

# Development of High-Definition Aqueous Polyvinylpyrrolidone Photoresists for Cathode Ray Tubes

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**ABSTRACT:** Aqueous photoresists that are capable of higher resolutions than are currently employed by the cathode ray tube (CRT) industry were developed by combining photoactivators, which activate at shorter UV wavelengths with polyvinylpyrrolidone (PVP). Two photoactivators were synthesized: 4,4'-diazido-2,2'-biphenyl disodium disulfonate (DABP), which has a maximum absorbance at 264 nm, and 4,4'-diazido-2,2'-biphenylethane disodium disulfonate (DABPE), which has a maximum absorbance at 258 nm. The PVP/DABP and PVP/DABPE photoresists successfully im-

aged a pattern with resolution as small as 4.4  $\mu\text{m}$  with photoactivator concentrations greater than 20% of the PVP concentration. Addition of silane and emulgen greatly improved the performance of the photoresists with more uniform coatings of thicknesses of up to 1.4  $\mu\text{m}$ . © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 1637–1644, 2006

**Key words:** polyvinylpyrrolidone (PVP); aqueous photoresist; cathode ray tube (CRT); display screening

## INTRODUCTION

In the CRT industry, increased resolution is being pursued due to the evident change from analog television to high-definition television (HDTV), particularly for high-resolution graphics computer displays. The resolution of a CRT is mainly determined by the size of the phosphor stripes made by a photolithography technique; the typical line width of a single color stripe for an analog television is  $\sim 300 \mu\text{m}$ , whereas a HDTV will require a width of  $\sim 75\text{--}100 \mu\text{m}$ .<sup>1</sup> During the photolithography process, UV light passes through an aperture grill, which acts as a mask with its many narrow slits, which causes diffraction of the light to occur, which is a function of both slit width and wavelength of light. Using a lower wavelength of light for exposure will reduce the diffraction effects and increase resolution, but the photoresist must be activated at this lower wavelength.

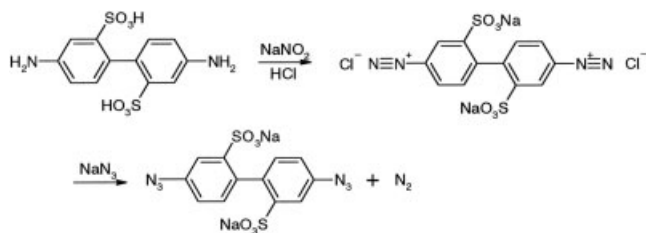
The traditional photoresist used in CRT manufacturing is PVA (polyvinyl alcohol)/ADC (ammonium dichromate) which has been used to deposit both the phosphors and the carbon black matrix.<sup>2</sup> Since the mid-1990s, PVP (polyvinylpyrrolidone)/DAS (4,4'-diazido-2,2'-stilbene disulfonic acid sodium salt) has replaced PVA/ADC for the carbon black matrix deposition.<sup>3</sup> The PVP/DAS photoresist offers higher res-

olution imaging since it absorbs at 335 nm compared to 385 nm for PVA/ADC, and it is more environmentally benign since it does not contain any toxic metals, such as chromium. Both PVA and PVP photoresists are water soluble. During the photolithography process, the UV radiation that penetrates through the mask cause crosslinking of the photoresist, making it insoluble to water. The pattern is then developed with water, which washes away the uncrosslinked photoresist. In our previous study, the fundamental chemical and physical properties of the PVP polymer were studied in which a correlation between the viscosity, *K*-value, and polymer wt % was formulated.<sup>4</sup> Also, in our second article, a review of display processing using PVP can be found, as well as the results of our investigation of the reaction kinetics of the PVP/DAS photoresist with exposure to different lighting conditions and to varying amounts of oxygen.<sup>5</sup>

Diffraction is a function of wavelength of light, slit width, and distance between the mask and the photoresist.<sup>6</sup> The distance of the mask is fixed for a CRT display of a given size, and the slit width needs to be narrower for higher resolution screens. For proximity printing, if a photoresist that absorbs at 264 nm is used, the line width that can be imaged can be decreased by about 15% compared to PVA/ADC and 11% compared to PVP/DAS.<sup>7</sup>

In this research, PVP was chosen as a starting point to develop aqueous photoresists with new photoactivators similar to DAS, but with shorter conjugation lengths, which absorb at shorter UV wavelengths. By changing just the photoactivator, all the benefits of the proven PVP photoresist system would be retained

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Scheme 1 Sandmeyer reaction with azide work-up.

with activation at a lower UV wavelength to image smaller features.

Diazides have been used as photoactivators in photoresist systems, but usually in organic solvents as they are not water soluble. The polymer portions of these photoresists from the literature are cyclized polyisoprene,<sup>8</sup> poly(vinylphenol),<sup>9</sup> and polymethyl methacrylate.<sup>10</sup> However, the only commercially available diazide photoactivator was DAS, which has been used with PVP, which is water soluble.<sup>11,12</sup> Therefore, the synthesis of azides was investigated. Several reactions for synthesizing aryl azide compounds exist.<sup>13</sup> Since 4,4'-diaminobiphenyl-2,2'-disulfonate sodium salt and water-soluble diamine compounds were commercially available, the Sandmeyer reaction was chosen to produce two diazide photoactivators: 4,4'-diazido-2,2'-biphenyl disodium disulfonate (DABP) and 4,4'-diazido-2,2'-biphenylethane disodium disulfonate (DABPE). Then, aqueous solutions of PVP and the photoactivators were made and tested as photoresists.

## EXPERIMENTAL

### Synthesis of the diazide photoactivators

To synthesize DABP, the Sandmeyer reaction was performed using 4,4'-diamino-2,2'-biphenyl disulfonic acid (Alfa Aesar) as the precursor, followed by an azide work-up, as shown in Scheme 1. The fine purple powder diamine (0.342 g) was readily dissolved in 88.0 mL of water in a 250-mL two-necked round bottom flask, which was placed in an ice bath. The diamine compound was reacted with nitrous acid (in slight excess of two times the stoichiometric ratio of diamine) to convert the diamine compound to a diazonium salt. Then, 2.0 mL of hydrochloric acid was added slowly to the flask; 1.50 g of sodium nitrite dissolved in 5.0 mL of water was added dropwise to the solution. The solution was then stirred for 1 h. Sodium azide (1.40 g) dissolved in 5.0 mL of water was then added dropwise to the solution. The solution bubbled vigorously after each drop of sodium azide solution was added. This final solution was allowed to stir for 2 h to ensure that the reaction was complete.

The solution was then evaporated in a vacuum oven at 60°C.

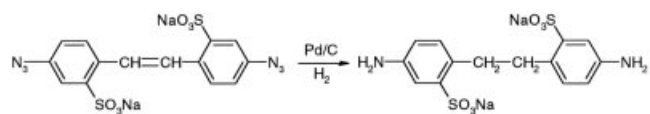
As DABPE is very similar molecularly to DAS, DAS (obtained from Sony Electronics, San Diego, CA) was used as a precursor; a hydrogenation reaction was used to convert the central double bond into a single bond as shown in Scheme 2. The DAS (1.0 g) was dissolved in 50 mL of methanol; 5 mol % Pd/C (0.27 g of 10 mol % Pd/C) was added to a thick-walled 500-mL flask. The flask was then attached to a hydrogenator or hydrogen-filled balloons.<sup>7</sup> The air inside the flask was evacuated and then filled with 80 psi of hydrogen gas. The flask was continuously agitated with a shaker and the reaction was allowed to proceed for 24 h. The Pd/C was removed using a paper filter, and the methanol was removed using a rotovap. <sup>1</sup>H-NMR was used after each hydrogenation reaction to determine the completion of the reaction. <sup>1</sup>H and <sup>13</sup>C-NMR were performed on the resulting azide compounds and the amine precursors for comparison.<sup>7</sup> Samples were dissolved in either DMSO or deuterated methanol and a spectrum was taken with a 500 MHz NMR. The hydrogenation reaction successfully converted the central double bond into a single bond, but it also converted the end azide groups into amines. The final product was an off-white colored powder compared to DAS, which has a deep orange color.

The Sandmeyer reaction followed by an azide work-up was then performed to convert these amine groups back into the azide groups to form DAPBE. An amount (0.416 g) of DAPBE was dissolved in 88.0 mL of water and placed in a two-necked round-bottomed flask, which was immersed in an ice bath. Then, the same procedure as described previously for producing DABP was followed.

### Characterization of the azides

The synthesized azide photactivators were characterized by UV-vis spectroscopy and FTIR. UV-vis spectroscopy was performed to measure the absorbance characteristics of the diazide compounds and the diamine precursors, using an HP 8452A diode array spectrophotometer. Samples were dissolved in water and placed in a quartz cuvet for measurement. Also, a 3 wt % PVP aqueous solution was also tested.

FTIR spectroscopy was performed using a Nicolet Magna-IR 550 on the final azide compounds as well as the precursors for comparison. The samples were pre-



Scheme 2 Hydrogenation reaction scheme.

pared by combining 2 mg of the sample with 0.2 g of KBr and grinding with a mortar and pestle. The mixture was then formed into a pellet and the FTIR spectrum was taken.

### Production and deposition of photoresists

Each of the synthesized photoactivators and DAS were mixed with K90 PVP (BASF) and water to produce a photoresist. (A *K*-value between 80 and 90 means that PVP has a molecular weight in the range of 900,000–1500,000.<sup>14</sup>) The PVP/DAS, which was used as a standard, was produced with 2 wt % PVP and 0.2 wt % DAS or 3 wt % PVP and 0.3 wt % DAS. The test photoresists were produced with either 2 or 3 wt % PVP and photoactivator concentrations that ranged from 0.2 to 1.2 wt %.

Solutions were also prepared with 0.05 wt % of both silane ((3-(2-aminoethylamino)propyl)trimethoxysilane), an adhesion promoter, and emulgen (polyoxyethylene octylphenyl ether), a surfactant.

Samples were spin-coated using a specialty coating systems, Spincoater Model P6700, with speeds of 100–300 rpm onto glass microscope slides or 3" × 3" squares of glass or quartz. The rotational speed was slowly increased to the chosen speed, and then held constant for 10 min to produce a uniform coating that was mostly dry. The samples then underwent a pre-exposure bake at 80°C for 10 min to evaporate all the water from the photoresist.

For each set of experiments a different mask was utilized based on the dimensions of the patterns of the mask, optical characteristics (i.e., UV transmittance), and functionality. The first mask used was created from an aperture grill of a 27" Sony Trinitron television. The metal strips of the aperture grill were glued to a square frame to keep the spacing of the slits to ~200 μm. A quartz mask with a series of lines and dots, made of chromium, of varying dimensions was used for most of the experiments. The widths of the lines ranged from 10 to 50 μm and the diameters of the dots ranged from 17 to 40 μm. To measure the highest achievable resolution of the photoresists, a 1951 USAF negative resolution target was used.<sup>15</sup>

A Sony light house, which is used in CRT manufacturing, was used for exposing the photoresists. The Sony light house utilized a high-pressure mercury lamp from ORC Science (Lake Forest, CA) with a spectrum distribution from 254 to 436 nm peaking at 365 nm (70 mW/cm<sup>2</sup>).<sup>16</sup> After exposure, the samples were developed in water to reveal the pattern. The samples were agitated slightly in a beaker of deionized water from 10 to 30 s. After developing, the samples were placed in an oven at 80°C for 10 min for drying.

### Exposure experiments

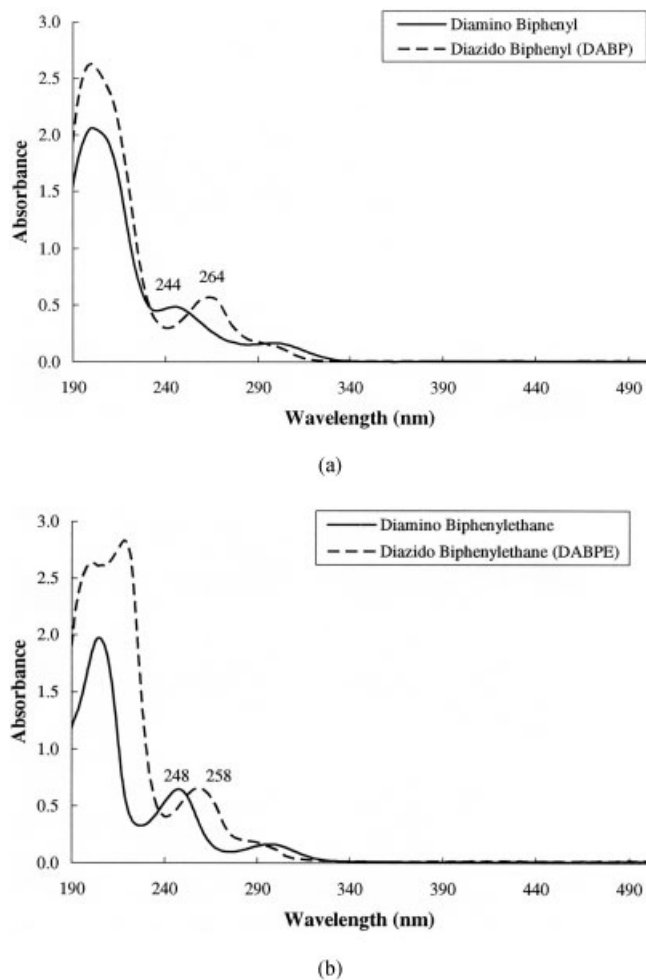
To test sensitivity, the PVP/DABP and PVP/DABPE photoresists were spin-coated onto five pieces of 3" × 3" quartz substrates. The modified aperture grill was used as the mask. The mask was placed on the back side of the quartz substrate (the side opposite of the photoresist), and samples were exposed at varying times using the Sony lighthouse. The samples were then developed in water and each sample thickness was then measured with a KLA-Tencor profilometer.

To perform the resolution target experiments, all three photoresists were spin-coated onto five pieces of 3" × 3" glass substrates. The resolution target was used as a mask to expose the pattern onto the photoresist samples. The samples were placed photoresist-side toward the mask and exposed at varying times using the Sony lighthouse. The samples were then developed to show a negative image of the resolution target. A Nikon optical microscope was then used to determine the smallest pattern that was still distinguishable as 3 bars. The group number and the element number of this pattern were then used to determine the width of each bar or resolution.<sup>16</sup> Also, a quartz mask was used for many of the experiments primarily due to its good UV transmittance of the quartz, since glass absorbs UV light especially in the deep UV region. The small line widths also gave a good indication of the resolution capabilities of the photoresists.

## RESULTS AND DISCUSSION

### Characterization of the diazide photoactivators

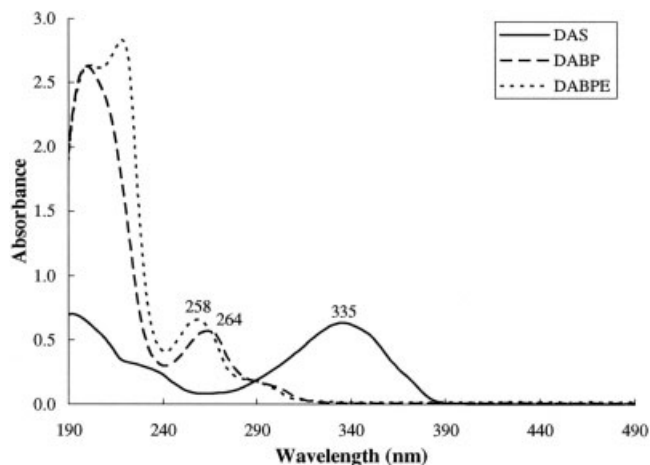
UV–vis spectroscopy was utilized to measure the absorbance characteristics of the diazide compounds and the diamine precursors. The UV–vis spectra of 4,4'-diamino-2,2'-biphenyl disulfonic acid and DABP are compared in Figure 1(a), which shows that a shift in the maximum absorbance occurred from the diamino biphenyl compound (244 nm) to the diazido biphenyl compound (264 nm) that was synthesized. The UV–vis spectra of 4,4'-diamino-2,2'-biphenylethane disodium disulfonate and DABPE are also compared in Figure 1(b). Again a shift occurs in the maximum absorbance, from 248 nm for the diamino biphenylethane compound to 258 nm for the diazido biphenylethane compound. The shift in UV absorbance from the diamino to the diazido compounds is due to an increase in the conjugation length of the molecules causing them to be excited at higher UV wavelengths, as expected. Figure 2 compares the UV spectra of DAS, DABP, and DABPE. The spectrum for DAS has a broad peak with a maximum absorbance at 335 nm. Again, a shift to lower UV absorbance is seen for DABP at 264 nm and DABPE at 258 nm with a decrease in conjugation length of these molecules compared to DAS. The



**Figure 1** UV-vis Spectra of (a) diamino and diazido biphenyl and (b) diamino and diazido biphenylethane.

peaks at about 200 nm are characteristic of benzene, which is present in all of the compounds studied. Figure 3 shows the spectrum of PVP, which has a maximum absorbance at 222 nm and should not mask the absorption of either photoactivator.

FTIR spectroscopy was performed to determine the formation of the azide during synthesis. The characteristic peak for an azide functional group is located at  $2100\text{ cm}^{-1}$ . The FTIR spectra of the diamino biphenyl and the diazido biphenyl compounds are compared in Figure 4(a). This figure shows that amine compound lacks the peak at  $2100\text{ cm}^{-1}$  and after the Sandmeyer reaction with the azide work-up, the product clearly has the peak at  $2100\text{ cm}^{-1}$  indicating that the azide compound was successfully synthesized from the amine precursor. The FTIR spectra of the diamino biphenylethane and the diazido biphenylethane compounds are compared in Figure 4(b) showing the appearance of the peak at  $2100\text{ cm}^{-1}$  with the reaction. The FTIR spectrum of DAS is compared to those of DABP and DABPE in Figure 5.

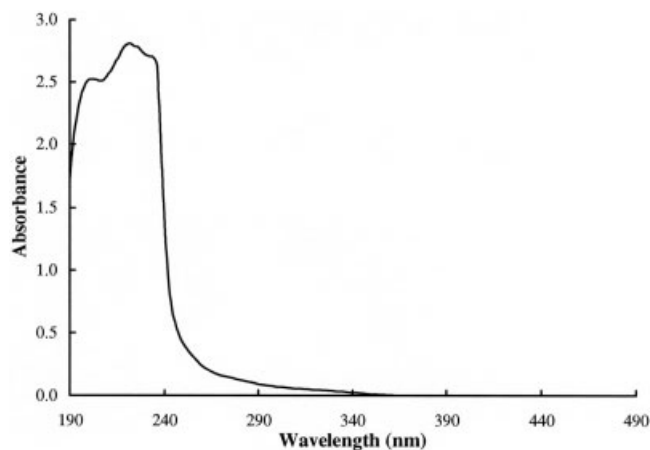


**Figure 2** Comparison of UV-vis Spectrum of DAS, DABP, and DABPE.

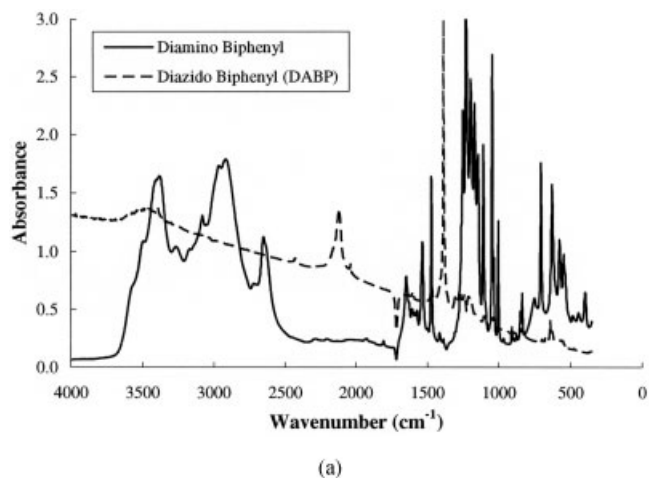
### Photoresist composition and resolution

The DABP and DABPE photoactivators were tested by mixing each with aqueous solutions of PVP, producing a pattern upon exposure and developing with water and then comparing to DAS. Many experiments were performed to make patterns with the PVP/DABP and PVP/DABPE photoresists.<sup>7</sup> The PVP concentration was varied between 2 and 3 wt % and the photoactivator concentrations used were between 0.2 and 1.2 wt %.

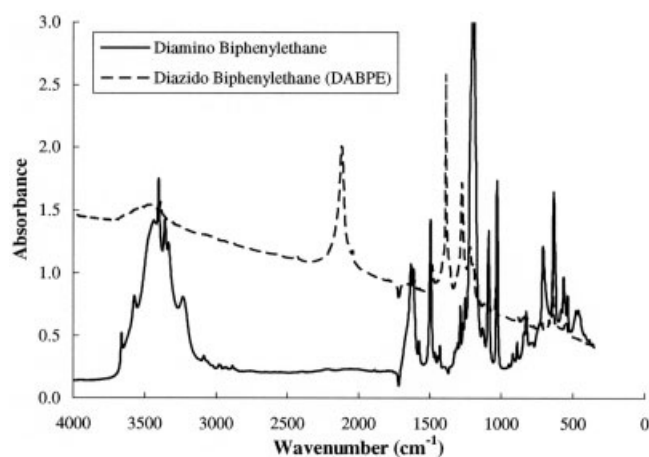
Initial experiments were performed using a 2 wt % concentration of PVP in the photoresist solutions. The DAS concentration used in industry is often 10% of the PVP concentration used, and was therefore, 0.2 wt % for a 2 wt % PVP solution. The DABP and DABPE concentrations used were 10, 20, and 40% of the PVP concentration, which equated to 0.2, 0.4, and 0.8 wt %. The photoresists were spin-coated between 150 and 250 rpm. At the faster rotational speeds, all of the



**Figure 3** UV-vis Spectrum of PVP.



(a)

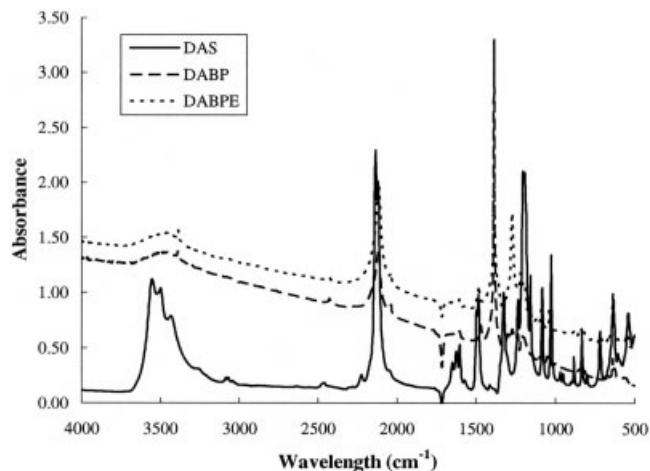


(b)

**Figure 4** FTIR spectrum of (a) diamino and diazido biphenyl and (b) diamino and diazido biphenylethane.

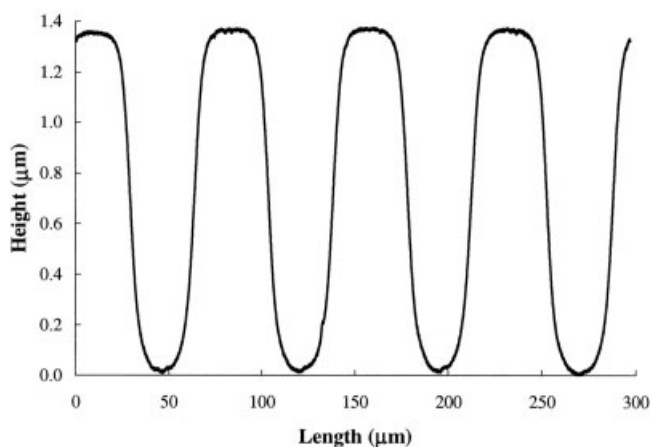
excess photoresist solution was propelled off of the substrate, which gave a more uniform coating. The samples with 0.2 wt % DABP and DABPE photoactivators did not successfully produce the pattern of the masks. However, photoresists with 0.4 and 0.8 wt % photoactivators did successfully produce the patterns for both photoactivators. Therefore, the photoactivator concentration was kept at 20% of the PVP concentration for the remainder of the experiments. Although these samples were able to produce the patterns of the masks used, the developed images were very faint. Profilometry measurements showed that the photoresist thickness ranged between 50 and 200 nm. The PVP concentration in the photoresist solutions was then increased to 3 wt %.

The 3 wt % PVP photoresist solutions increased the viscosity of the solutions. The DAS concentration was 0.3 wt % and the DABP or DABPE concentrations were 0.6 wt %. Although the photoresist thickness was increased after the spin coating step, the developed images still seemed faint. Therefore, the use of additives on the photoresist solutions was studied.



**Figure 5** Comparison of FTIR Spectra of DAS, DABP, and DABPE.

The PVP/DAS photoresist used in industry often has surfactants and other additives to increase the performance of the photoresist. Two of these additives that are often used are silane, an adhesion promoter, and emulgen, a surfactant. Thus, 0.05 wt % of silane and emulgen were added to all three photoresist solutions using 3 wt % PVP. During the spin coating process, a decrease in surface tension was observed for the photoresist solutions. After developing the samples with the silane and emulgen added, the patterns were much more apparent. Profilometry of these samples showed that the photoresist thickness after developing was about 1  $\mu\text{m}$  or greater, instead of a few hundred nanometers. Figure 6 shows a profilometry of lines for a 3 wt % PVP/0.6 wt % DABP sample with silane and emulgen, spin-coated at 200 rpm and exposed for 5 min using the Sony lighthouse. This figure shows that the photoresist thickness after developing was  $\sim 1.4 \mu\text{m}$ . Therefore, the silane and emulgen ad-



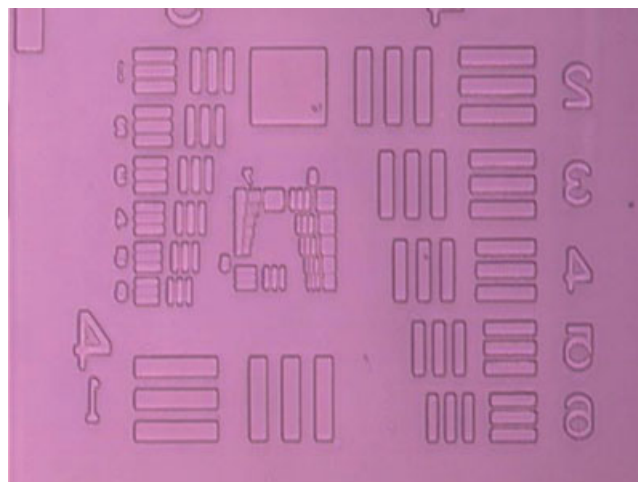
**Figure 6** Profilometry of the PVP/DABP photoresist with silane and emulgen.

dition, although in small quantities, can greatly affect the performance of these photoresists. The emulgen increases the wetting of the solution and allows for a much more uniform coating of the photoresist. The additives also seem to help keep the exposed photoresist in tact during the developing step. Therefore, the remaining experiments used photoresists with these additives.

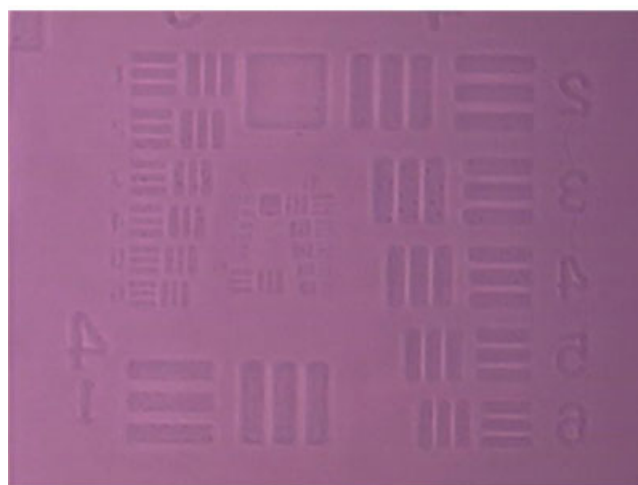
The DABP, DAS, and DABPE photoresists were deposited onto glass by spin-coating at 225 rpm for 10 min, followed by a pre-exposure bake for 10 min at 80°C. The samples were exposed using the USAF resolution target and the Sony lighthouse for various times.<sup>7</sup> Figure 7(a) shows an optical micrograph of the PVP/DAS photoresist exposed for 1 s. In this image of the PVP/DAS photoresist, distinct bars are apparent down to Group 6: Element 6, which equates to 4.4  $\mu\text{m}$  lines,<sup>16</sup> which is the highest resolution achievable under these exposure conditions. Unfortunately, the USAF resolution target was made out of soda lime glass, which absorbs almost all UV light below 300 nm.<sup>7</sup> Although the DABP and DABPE photoactivators have their maximum absorbances at 264 and 258 nm, respectively, they do absorb in the 300 nm range to a lesser extent. Therefore, exposure of these photoactivators was possible through the USAF resolution target, but longer exposure times were required between 30 and 180 s. Figure 7(b,c) compared the images of the PVP/DABP and the PVP/DABPE photoresists exposed for 180 s, respectively. Both of these photoresists gave similar results, and again the smallest set of distinct bars is Group 6: Element 6, which equates to 4.4  $\mu\text{m}$  lines.<sup>16</sup> If the same experiments were performed using a mask made out of quartz instead of glass, it is conjectured that the resolution would be much improved.

With the PVP/DABP photoresist using a quartz mask, profilometry measurement were made of 25  $\mu\text{m}$  lines with exposure times from 15 to 300 s and 10  $\mu\text{m}$  lines with exposure for 15 and 300 s as shown in Figure 8. The PVP/DABPE photoresist was also successful in imaging 25 and 10  $\mu\text{m}$  lines as shown in Figure 9. Both Figures 8 and 9 show an increased height of the image with increased exposure time. The data from these figures are too inaccurate to yield a meaningful sensitivity curve of image height versus exposure time. The sample exposed for 5 min produced lines with flatter tops than that for 15 s which has rounded tops. (A flat profile is preferable for CRT applications.) All three photoresists imaged the lines with flat tops, although slight footing (i.e., a wider base than top of the profile) was present for the DABP and DABPE samples.<sup>7</sup> The footing may have been caused by insufficient developing or slight overexposure of the samples.

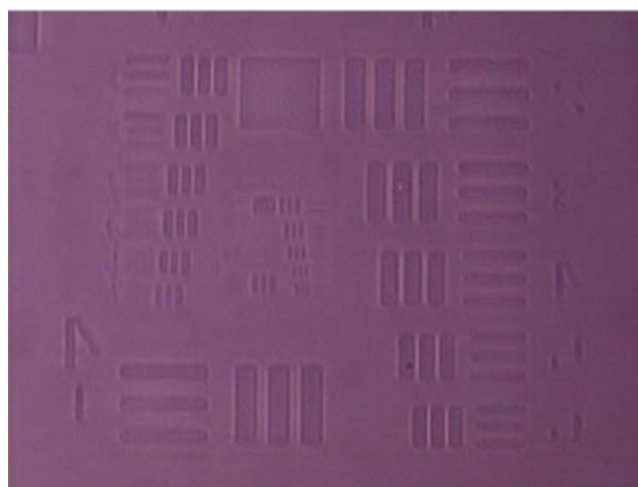
Figure 10 shows a comparison of profilometry for the three photoresists imaging 10  $\mu\text{m}$  lines. The PVP/



(a)

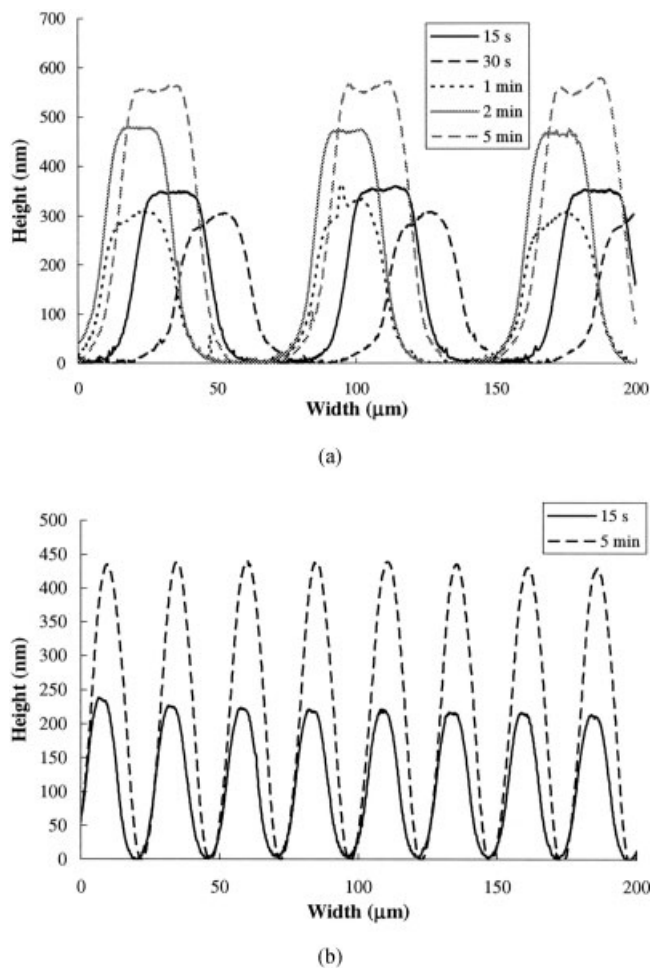


(b)



(c)

**Figure 7** USAF resolution target image of (a) PVP/DAS exposed for 1 s, (b) PVP/DABP exposed for 180 s, and (c) PVP/DABPE exposed for 180 s. [Color figure can be viewed in the online issue, which is available at [www.interscience.wiley.com](http://www.interscience.wiley.com).]



**Figure 8** Profilometry of (a) 25  $\mu\text{m}$  lines and (b) 10  $\mu\text{m}$  lines using PVP/DABP photoresist at varying exposure times.

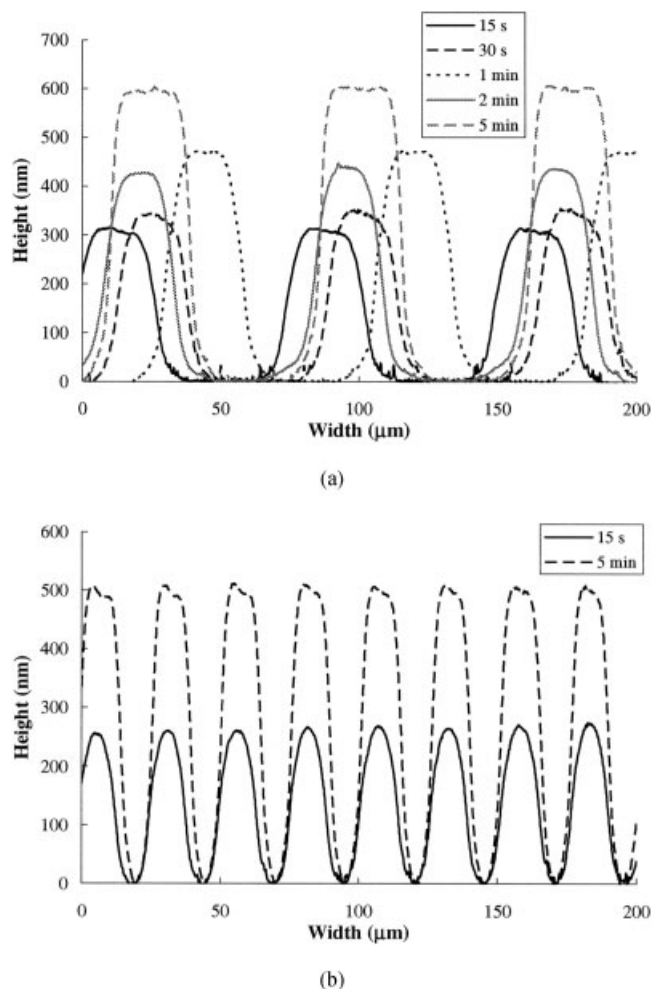
DAS sample was exposed for 1 s, while the PVP/DABP and PVP/DABPE samples were exposed for 5 min. Although all three photoresists successfully produced the 10  $\mu\text{m}$  lines, the profiles of the lines were not the same. The PVP/DABP sample had lines with pointed tops, the PVP/DABPE sample had lines with nearly flat tops, and the PVP/DAS sample had lines with flat tops. Although the high-pressure mercury lamp used does emit in the 250–270 nm range, the intensity of these emissions is only 25% that of the peak at 365 nm.<sup>16</sup> Therefore, this lamp is more suited for exposing the PVP/DAS photoresist. Utilizing a UV source with more intense emissions in the 250–270 nm range would produce better results for the PVP/DABP and PVP/DABPE photoresists.

### CONCLUSIONS

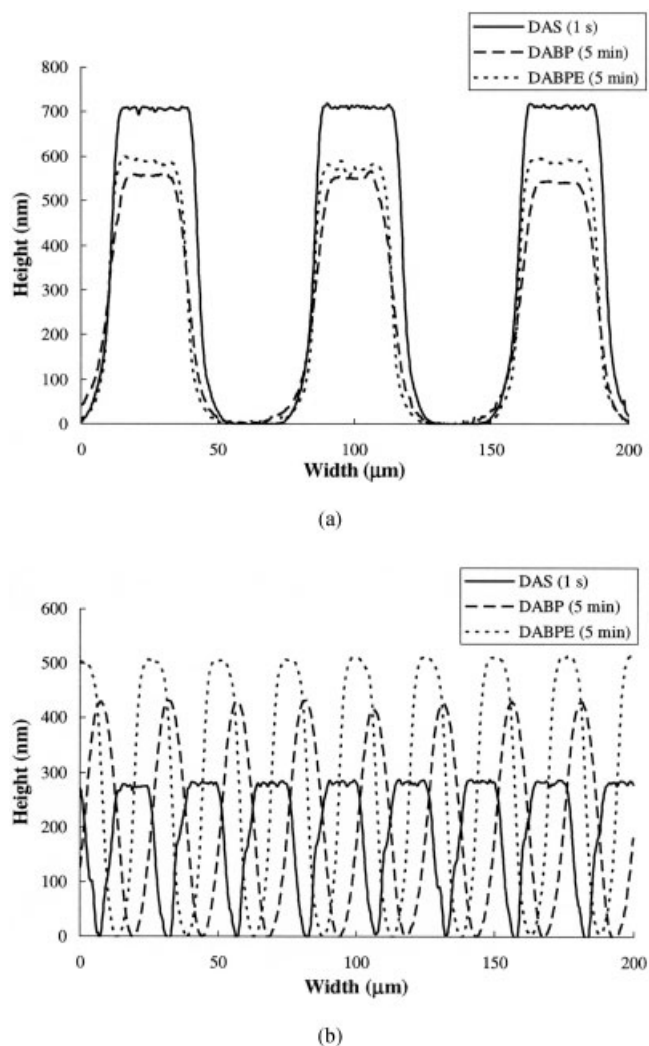
Two new photoactivators for PVP, 4,4'-diazido-2,2'-biphenyl disodium disulfonate (DABP) and 4,4'-diazido-2,2'-biphenylethane disodium disulfonate

(DABPE), were successfully synthesized, which have a maximum absorbance at 264 and 258 nm, respectively. The DABP and DABPE photoactivators were mixed with aqueous PVP to produce photoresist solutions, which were successful in producing patterns when their concentrations were greater than 20% of the PVP concentration. Addition of silane and emulgen greatly improved the performance of the photoresists. By using silane and emulgen, photoresist thicknesses of up to 1.4  $\mu\text{m}$  were obtained compared to thicknesses under 200 nm without their addition. The emulgen also decreased the surface tension of the photoresist solutions, which allowed for more uniform coatings.

The resolution tests using the 1951 USAF resolution target show that both the PVP/DABP and PVP/DABPE photoresists had similar resolution capabilities. Both photoresists could produce the pattern down to about 4.4  $\mu\text{m}$  with this mask on glass. Using a quartz resolution target and a light source with a more intense and narrower emission distribution around 250–270 nm, both photoresists should be ca-



**Figure 9** Profilometry of (a) 25  $\mu\text{m}$  lines and (b) 10  $\mu\text{m}$  lines using PVP/DABPE photoresist at varying exposure times.



**Figure 10** Comparison of profilometry of photoresists imaging (a) 25  $\mu\text{m}$  lines and (b) 10  $\mu\text{m}$  lines.

pable of imaging lines smaller than 4.4  $\mu\text{m}$ . Both photoresists also showed similar results in imaging the lines and dots with a quartz mask. The photoresists were successful in imaging the 10  $\mu\text{m}$  lines and the 17  $\mu\text{m}$  dots.<sup>7</sup> Both photoresists showed an increase in feature height after developing with increased exposure time. Although both photoresists performed very well, the PVP/DABPE photoresist performed slightly better. The lines imaged with the PVP/DABPE photoresist had straighter walls and had flatter tops than the PVP/DABP photoresist, which looked slightly undercut and had more pointed tops with the 10  $\mu\text{m}$  lines.

These tests indicate that DABP and DABPE perform well with PVP as photoresists. Since they activate at shorter UV wavelengths than DAS, they will have less diffraction effects and allow for smaller features to be imaged. The photoactivator is the only component that needs to be changed in these new PVP photoresists, and since they only comprise of about 0.2–0.6 wt % of the photoresist, the cost for switching over to these new photoactivators should be minimal. These tests were so shown proof of concept<sup>17</sup> and further optical testing with improved light sources, quartz substrates and proximity exposure may show improved performance. Also, other diazide photoactivators with added functional groups to the aryl ring may be able to be synthesized and activated at slightly shorter UV wavelengths with PVP, which may further increase sensitivity and performance.

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