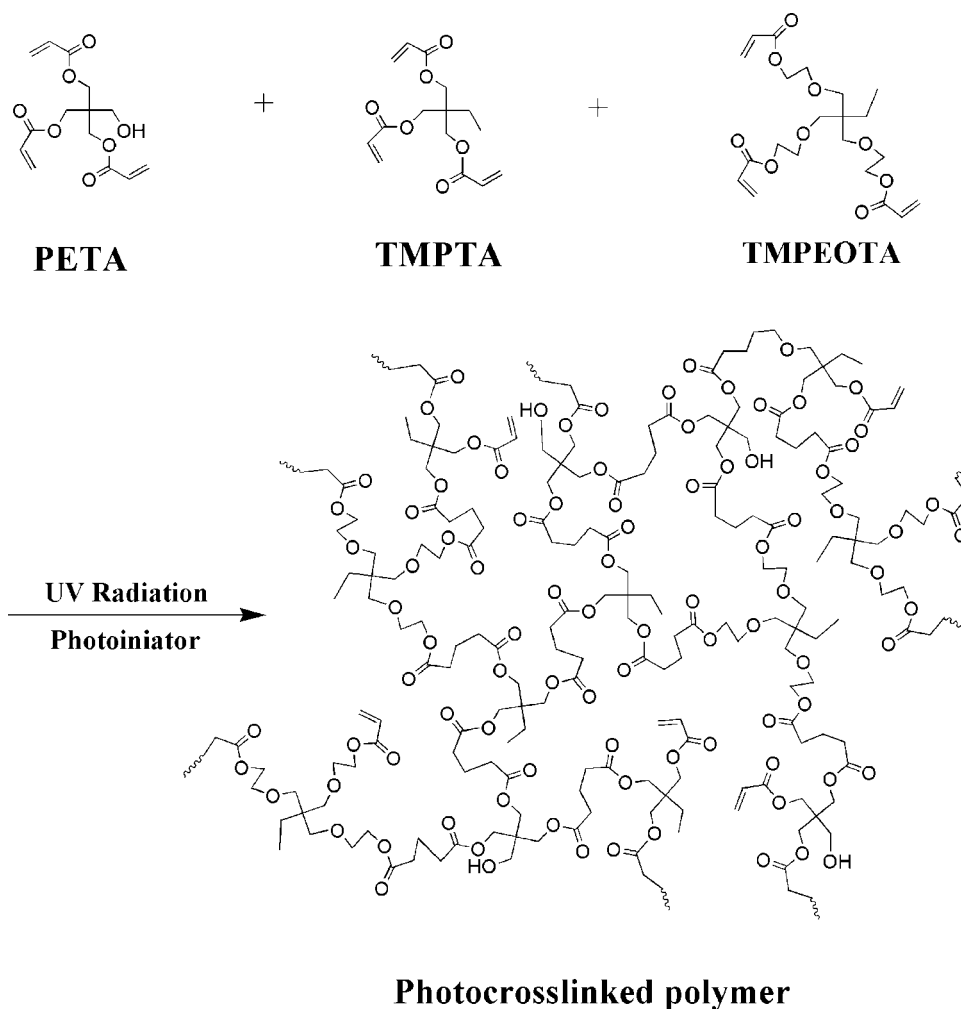
Photosensitive Ag paste formulation and photolithographic process

Photosensitive Ag pastes were prepared initially by the mixing of a Ag/glass frit powder mixture and photosensitive vehicle composed of poly(iBMA-co-MAA-co-HEMA) as a binder, methanol as a solvent, *n*-octadecyltriethoxy silane as a dispersant, a photoinitiator, and UV-curable monomers such as PETA, TMPTA, and TMPEOTA with a mechanical stirrer followed by a three-roll mill (Exakt 50, Hamburg, Germany) for 2 h. Scheme 2 presents the photocrosslinking mechanism of the UV-curable monomers.

Photosensitive Ag paste was screen-printed onto a glass substrate and dried at 110°C in an oven for 15 min. Then, a photomask with an 80- μm line width and a 300- μm distance between adjacent repeating



Scheme 1 Synthesis of polymeric binders prepared by free-radical terpolymerization of iBMA, MMA, and HEMA.



Scheme 2 Photocrosslinking mechanism of UV-curable monomers.

lines was placed on top of the dried Ag paste layer and irradiated with a UV lamp to a total dosage of 100–500 mJ/cm². After UV exposure, the Ag layer was developed with a 0.3 wt % Na₂CO₃ alkaline aqueous solution at 30°C and 1.0 kg/cm² of pressure

for 20 s and then dried at 100°C in an oven for 10 min. After drying, the patterned Ag electrode was examined with an optical microscope (STM6, Olympus, Tokyo, Japan) to check the line width of the Ag electrode patterns.

TABLE I
Characteristics of Polymeric Binders Synthesized with Different Feed Ratios of the Monomers and Initiator/CTA

Group		iBMA : HEMA : MAA feed ratio (wt %)	Initiator	CTA	M_w (g/mol)	M_w/M_n	Acid value (mg of KOH/g of polymer)
A	EBP-1	70 : 10 : 20	0.3	0.2	17,290	1.92	79.08
	EBP-2	68 : 10 : 22	0.3	0.2	16,330	1.65	84.52
	EBP-3	66 : 10 : 24	0.3	0.2	14,540	1.58	90.49
	EBP-4	64 : 10 : 26	0.3	0.2	13,550	1.60	97.68
	EBP-5	62 : 10 : 28	0.3	0.2	12,680	1.62	106.55
B	EBP-6	70 : 10 : 20	0.2	0.2	26,830	1.25	79.44
	EBP-7	68 : 10 : 22	0.2	0.2	25,100	1.62	83.77
	EBP-8	66 : 10 : 24	0.2	0.2	24,600	1.43	88.98
	EBP-9	64 : 10 : 26	0.2	0.2	23,320	1.61	94.98
	EBP-10	62 : 10 : 28	0.2	0.2	22,820	1.37	104.92

M_w = weight-average molecular weight; M_n = number-average molecular weight.

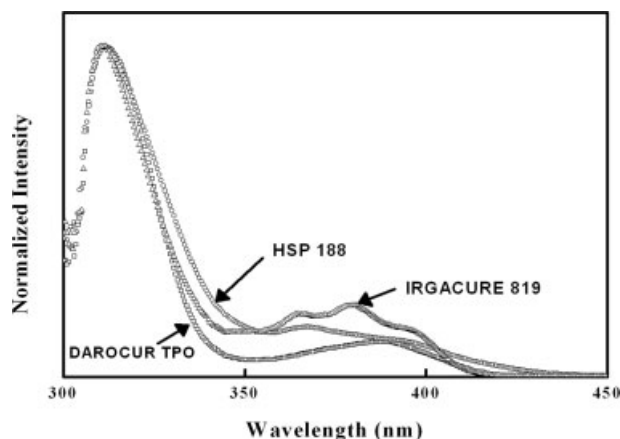


Figure 1 UV absorption spectra of the photoinitiators.

Instrumental analysis

The ultraviolet–visible (UV–vis) absorption spectra were obtained with a Shimadzu UV-2100 (Kyoto, Japan). The molecular weights and molecular weight distributions of the synthesized polymeric binders were measured with a Waters gel permeation chromatograph with tetrahydrofuran (THF) as an eluent. The acid value of the polymeric binders was measured through the titration of a binder solution in a mixed solvent (toluene/ethanol = 1/1 v/v) with a 0.1N KOH solution in the presence of 1.0% phenolphthalein indicator (ASTM D 4662). Photosensitive steps were measured with a step tablet (Eastman Kodak Co., Rochester, NY), which consisted of a series of 21 steps that varied in density from totally clear (step 1) to totally opaque (step 21) through the photolithographic process. The UV-light intensity and dose were measured with a radiometer (VLX-3W, Vilbert Lourmat, Paris, France).

RESULTS AND DISCUSSION

Properties of the polymeric binders

The general functions of polymeric binders are (1) to optimize screen-printing processability of pastes with good dispersion of inorganic materials such as Ag powder and glass frit and (2) to give the pastes optimized pattern formation that can be formed by the development of the UV-unexposed area with an alkali solution.¹⁶ Therefore, polymeric binders were prepared to investigate the effects of the molecular

weights and acid values on the photosensitivity of the paste. The polymerization of the polymeric binders was performed through changes in the feed ratio of the monomers and contents of an initiator/CTA, resulting in polymeric binders of group A (in a molecular weight range of 12,689–17,290 g/mol) and group B (in a molecular weight range of 24,320–26,830 g/mol) with an acid value range of 79.08–106.55 mg of KOH/g of polymeric binder, which depended on the content of MAA, as presented in Table I.

Effect of the photoinitiators on the photosensitivity

The effect of the photoinitiators on the photosensitivity of the paste was investigated with a halogen lamp as a UV source with wavelengths of 312 and 365 nm. To select a suitable photoinitiator, UV–vis absorption spectra of three different types of photoinitiators were measured in THF solutions (concentration = 5.0×10^{-5} g/mL), as shown in Figure 1. All photoinitiators tested here showed characteristic absorption bands in a range of 350–450 nm due to the $n-\pi^*$ transition of the P=O group, in addition to the carbonyl absorption around 310 nm.^{17,18}

As presented in Table II, the photosensitive Ag pastes were first formulated with 66.5 wt % Ag powder, 3.5 wt % glass frit, 7.6 wt % polymeric binder, 7.2 wt % reactive monomer, 14.1 wt % solvent, and 1.1 wt % dispersant and then prepared sequentially with a planetary mixer and a three-roll mill.¹⁹ Three different types of photoinitiators (Irgacure 819, HSP-188, and Darocur TPO), ranging from 0 to 20 wt % on the basis of the reactive monomers, were formulated with the photosensitive Ag pastes, and then the results of the step level of the paste formulations were measured with the step tablet at a constant light intensity (20 mW/cm^2) and dose (300 mJ/cm^2), as shown in Figure 2. Regardless of the types of photoinitiators used, the step level of all paste formulations apparently reached a maximum level when 15 wt % photoinitiator on the basis of the reactive monomers was used. Under the same UV-light condition used for studying the step level with the step tablet, Ag electrode patterns were also fabricated with 15 wt % photoinitiator on the basis of the reactive monomers, and they are shown in Figure 3. The relationship between the step level and pattern formation was studied by the comparison of Figures 2

TABLE II
Typical Formulation of Photosensitive Ag Pastes

Component	Photosensitive vehicle				Inorganic powder	
	Binder polymer	Solvent	Reactive monomer	Dispersant	Ag powder	Glass frit
Composition (wt %)	7.6	14.1	7.2	1.1	66.5	3.5

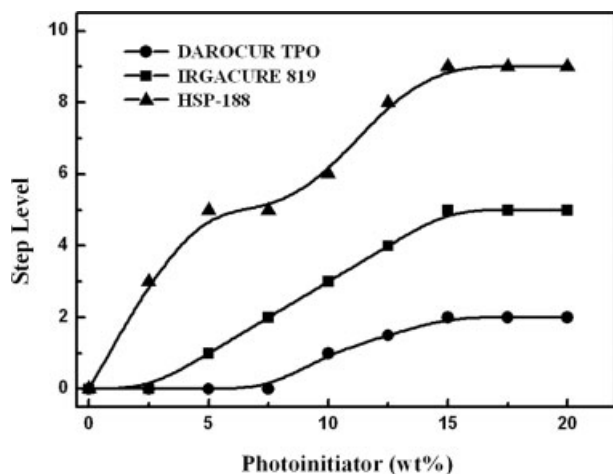


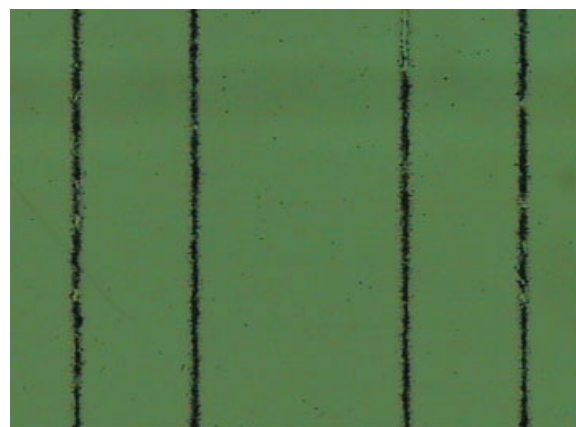
Figure 2 Effect of the contents of the photoinitiators on the photosensitivity of the pastes.

and 3. The step level obtained with Darocur TPO as the photoinitiator was saturated with the step level of 2, and line patterns formed by the use of a photo-mask with an 80- μm line width were peeled off under the developing process, as presented in Figure 3(a). Line patterns formed by the use of Irgacure 819, which saturated the step level of 5, exhibited partial peel-off, as shown in Figure 3(b). It can be observed in Figure 3(c) that the formulation of Ag paste containing HSP-188 showed patterns with good adhesion and sharpness, resulting in the step level of 9, which was the highest degree of photopolymerization among the three formulations with different initiators. Although the understanding of the mechanism of photopolymerization in these types of thick composite films was hampered by various processing factors, the results of the step tablet in this study revealed that the step level was saturated above a certain content of photoinitiators, and the highest step level of 9 was obtained with the Ag paste formulation with HSP-188 as the photoinitiator.^{19–21} Therefore, optimum photosensitivity in the formulations of the pastes was achieved with 15 wt % photoinitiator on the basis of the reactive monomers in the pastes; in particular, HSP-188 showed the best performance among them.

Effect of the molecular weight and acid value of the polymeric binders

The effects of the molecular weights and acid values of the polymeric binders on the step level of the pastes were investigated. It has been generally accepted that the step level decreases with an increasing acid value of the polymeric binders because of the increased possibility of peel-off by swelling under developing conditions with an alkali aqueous solution. As presented in Figure 4(a,b),

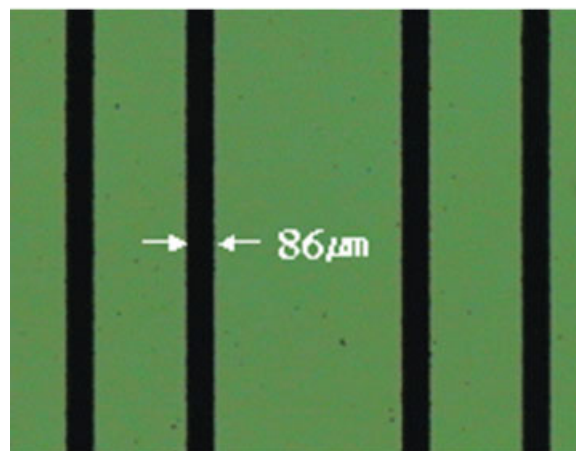
however, the molecular weight and acid value of the polymeric binders were affected little on the step level. This might be due to the fact that because of



(a)



(b)



(c)

Figure 3 Images of Ag electrode patterns obtained after a photolithographic process with different photoinitiators: (a) 15 wt % Darocur TPO, (b) 15 wt % Irgacure 819, and (c) 15 wt % HSP-188. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

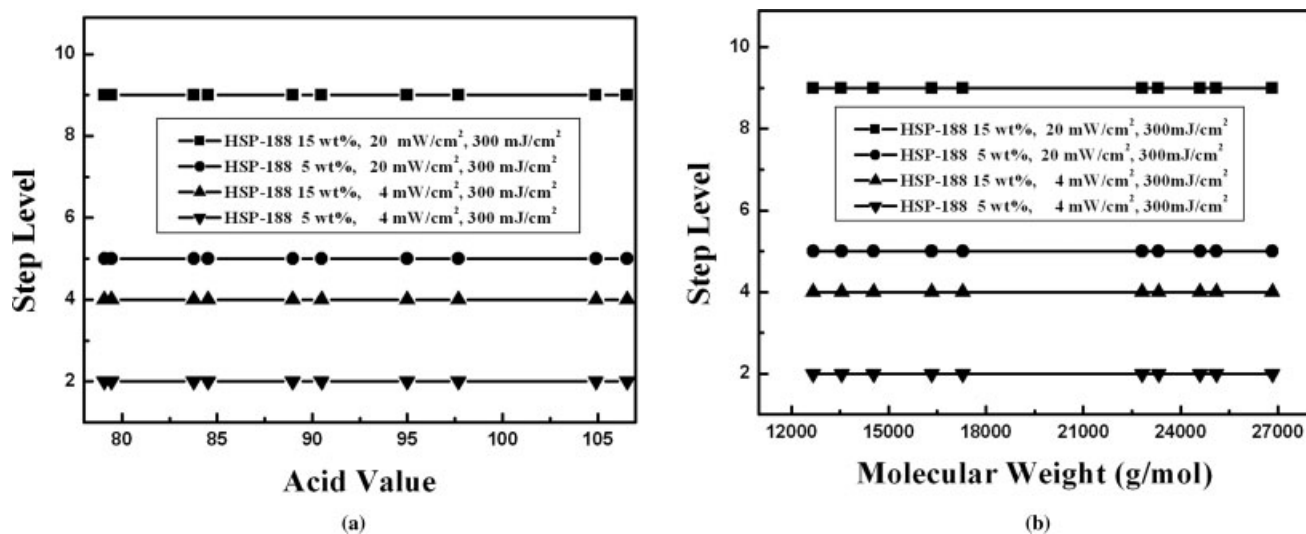


Figure 4 (a) Effect of the acid values of the polymeric binder on the photosensitivity and (b) effect of the molecular weights of the polymeric binder on the photosensitivity.

the higher efficiency of photoinitiation, a certain content of the photoinitiator in the paste gave rise to a crosslinking density high enough to prevent penetration of the alkali developing solution.²² According to the results from the step tablet, however, the step level varied from 2 to 9, depending on the light intensity and photoinitiator content. Therefore, it appears that processing variables such as the photoinitiator content and light intensity were major factors for the adhesion property of the patterns that formed in comparison with the molecular weight and acid value of the polymeric binders in the Ag paste formulation for PDPs.

Effects of the UV-light intensity and dose

The effects of the light intensity and dose on the step level are presented in Figure 5 and were determined with the following conditions: HSP-188 photoinitiator contents of 5 and 15 wt % on the basis of the reactive monomers, light intensities of 4 and 20 mW/cm², and light doses ranging from 20 to 400 mJ/cm². The step level increased with the light dose and then reached the maximum level over a light dose of 300 mJ/cm². This implied that certain levels of the photoinitiator, light intensity, and dose were necessary for a photosensitive material. The step level of 9, which was the highest photosensitivity obtained from formulations in this study, could be achieved especially with the formulation with an HSP-188 photoinitiator content of 15 wt % on the basis of the reactive monomers at a light intensity of 20 mW/cm². In the case of the same HSP-188 photoinitiator, the light intensity was considered to be the more important factor for the photosensitivity than the photoinitiator content. Because the formulation

with an HSP-188 photoinitiator content of 15 wt % on the basis of the reactive monomers at a light intensity of 4 mW/cm² showed the step level of 4, the formulation with an HSP-188 photoinitiator content of 5 wt % on the basis of the reactive monomers at a light intensity of 20 mW/cm² exhibited the step level of 5. On the other hand, patterns prepared with an HSP-188 photoinitiator content of 5 wt % on the basis of the reactive monomers at a light intensity of 4 mW/cm² showed the very low step level of 2, resulting in peeling-off patterns under the developing process.

Effects of the photoinitiator content, light dose, and intensity on the line uniformity

To study the effect of the HSP-188 photoinitiator content, light dose, and intensity on the variation of

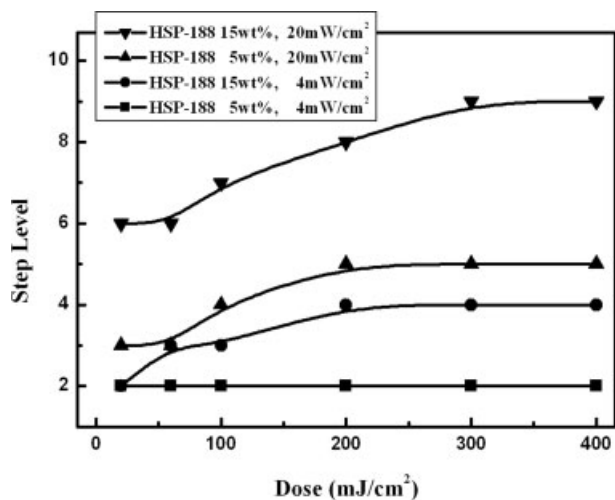


Figure 5 Effects of the light intensity and dose on the photosensitivity.

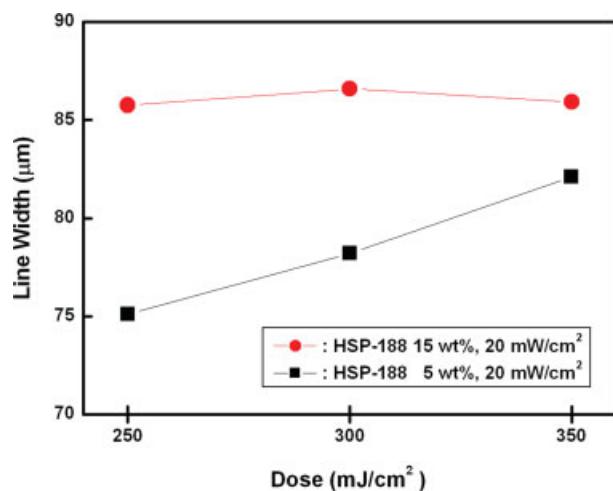


Figure 6 Variation of the line width uniformity as a function of the photoinitiator content and light dose. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

the line width, patterns on a 10 cm × 10 cm plate were fabricated with the photolithographic process. First, the variation of the line width measured under a fixed light intensity of 20 mW/cm² and a changing light dose ranging from 250 to 350 mJ/cm² is presented in Figure 6. For patterns prepared with a photoinitiator content of 5 wt % at different light doses such as 250, 300, and 350 mJ/cm², line widths were measured to be 75.10, 78.20, and 82.12 μm, respectively. With light doses ranging from 250 to 350 mJ/cm², the total deviation of line widths was found to be 3.52. On the other hand, line widths of patterns prepared with a photoinitiator content of 15 wt % at different light doses such as 250, 300, and 350 mJ/cm² were measured to be 85.76, 86.60, and 85.94 μm, respectively. The total deviation of line widths formed by Ag pastes that contained a photoinitiator content of 15 wt % was greatly reduced to 0.44 versus 3.52 with a photoinitiator content of 5 wt %.

Second, the variation of line width measured under a fixed light dose of 300 mJ/cm² and a changing light intensity ranging from 15 to 25 mW/cm² is presented in Figure 7. For patterns prepared with a photoinitiator content of 5 wt % at different light intensities such as 15, 20, and 25 mW/cm², line widths were measured to be 72.80, 78.20, and 84.10 μm, respectively. With a light intensity ranging from 15 to 25 mW/cm², the total deviation of line widths was found to be 5.65. On the other hand, line widths of patterns prepared with a photoinitiator content of 15 wt % at different light doses such as 15, 20, and 25 mW/cm² were measured to be 85.70, 86.60, and 86.40 μm, respectively. The total deviation of line widths formed by Ag pastes that contained a photoinitiator content of 15 wt % was greatly

reduced to 0.62 versus 5.65 in with a photoinitiator content of 5 wt %.

This result revealed that line width uniformity could be achieved with a photoinitiator content of 15 wt % on the basis of the reactive monomers because of the optimized efficiency of photoinitiation in photosensitive Ag pastes with a thick layer, regardless of the variation of the light dose from 250 to 350 mJ/cm² and intensity from 15 to 25 mW/cm².

Fabrication of the Ag electrode by the photolithographic process

Figure 8 presents scanning electron microscopy (SEM) images of patterns formed with photoinitiator contents of 5 and 15 wt % under the same photolithographic conditions: a light dose of 20 mW/cm² and a light intensity of 300 mJ/cm² followed by the temperature increasing to 580°C at a heating rate of 0.8°C/min and sintering at 580°C for 30 min. The resulting pattern exhibited sheet resistance of 5.5 mΩ/□, regardless of the photoinitiator content. As can be seen in Figure 8(a,b), the patterns showed nonuniformity of line width with edge curl because of the low degree of photopolymerization at a photoinitiator content of 5 wt %. Figure 8(c,d) presents patterns with excellent sharpness but no edge curl because higher crosslinking at a photoinitiator content of 15 wt % created stronger adhesion to the glass after the developing process and prevented shrinkage after sintering.²³

CONCLUSIONS

Photosensitive Ag paste was formulated by the optimization of the photosensitivity to maintain line

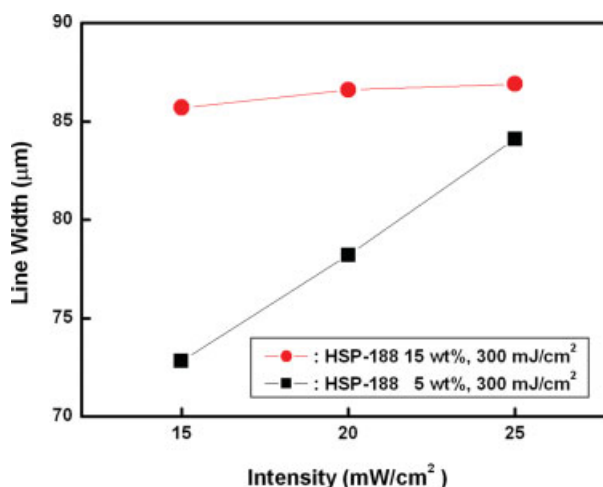


Figure 7 Variation of the line width uniformity as a function of the photoinitiator content and light intensity. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

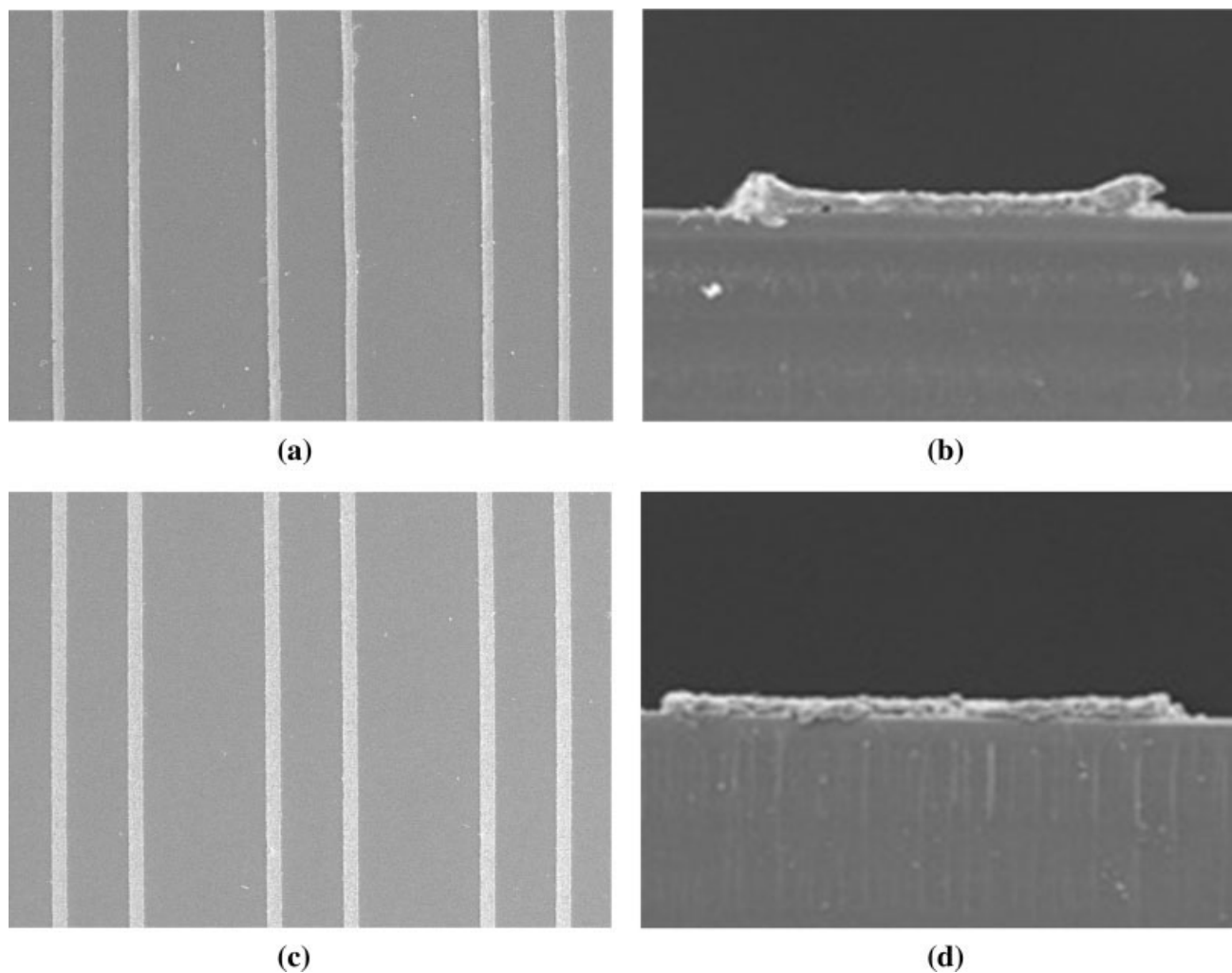


Figure 8 SEM images of Ag electrode patterns formed by the use of the same photolithographic and sintering conditions, except for two different photoinitiator contents: (a) top view with a photoinitiator content of 5 wt % (magnification: 500 \times), (b) cross-sectional view with a photoinitiator content of 5 wt % (magnification: 500,000 \times), (c) top view with a photoinitiator content of 15 wt % (magnification: 500 \times), and (d) cross-sectional view with a photoinitiator content of 15 wt % (magnification: 500,000 \times).

uniformity of Ag electrodes because a decrease in the line uniformity of large-sized PDPs over 60 in. was caused by inhomogeneous light intensity and dose during irradiation by a proximity type. Variables affecting the photosensitivity of the photosensitive Ag paste were investigated in terms of the photoinitiator, binder polymer, light intensity, and dose. In the study, a uniform line width of Ag electrodes for large-sized PDPs was obtained by optimization of the photosensitivity of the paste with a suitable photoinitiator amid many process variables. This revealed that a photoinitiator content of 15 wt % on the basis of the monomer led to higher photosensitivity of the paste, regardless of the light dose; in particular, the photoinitiator HSP-188 exhibited the highest photosensitivity with a step level of 9.

However, the molecular weights and acid values of the binder polymers little affected the photosensitivity of the paste. Although it changed greatly with the light intensity, the photosensitivity of the paste was saturated over a light dose of 300 mJ/cm². Despite deviations, such as the light dose ranging from 250 to 350 mJ/cm² and the light intensity ranging from 15 to 25 mW/cm², the results for the variation of line width gave rise to good line uniformity, with standard deviations of 0.44 and 0.62 when the photosensitive paste contained a photoinitiator content of 15 wt % on the basis of the amount of the monomer. These results could be expected to be used in recent PDP production processing to prevent image quality errors or so-called mura caused by nonuniformity of line width, especially in large-sized PDPs.

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