Low-Temperature-Curable and Positive-Type Photosensitive Polyimide with High Mechanical Strength, High Resolution and Good Pot-Life Based on Chain Extendable Poly(Amic Acid), Thermal Degradable Crosslinker, Chain Extender, Thermal Base Generator and Photoacid Generator

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A positive-type photosensitive polyimide (PSPI) based on a chain extendable poly(amic acid) (PAA), a thermally degradable cross-linker 1,3,5-tris[(2-vinylxyloxyethoxy]benzene (TVEB), a photoacid generator (PAG) (5-propylsulfonyloxyiminom-5H-thiophene-2-yldene)-(2-methylphenyl)acetonitrile, a chain extender diphenyl isophthalate (DPI) and a thermal base generator (TBG) t-butyl 2,6-dimethylpiperidine-carboxylate has been developed. The PAA prepared from 3,3′,4,4′-biphenyltetraacyclic dianhydride and 4,4′-oxydianiline was end-capped with di-tert-butyl dicarbonate, and undergoes a chain extending reaction during a curing stage to constitute a linear structure. This advancing PSPI exhibited high resolution of 3 μm, good mechanical properties, low residue content of cross-linker, low imidization temperature (250 °C) catalyzed by TBG, preventing corrosion of Cu circuits due to neutralization of photogenerated acids with bases from TBG and the extended pot-life by using the chain extender with high hydrolytic stability, inducing a wide-ranging applicability.

Keywords: Positive-type photosensitive polyimide, Chain-extendable poly(amic acid), Photoacid generator, Thermal base generator, Low-temperature curable, Good pot-life

1. Introduction

Polyimides (PIs) are topnotch constructing polymers, which are widely used as buffer coatings, passivation layers and insulation layers, for the redistribution in microelectronics, due to their exceptional thermal stabilities and excellent mechanical performances [1-5]. To obtain the miniaturization properties in most of the applications, adoption of photolithography techniques is highly required [6-10]. The conventional photolithographic process widely used in this area is to make a PI pattern by etching via a photoresist pattern. Lately, photosensitive PIs (PSPIs) are being noticed broadly, because of their simple process for streamlined patterning without the use of any photoresist. Amongst the two types of PSPIs, namely positive-type PI and negative type PI, more attempts have been made to develop the positive-type PSPIs compared to the negative PSPIs; by virtue of their several advantages, such as a lower swelling effect resulting in higher resolution and the use of an environment-friendly alkaline aqueous solution as a developer. Additionally, formation of a V-type pattern by the positive-type PSPIs is also a highly desired property in wire-bonding processes. Despite of those interesting advantages, positive type PSPIs are rarely explored due to the unacceptable high dissolution rate of the...
fabricated precursor PAAs by the developer; furthermore, it is incapable to create a reasonable dissolution contrast between the unexposed and exposed areas owing to the high acidity of the carboxylic acid groups on the main chain of PAAs. Accordingly, only limited alkaline-developable positive-type PSPIs have been reported in which cross-linked, partially esterified and fluorinated PAAs are used to reduce the dissolution rate in the alkaline developer [11-15].

Cross-linking reaction between PAA molecular chains decreases the dissolution rate appropriately with the 2.38 wt% TMAH \textsubscript{aq} solution. Ueda and colleagues reported a positive type PSPIs with a multi-functional cross-linker and chemical amplified mechanism in 2009 [16]. The lithographic composition was comprised of PAAs, a vinyl ether cross-linker, a TBG and a PAG. Despite of having this thoroughly simplified configuration and the sharpened pattern made by these PSPIs, the residual cross-linker after curing process dramatically detriment the mechanical properties of the film. With the intention of overcoming the constraints, Hsu and colleagues reported a positive type PSPIs with a thermally degradable cross-linker and chain-extending mechanism of PAAs in 2020 [17]. This lithographic composition was comprised of N-Boc PAAs, a vinyl ether cross-linker, a chain extender tetracarboxylic dianhydride and a PAG which has controlled molecular weight for lithography and goes through a chain-extending reaction in curing stage. These PSPIs accentuate both high resolution and great mechanical properties. However, the low hydrolytic stability of the chain extender, corrosion of Cu circuits in microchips by a photogenerated acid from PAG \textsuperscript{[18]} and high imidization...
multi-functional cross-linker and chemical colleagues reported a positive type PSPIs with a 2.38 wt% TMAH aq solution. Ueda and chains decreases the dissolution rate appropriately alkaline developer [11-15]. PAAs are used to reduce the dissolution rate in the cross-linked, partially esterified and fluorinated positive-type PSPIs have been reported in which accordingly, only limited alkaline-developable carboxylic acid groups on the main chain of PAAs. exposed areas owing to the high acidity of the dissolution contrast between the unexposed and furthermore, it is incapable to create a reasonable amplified mechanism in 2009 [16]. The lithographic composition was comprised of PAAs, a vinyl ether cross-linker, a chain extender and great mechanical properties. However, the low temperature reduce the opportunity of commercialization. Park and colleagues reported a positive type PSPIs with an esterification of PAA and end-capped with a reactive end-capper, 5-norbornene-2,3-dicarboxylic anhydride (NDA) [19]. The poly(dicarboxylic acid) (PAE) effectively reduced the dissolution rate of PAA to get a 5-micron pattern and the cross-linking of NDA results in increased mechanical properties. In this process, the cross-linking of NDA starts at 270 °C via a reverse Diels–Alder reaction method. It is clearly understood that PAEs required higher temperature for imidization compare to PAAs [20]. Apart from this, the reactive end group forms a cross-linking network which brings brittleness to the film. Consequently, the NDA end-capped PAE is unable to imidize at low temperature and to achieve high elongation.

In the light of these considerations, we report a more advanced PSPI configuration based on a chain-extendable PAA end-capped with N-Boc group, a thermal degradable crosslinker 1,3,5-tris(2-(vinyloxy)ethoxy)benzene (TVEB), a photoacid generator PAG (5-propylsulfonyloxyimino-5H-thiophen-2-ylidene)-2-(methylphenyl)acetonitrile (PTMA), a chain extender diphenyl isophthalate (DPI) with high hydrolytic stability and a thermal base generator TBG, t-butyl 2,6-dimethylpiperidine-carboxylate (BDPC). The synthesis of chain extendable PAA end-capped with N-Boc group is carried out sequentially. Initially, the polymer chain was prepared by reacting 3,3',4,4'-biphenyltetracarboxylic dianhydride (BPDA) and 4,4'-oxydianiline (ODA) in the ratio of 0.92 to control the molecular weight below 10,000 Da to improved resolution. Later, the obtained polymer chain was end-capped with di-tert-butyl dicarbonate (DIBOC), and then finally in the last stage by adding 0.16 equivalent of diphenyl isophthalate (DPI) chain-extension was prompted in the curing stage (Scheme 1). The configuration of PSPI is archetypal, TVEB, PTMA, BDPC and DPI were dissolved in the PAA solution. First, the film was coated using spin-coater and then baked on a hot plate. In this prebake stage (PB), the reaction between carboxylic acid group of PAAs and vinyl group of TVEB forms a cross-linkage, which would be responsible to decrease the dissolution rate of PAAs in TMAHaq solution. In the next stage, the film was exposed to i-line radiation, then the area of the film which is exposed to i-line generates propanesulfonic acid from PTMA. Furthermore, in this post-exposure bake (PEB) stage of the PAA film, the cross-linked networks in the exposed areas will break down by the generated propanesulfonic acid; due to the acid-catalyzed cleavage of the hemiacetal ester group formed in the cross-linked reaction. Later in the developing stage, a positive tone image was composed by developing the film in a 2.38 wt% TMAHaq. Moreover, the dissolution rate of PAA in the exposed area was relatively very high, and the dissolution rate contrast between the exposed and unexposed areas is considerably high enough to make a pattern with a resolution of 3 μm. At last in the imidization stage, decross-linking reaction occurs around 140 °C. The catalyzed imidization of the PAA also starts at 150 °C with the aid of in-situ generated dimethylpiperidine from the thermal decomposition of BDPC. Simultaneously, the deprotection of N-Boc groups on the ends of the PAA molecular chains occurs and produces the amine groups. Afterwards, the deprotected PAA molecular chains react with the DPI in the film at 160–250 °C; which would lead to the extension of PAA molecular chain constituting a linear structure (Scheme 2). Furthermore, the base generated from TBG neutralizes the acid from PAG. Hence, this advanced PSPI possess several advantages, such as high resolution, good mechanical properties due to the low residual content of cross-linker, no possibility of corrosion of Cu circuits, low imidization temperature and extended pot-life by using the chain extender with high hydrolytic
stability, which are otherwise highly desired for varied range of applications.

2. Experimental

2.1. Materials
Anhydrous N-methyl-2-pyrrolidone (NMP) dimethyl sulfoxide (DMSO), 1,1,1-tris(hydroxymethyl)propane and phloroglucinol were purchased from Acros. 3,3’,4,4’-biphenyltetracarboxylic dianhydride (BPDA) and 4,4’-oxydianiline (ODA), and 2-chloroethyl vinyl ether were purchased from TCI. Di-tert-butyl dicarbonate (DIBOC) and 2.38 wt% TMAH aqueous solution were purchased from Alfa Aesar. A photoacid generator (PAG) (5-propylsulfonyloxyimino-5H-thiophene-2-ylidene)-(2-methylphenyl)acetonitrile (PTMA) was obtained from Changzhou Tronly New Electronic Materials company. Potassium carbonate, 2,6-dimethylpiperidine, 4-(dimethylamino)pyridine and diphenyl isophthalate (DPI) were purchased from Sigma-Aldrich. Other reagents and solvents were used as received.

2.2. Polymer synthesis
Oligomer PAA end-capped with N-Boc group poly(amic acid) was prepared by the addition of BPDA (4.60 g, 15.6 mmol) in a powder form to a mechanically stirred solution of ODA (3.40 g, 17.0 mmol) in NMP (32 mL) under a nitrogen atmosphere. The polymerization was carried out at room temperature for 8 h. DIBOC (0.593 g, 2.72 mmol) was then added to this solution and reacted further for 2 h. DIBOC introduced poly(amic acid) solution was cooled down to room temperature and kept at −15 °C. The molecular weight is found to be 7500 Da which is determined by 1H NMR.

2.3. Characterization of degree of imidization
The polymer solution (PAA) with solid content of 20 wt% was spin-coated on a silicon wafer, baked at 80 °C on a hotplate for 60 min in air. A reference PI film was prepared by heating at different condition for 1 h. Absorption intensities on a FTIR spectrum at 1376 cm⁻¹ (A₁₃₇₆) attributed to the C=N stretching of an imide group and at 1500 cm⁻¹ (A₁₅₀₀) attributed to C=C stretching of a phenyl group were measured, and the degrees of imidization were determined using the following equation:

\[
\text{Imidization (\%)} = \frac{A_{1376}}{A_{1500(\text{samp})}} - \frac{A_{1376}}{A_{1500(\text{init})}} \times 100 \quad (1)
\]

where subscripts between parentheses that followed \(A_{1376}/A_{1500}\) in the equation indicate the states of the polymer films; for example, (samp) is the polymer sampled at each heating temperature (100–250 °C); (init) is initially prebaked PAA at 80 °C for 1 h and (imide) is the fully cured polymer 350 °C for 1 h in air.

2.4. Dissolution rate
The PAA films with 4-μm thickness were obtained by spin-coating from the solutions on glass wafers. These films were pre-baked under each condition and then exposed to a filtered super-high pressure mercury lamp at 365 nm (i-line). The dissolution rate (Å/s) of the film was determined from the changes obtained in the film thickness before and after the development with the mixture of 2.38 wt% TMAH₃aq followed by rinsing with water.

2.5. Photosensitivity
The photosensitive polymer film with 4-μm thickness was prepared by dissolving TVEB, PTMA, TBG and DPI into the N-Boc PAA in NMP. The composition was N-Boc PAA (77.1 wt%), TVEB (7.7 wt%), PTMA (3.5 wt%), TBG (7.7 wt%) and DPI (4 wt%). The solution was spin-coated on a glass wafer, prebaked at 110 °C for 3 min, and exposed at a wavelength of i-line with different exposure dosage. The PEB was carried out at 90 °C for 2 min, then developed with a 2.38 wt% TMAH₃aq for 4 sec at room temperature and finally rinsed with water. A characteristic photosensitive curve was obtained by plotting a normalized film thickness against the exposure dose (unit: mJ cm⁻²).

2.6. Measurements
The 1H spectra were obtained on an Agilent Unity-400 MHz (1H at 400 MHz). Fourier transform infrared (FTIR) spectra were obtained with a Perkin Elmer FTIR Spectrum One spectrophotometer. The film thickness was measured with a Kosaka Laboratory Ltd ET-4000 surface profiler. The exposure to irradiation at a wavelength of i-line was carried out by a Kyowariken K-310P-100S. Film strips of 80-mm length and 25-mm width were prepared by cutting PI films, and mechanical properties of PI films were measured using a Shimadzu testing machine (EZ-L) by tensile test mode with a testing rate of 5 mm/min. Inherent viscosity measurements were carried out using an Ostwald viscometer, the sample concentration is 0.5 g dL⁻¹ in NMP at 30 °C.
3. Results and discussion

3.1. Selection of cross-linker, PAA, chain extender and thermal base generator

It is highly desired for any cross-linker to be thermally stable both in the PB and PEB stages within the range of 90~130 °C for chemically amplified system. On the other hand, thermally degradable cross-linker is much preferred in the curing stage, to decrease the residue of it in the PI film for better mechanical property. For that reason, Okazaki et al. [21] used TVEB as a thermally degradable cross-linker; the undecorated aromatic structure of TVEB resulted a lower $T_d$ of 222 °C; moreover, with a suitable compatibility between TVEB and PAA. The biphenyl-type PAA is having a relatively rigid structure with high glass transition temperature, which in turn produces a moderate coefficient of thermal expansion (CTE) value. Furthermore, PAA also possesses high flexibility due to its higher elongation property at break. Accordingly, a biphenyl-type PAA derived from ODA and BPDA was selected as the polymer matrix.

Molecular weight control of PAA is crucial in lithography and most commonly, the molecular weight of PAA below 10,000 Da is much suitable to carry out lithography. Lower molecular weight enhances the dissolution rate of the PAA in TMAH$_{aq}$ and could achieve higher contrast. Conversely, higher molecular weight provides acceptable mechanical properties. Taking both the factors into consideration, we have adopted end-capping of PAA with tert-butyl carbamate groups to control the molecular weight of PAA and also utilized DPI to serve as a chain extender. In the lithographic stage, the N-Boc PAA with low molecular weight exhibited a moderate dissolution rate for making a pattern with high-resolution. Whereas, in the curing stage, the tert-butyl carbamate group was deprotected automatically to leave a primary amine group at 150 °C which was reacted smoothly with DPI in the matrix around 160~170 °C to produce a PAA with higher molecular weight.

The phenol group of DPI served as a good leaving group for the substitution reaction with the primary amine group; and also the meta substituted structure increased the solubility of DPI in the NMP solvent. Compared to the conventional reactive end group process, which requires high proceed temperature to form a cross-linking network, the chain extending process occurs at a temperature below 200 °C; which made the low-temperature imidization process feasible, moreover the linear structure ultimately provided the desired higher elongation. Besides, the high hydrolytic stability of DPI, it delivers an extended pot-life too. PAAs are generally converted to PIs through thermal imidization at closely 350 °C; however, the high operating temperature would damage the electronic devices very easily. We have adopted BDPC as an effective TBG, by following the earlier published data reported in Ueda et al.; [22] which could easily facilitate a solid-phase imidization of PAA below

| No. | BDPC (wt%)$^c$ | TVEB (wt%)$^c$ | PTMA (wt%)$^c$ | Curing temp. (°C) | Degree of imidization (%) | Modulus (GPa) | Tensile strength (MPa) | Elongation (%) |
|-----|----------------|----------------|----------------|------------------|--------------------------|--------------|----------------------|---------------|
| 1   | -              | -              | -              | 350              | 98.9                     | 1.53         | 130                  | 18.9          |
| 2$^b$ | -              | -              | -              | 350              | 99.4                     | 1.43         | 116                  | 21.3          |
| 3   | 10             | -              | -              | 250              | 99.5                     | 1.46         | 117                  | 15.7          |
| 4   | 10             | -              | -              | 200              | 98.7                     | 1.34         | 109                  | 12.2          |
| 5   | -              | 15             | 4.5            | 350              | 99.0                     | 1.44         | 119                  | 15.0          |
| 6   | 10             | 15             | 4.5            | 250              | 99.2                     | 0.87         | 66.1                 | 7.02          |
| 7   | 10             | 15             | 4.5            | 200              | 99.1                     | brittle      | brittle              | brittle       |
| 8   | 10             | 10             | 4.5            | 250              | 98.8                     | 1.41         | 114                  | 14.5          |

$^a$ Molar ratio of ODA/BPDA/DIBOC/DPI = 1/0.92/0.16/0.08. Curing condition: heating from 50 °C to $T_{ini}$ °C at the rate of 4 °C min$^{-1}$ and fixing at $T_{ini}$ °C for 1 h without irradiation.

$^b$ Using BPDA as chain extender.

$^c$ wt % compare to PAA.
data reported in Ueda et al.; [22] which could easily facilitate a solid-phase imidization of PAA below 200 °C. Moreover, 2,6-dimethylpiperidine generated from BDPC neutralizes the acid from PAG, preventing corrosion of Cu circuits and would evaporate at the curing stage itself without affecting any of the mechanical properties.

3.2. Mechanical properties

Table 1 summarizes the mechanical properties of PI films prepared from N-Boc PAA with different loadings of TBG, TVEB and PAG. First, the chain extension induced by DPI and BPDA were compared in same loading and curing conditions (Table 1, entries 1 and 2). The results showed similar mechanical properties and demonstrated the equivalent effect of chain extension caused by both DPI and BPDA. However, the DPI induced films showed more rigid properties, such as higher modulus, higher tensile strength and lower elongation, compared to the BPDA films which contains flexible units. In order to understand the temperature effect, the degree of imidization of PI film catalyzed by BDPC at 200 and 250 °C are recorded by FTIR (Table 1, entries 3 and 4). The results showed that the mechanical properties are strongly correlated to the curing temperature. It is clearly understood that even with the high degree of imidization, high $T_g$ of the PI (270 °C) [23] hinders the packing of polymer chains compared to the high curing temperature (350 °C). In addition, the inadequate chain extension at the lower temperature also may result in poor mechanical properties. Next, the effect of loading TVEB on the mechanical properties was studied in detailed. From the results (Table 1, entries 5 to 8) it is understood that the loading of TVEB would deplete the mechanical properties. It is evident that the small molecules would remain in the PI film at the curing temperature higher than the $T_d$ of TVEB and would destroy the packing of the polymer chains (Table 1, entries 5 and 6). Furthermore, the PI film became brittle at the curing temperature below the $T_d$ of TVEB (Table 1, entry 7). However, with the lower loading of TVEB and at reasonable curing temperature, the mechanical properties of the PI film exhibit an adequate quality for the industry requirement (Table 1, entry 8).

3.3. Lithographic evaluation

The effects of TVEB loading (compared to pristine N-Boc PAA), prebake temperature, PEB temperature, and exposure to the UV light were investigated to get a good contrast between the exposed and unexposed areas. The film was obtained by spin-casting a solution of N-Boc PAA containing TVEB and PTMA on a glass plate, and then prebaking at a set temperature for 3 min in air. This photosensitive polymer film was irradiated with UV light at 365 nm ($i$-line) using a super high-pressure mercury lamp, baked after exposure at a set temperature, and developed with a 2.38 wt% TMAH$_{aq}$ at room temperature. To explicate the difference of dissolution behavior between the exposed and unexposed areas, the dissolution rates were estimated by the change in film thickness before and after development.

![Fig. 1. Effect of TVEB loading to PAA on the dissolution rate for the PAA/PTMA (95.5/4.5 wt/wt) resist system in exposed (open diamond) and unexposed area (solid square). The prebake, the $i$-line exposure, and PEB were fixed as 110 °C for 3 min, 200 mJ cm$^{-2}$ and at 90 °C for 2 min, respectively.]

![Fig. 2. Effect of prebake temperature on the dissolution rate for the PAA/TVEB/PTMA/TBG/DPI (77.1/7.7/3.5/7.7/4 wt/wt/wt/wt/wt) resist system in exposed (open diamond) and unexposed area (solid square). The prebake time, $i$-line exposure, and PEB were fixed for 3 min, 200 mJ cm$^{-2}$ and at 90 °C for 2 min, respectively.]


The effects of TVEB loading (compared to Boc PAA), prebake temperature, PEB (77.1/7.7/3.5/7.7/4 wt/wt/wt/wt/wt) resist system with the following condition. The prebake, the i-line exposure and PEB were fixed as 110 °C for 3 min, 200 mJ cm⁻² and at 90 °C for 2 min, respectively.

First, the effect of TVEB loading on the dissolution rate of the film was investigated (Fig. 1), where each film was prebaked at 110 °C for 3 min, exposed to 200 mJ cm⁻², and then post exposure baked at 90 °C for 2 min. The dissolution rate of the unexposed area decreases by means of increasing TVEB loading and becomes under 1,000 Å/s with 10 wt% TVEB loading to the N-Boc PAA, while the dissolution rate of exposed area is around 10,000 Å/s. The dissolution contrast (DC) between the exposed and unexposed areas in the 2.38 wt% TMAHₐq exceeds 10 times.

Next, the effect of the prebake temperature on the dissolution rate was investigated and shown in Fig. 2. The cross-linking density generated in the form of hemiacetal ester is insufficient at 90 °C and results in the excessive dissolution rate of unexposed area. As the temperature raised above 100 °C, the cross-linking density is adequate to decrease the dissolution rate of unexposed area and to be decross-linked by propanesulfonic acid generated by PTMA in the exposed area. As a result, the DC is more than 10 times between 100–120 °C. When the temperature reached 130 °C, the hemiacetal ester was reversibly converted into carboxylic acid and vinyl ether and lead to an increment of dissolution rate of unexposed area.

Then, the effect of PEB temperature was studied, as shown in Fig. 3. By altering the PEB temperature from 90 to 130 °C, an adequate DC is obtained in the range of 90–120 °C, which indicates that an acidolytic de-crosslinking by PTMA occurs at exposed area in a wide temperature range. Analogous to prebake stage, a thermal de-crosslinking happened at 130 °C and caused an increment of dissolution rate of unexposed area.

Finally, the sensitivity curve for the 4-μm thick polymer film shown in Fig. 4 indicates the excellent sensitivity \( D_0 \) of 40 mJ cm⁻² and a good contrast \( \gamma_s \) of 4.2 based on the basis points of these preliminary optimization studies.

3.4. Image formation of PSPI

The SEM image in Fig. 5 declares the result of PI positive pattern after a series of treatments, such as...
prebake at 110 °C for 3 min, exposure to 200 mJ cm\(^{-2}\) of \(i\)-line, post-baking at 90 °C for 2 min, and developing with a 2.38 wt% TMAH\(_{aq}\). A clear, positive PAA pattern with a 3-μm feature was observed when a 4-μm thick film was used. The printed pattern was converted to the PI pattern without deformation by heating at elevated temperatures up to 250 °C for 1 h in nitrogen (Fig. 5).

4. Conclusion

A five-component PSPI based on a chain extendable PAA, PTMA as a PAG, TVBE as a thermally degradable cross-linker, BDPC as a TBG, DPI as a chain extender has been elaborated to evolve into next generation PSPIs and approve (i) direct formulation of PSPIs using chain extendable PAAs, (ii) high sensitivity and high-resolution pattern formation, (iii) high mechanical properties, (iv) low imidization temperature and (v) extended pot-life. The PSPI exhibited an excellent sensitivity of 40 mJ cm\(^{-2}\) and a high contrast of 4.2. A clear positive-imaged pattern with 3-μm resolution was obtained in 4-μm thick PSPI film on a glass plate after development with a 2.38 wt% TMAH\(_{aq}\). The PSPI film cured at 250 °C for 1 h had high mechanical properties. This new formulation method improves the resolution, mechanical properties, processing temperature, and a pot-life of PSPI compared with the former method. Furthermore, TBG is very effective for removing the possibility corrosion of Cu circuits. Therefore, the advancing PSPI can be one of the prospects of the next generation electronic packaging materials.

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