Journal of Applied Polymer Science

Preparation and characterization of printable solder resist inks based on hyperbranched polyester

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ABSTRACT: Two novel solder resist inks containing hyperbranched epoxy resin (HBPE) for thermal curing and hyperbranched epoxy acrylate resin (HBPEA) for UV-curing were introduced in this work. Different generations of HBPE and HBPEA were synthesized and their chemical structures were determined by FT-IR. Both curing reactions were monitored under differential scanning calorimetry (DSC) and photo-DSC. For HBPE, the curing temperature of 7th generation was only 91°C and for HBPEA, the curing duration of 7th generation was under 10 s. The thermal stabilities of cured resins were much more stable than linear resin, as the decomposition temperatures of HBPE and HBPEA were both over 400°C. The ink containing HBPE or HBPEA jetted by piezoelectric printer showed excellent accuracy and consistency of linewidth and the morphologies of cured pattern were observed through a stereo microscope. Other performances of solder masks were tested under China Printed Circuit Association (CPCA) standard (CPCA/JPCA 4306-2011), which satisfy all requirements of printed circuit board soldering procedure. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 41805.

KEYWORDS: dendrimers; differential scanning calorimetry (DSC); hyperbranched polymers and macrocycles; morphology; polyesters; resins

Received 26 August 2014; accepted 22 November 2014
DOI: 10.1002/app.41805

INTRODUCTION

Printed electronics have enjoyed a booming research period as numerous scientists and institutes devoted to optimizing conductive inks and improving methods to prepare high reliability conductive pattern. However, the smoothness and evenness of solder mask still could not improve in recent years, which became limitations of image processing of printed circuit board (PCB).[1]

Recent studies on solder mask mainly focused on inks for high-resolution screen printing and ink-jet printing. Screen printing still is one of the most universal technologies for PCB manufacturing. Because this technique requires photo mask, UV exposure, and clearance to generate pattern,[2] which has much more complex procedures and causes waste of materials as well as pollutions, ink-jet printing was invented to solve those problems.[3] Compared to screen printing method, solder mask prepared by ink-jet printing has unique advantages, for instance, (1) materials saving, solder mask only covered required area; (2) selective printing, clearance for uncured area is not required and no solder resist ink filled in the blind or buried vias and holes, which could not fully be removed during clearance by traditional method thus causing PCB failure; (3) one step process, solder resist ink directly printed on the substrate instead of complicated screen printing procedure, which will reduce procedure duration and also human cost; (4) high-resolution, 60 μm linewidth of solder mask will be reported in this article while traditional method has only reached 100 μm; and (5) high registration accuracy, each panel could go through individualized registration process and facilitating accurate solder mask deposition which satisfied the high-density SMT requirements while the insufficient registration accuracy between film and PCB by screen printing limited its application in high-density SMT.[4,5] Figure 1 presents the advantages of ink-jet printing compared to screen printing.

To satisfy the requirements of ink-jet printer, scientists started to modify the traditional resin to lower its viscosity, which ensure the ink could be jetted continuously. However, with the deepening of research, some issues emerged, for instance, linear polymer was observed degradation during ink-jet printing,[6] the stabilized linewidth of printed solder resist ink could not control well especially during the beginning part,[7] the ink jetted on the substrate would spread before curing, thus decreasing the accuracy.[8,9] Those problems were mainly because traditional

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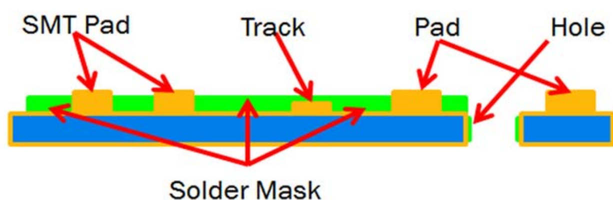
Screen Printing



Ink-jet Printing

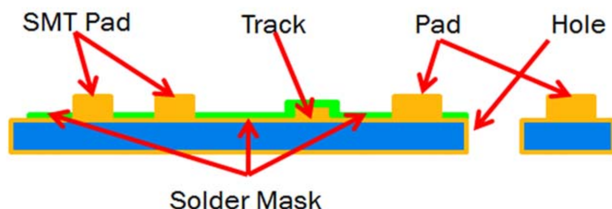


Figure 1. Comparison between solder masks prepared by screen printing and ink-jet printing. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

solder resist ink consists of linear polymers, which has shear-thinning nature and thixotropy. The viscosities of these inks were strongly affected by flow speed of polymer, for instance, if the ink flows fast, the viscosity is low, but if the ink flows slow, the viscosity increases. Thus makes the first few drops of ink extremely difficult to jet due to its high viscosity before ink started to flow. So the accuracy of solder mask prepared by ink-jet printing were strongly affected by the timing of printing, at the initial stages the cured mask showed extremely poor accuracy both linewidth and facilitating accuracy but much improved latter. And also the curing speed of ink containing thermal setting resin was not fast enough, which could spread on the substrate before curing.

To maintain stability of solder mask prepared by ink-jet technology, the non-Newton ink should be modified to Newton fluid theoretically, which has no shear-thinning nature and thixotropy. However, if the ink shows complete Newton fluid property, the droplet jetted on the substrate will easily spread, causing inaccuracy of solder mask pattern.[10] So the ideal ink should exhibit fluid property between Newton fluid and non-Newton fluid. Unlike linear polymer and highly symmetrical dendrimer, hyperbranched polymer shows fluid property just between Newton fluid and non-Newton fluid because of its unique molecule structure. Because hyperbranched polymer has no molecular chain entanglement, low viscosity, high solubility, numerous functional group at the terminal units but relative low steric hindrance, which means hyperbranched polymer could be more active during curing reaction and also easily synthesized, it could be a perfect replacement for traditional linear epoxy resin and epoxy acrylate resin to prepare solder resist inks.[11–13]

In this article, hyperbranched polyester epoxy resin (HBPE) and hyperbranched polyester epoxy acrylate resin (HBPEA) were synthesized and FT-IR was employed to ensure the chemical structure. Both photoinitiator and thermal curing agent were mixed with HBPE and HBPEA, respectively, to form solder resist inks and various active dilutes were used to adjust viscosity. The ink jetted by a piezoelectric jet printer on different substrate including PET, PI and glass, cured by heat and UV light source to form solder mask, respectively. The curing process was monitored under differential scanning calorimetry (DSC). Thermal stability of solder mask was measured by thermal gravity analysis (TGA). The morphologies of cured pattern were observed through a stereo microscope. Other mechanical properties and chemical resistance were tested under China Printed Circuit Association CPCA/JPCA 4306-2011 standard.

EXPEREMENTAL

Materials

1,1,1-Tris(hydroxymethyl)propane (TMP), dimethylolpropionic acid (DMPA), *p*-toluene, sulfonic acid (TSA), and ethylene glycol diglycidyl ether, which were all analytically pure, were purchased from Aladdin Industrial Corporation, Shanghai, China. Acrylic acid (AA), 4-hydroxybenzenecarboxylic acid (PHBA), epichlorohydrin (ECH), 1,4-benzenediol, and thermal curing agent triethylene tetramine (TETA), which were also analytically pure, were obtained from Sinopharm Chemical Reagent Beijing. UV curing agent 2-hydroxy-2-methyl-1-phenyl-propan-1-one (1173) was kindly provided by Nanjing Wali Chemical Technology. Active dilutes such as ethoxylated trimethylolpropane triacrylate (EO-TMPTA) and diethylene glycol diacrylate (DEGDA) were provided by Guangzhou Deco Composite Technology.

Synthesis

Synthesis schemes of HBPE and HBPEA are shown in Figure 2. TMP, DMPA, and TSA were mixed in a three neck reaction flask equipped with a mechanical stirrer, an argon inlet, and outlet. The flask was immersed into an oil bath heated to 140°C with argon gas stream to remove water for 30 min。 The argon flow was set to approximately 1 bubble per second and the rotate speed of mechanical stirrer was kept under 150 r/min. Then sealed the flask and shut off the argon, vacuumized the flask by a water pump. Increased the speed of stirrer to 400–500 r/min for 2 h and first generation of hyperbranched polyester (HBP-G1) was synthesized. Then added certain amount of DMPA and TSA listed in Table I to prepare next generation of hyperbranched polyester. With above method, HBP-G1~HBP-G7 were synthesized in our lab.

Hydroxy-terminated HBP were then mixed with PHBA and catalyst TSA (0.5wt %PHBA) in a three neck flask also equipped with a mechanical stirrer, an argon inlet and outlet. The flask were then immersed into an oil bath at 180°C. With argon gas stream to remove water, the reaction duration was 5h to gain phenolic hydroxy-terminated HBP. Reduced the temperature of oil bath to 80°C and then excessive ECH was added into the flask at one time while the 30wt % sodium hydroxide aqueous solution were added dropwise. After 3h reaction, the resin was extracted from the mixture by toluene. The resin was washed by distilled water three times and pure hyperbranched polyester epoxy resin (HBPE) was obtained.

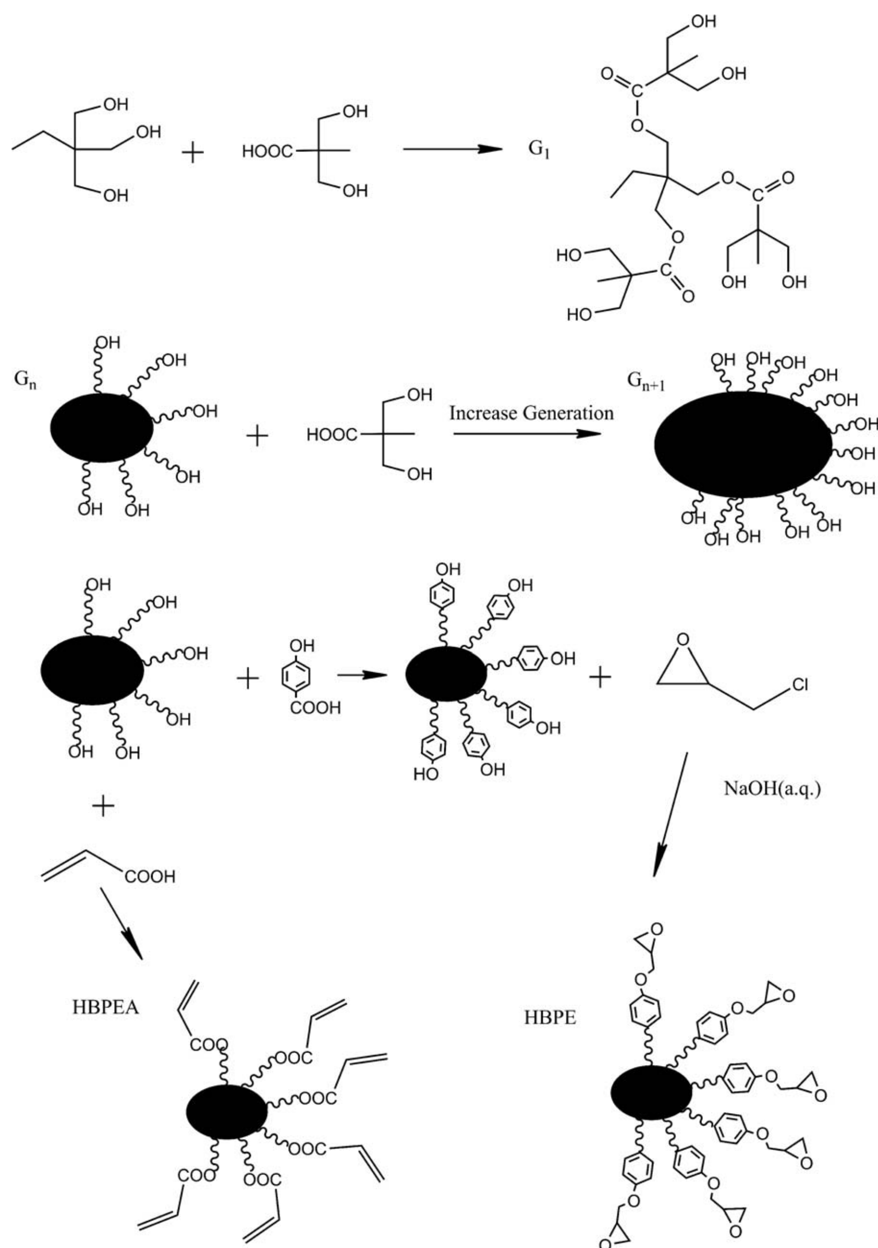


Figure 2. Synthesis schemes of HBPE and HBPEA.

Meanwhile, hydroxy-terminated HBP were then mixed with acrylic acid (AA) and 1,4-benzenediol in a four neck flask with mechanical stirrer, a spherical condenser and a thermometer. The 1,4-benzenediol was added to avoid oxidization. The flask was put into an oil bath at the temperature of 80°C for 1.5 h. The mixture was deposed into a large beaker with distilled water. After strong stirring, let the mixture stand over 3 h, remove the water to gain the hyperbranched polyester epoxy acrylate resin. The resin should be purified by washed with distilled water for at least three times.

CHARACTERIZATION AND MEASUREMENT

Table I shows the theoretical value of OH of hyperbranched molecule, which increases as the generation of hyperbranched molecule grows. But the ultimate value is close to 1.72, thus nearly no significantly increase after 7th generation of hydroxy-terminated HBP.[14] Considering the difficulties of synthesis and no distinct improved content of functional group, 2nd generation (G2), 3rd generation (G3), 5th generation (G5) and 7th generation (G7) of HBP were selected to study their synthetic reactions, curing conditions and thermal properties. The resins were formulated with active dilutes and other solvents to provide inks. The inks were jetted by Epson R230 piezoelectric printer. The patterns were cured by vacuum oven as well as UV light source to form solder mask. The performances of solder masks were tested under CPCA standard (CPCA/JPCA 4306-2011).

The synthesis of HBPE and HBPEA were monitored under a Nicolet Nexus 470 FT-IR spectrometer. Because HBPE has phenyl structure which could improve heat resistance of resin but

Table I. The Amount of DMPA and TMP to Synthesize Different Generation of Hyperbranched Polyester

Core (TMP,1 mol)			
Generation	DMPA (mol)	Hydroxy-terminated group (mol)	Theoretical value of OH (mol/100 g)
HBP-G1	3	6	1.24
HBP-G2	6	12	1.44
HBP-G3	12	24	1.57
HBP-G4	24	48	1.64
HBP-G5	48	96	1.68
HBP-G6	96	192	1.70
HBP-G7	192	384	1.71

also can be easily decomposed under UV light.[15] Thus, HBPE was cured by heat while HBPEA was cured under UV light source. The formulations of inks prepared with HBPE and HBPEA, respectively, were listed in Tables II and III.

Ink-1 was cured by heat and its curing reaction was analysed by Q2000 DSC made by TA instrument with the heating rate 15°C/min, while ink-2 was harden under UV light-source, whose curing reaction was studied using Photo-DSC 204 F1 made by NETZSCH-Gerätebau GmbH with the illuminance of the UV optical source was 1 w/cm^2 and the wavelength was 350 nm.[16] The inks were jetted by Epson R230 piezoelectric printer and all patterns were printed onto PET films. The parameter of printer was listed in Supporting Information Table S1. For ink-1, a preheated platform was utilized to reduce curing duration and for ink-2, a UV light source was lighted as the ink jetted on the substrate. All these procedures were taken to prevent ink spreading. The cured pattern was observed a stereo microscope KH-7700 made by HIROX. Thermal stability of solder mask was studied by a Q500 TGA, which was also made by TA instrument. 5–10 mg cured solder resist was put into an alumina crucible under nitrogen atmosphere. The heating rate was 15°C/min from 50°C to 650°C. The assessment of thermostability was based on CPCA / JPCA 4306-2011standard, which will be detailed in the following part.

Other performances of solder masks were tested under CPCA standard (CPCA/JPCA 4306-2011). Pencil Hardness was tested by QHQ-A pencil hardness tester made by Shanghai Shengpu Scientific Instruments with exerts a force of 7.5 N. For rigid printed board, the pencil hardness should be over 4H but for flexible printed board, only 2H would meet the standard. The test conditions of chemical resistance property were listed in Table IV. The cured board with solder mask was immersed in various liquid, and then cleared with distilled water. The board was dried under room temperature and should exhibit no swelling, no blistering, nor sticky. Besides, the board should bear at least three time soldering condition with the temperature 280–288°C and duration 10 s to meet lead free soldering resistance requirements. The insulation resistance was tested by an insulation tester provided by Fluke China.

Table II. Solder Resist Ink Containing HBPE (Ink-1)

Content	Weight ratio (wt %)
HBPE	60
TETA	5
ethylene glycol diglycidyl ether	35

Table III. Solder Resist Ink Containing HBPEA (Ink-2)

Content	Weight ratio (wt %)
HBPEA	60
1173	5
EO-TMPTA	15
DEGDA	25

RESULTS AND DISCUSSION

FT-IR Spectrums of HBPE and HBPEA

Seventh generation of HBP was selected as example to demonstrate the process of synthesis because no significant differences between each generation of same reaction product.[17] Other results such as HBP-G2 and its resins were shown in Support Information Figure S1. Figure 3(A–C) show the preparation of HBPE while (A) and (D) demonstrate the synthesis of HBPEA. As Figure 3(A) shows the FTIR of HBP-G7, the 1737 cm^{-1} represented $\nu_{C=O}$ while 1230 and 1126 cm^{-1} indicates ν_{O-C-O}, which proves the reaction between carboxyl and aliphatic hydroxy. Absorption peaks at 1609, 1592, 1515, and 1469 cm^{-1} of Figure 3(B) demonstrate phenyl is grafted onto hydroxy-terminated HBP. Figure 3(C) presents a remarkable increase of absorption peak at 1037 cm^{-1} (ether group ν_{C-O}, etherification reaction of phenol hydroxy group and epichlorohydrin) and also a weak peak at 910 cm^{-1} (epoxy group), which indicates the epoxy group terminated HBPE are successfully synthesized. Figure 3(D) illustrates the structure of G7-HBPEA, as absorption peak at 3080 cm^{-1} shows $\nu_{C=C-H}$ and 810 cm^{-1} corresponds to $\delta_{C=C-H}$, which proved the epoxy group was modified to C=C. All the results confirmed synthetic procedure of HBPE and HBPEA in Figure 2.

Curing

Thermal Curing of HBPE. Theoretically, it could be expected that the heat of cure in HBP system is higher than that in linear polymer system because of the number of active group increase. However, the activation energy for cure reaction is lower in HBP

Table IV. Test Conditions of Chemical Resistance

Chemicals	Temperture (°C)	Immersion time (min)
Isopropanol (C.P.)	25 ± 2	30
10% Sulfuric acid aqueous solution (by volume)	25 ± 2	10
10% NaOH aqueous solution (by weight)	25 ± 2	30

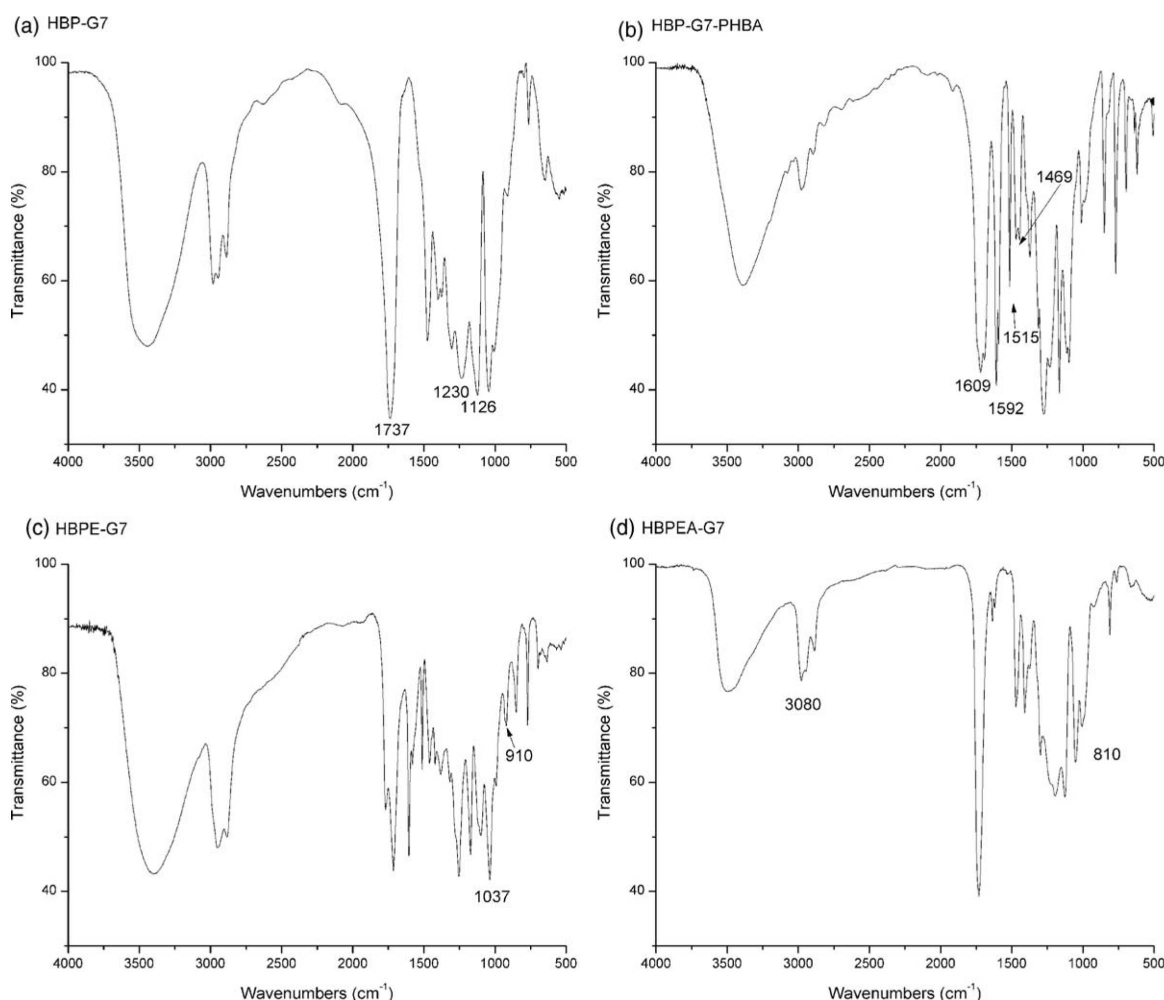


Figure 3. FT-IR spectrums of (a) HBP-G7, (b) HBP-G7-PHBA, (c) HBPE-G7, and (d) HBPEA-G7.

system.[18] Thus, in contrast of initial curing temperature, HBPE is much lower than universal epoxy resin E51 with the same amount of curing agent (5 wt % TETA). With the generation of HBPE increasing, the curing temperature decrease from 112°C to 91°C, nearly 20°C as Figure 4 shows. All the curing reactions were monitored by DSC and the degrees of each sample curing were over 90% (The degree of curing was discussed in Supporting Information as G7-HBPE was demonstrated). The reason why curing temperature of HBP is lower than linear polymer is relative high concentration of active end group. Because the low curing temperature, solder resist ink based on hyperbranched polyester could now be used on low deflection temperature flexible substrate and widen the application in flexible electronics.

UV Curing of HBPEA. The UV curing process was studied by photo-DSC, as Figure 5 illustrated. The durations of curing process are all less than 5 s regarding different generation of HBPEA. The curing speed could be effected by concentration of active group (C=C) or concentration of free radicals generated by photoinitiator under UV light.[16,19,20] Since G2-HBPEA to G7-HBPEA have quite different concentrations of active group (C=C), the domination of curing may be the concentration of free radicals. According to previous research, 1173 photoinitiator breaks into radicals about 11 ms after it absorb UV photon.[21] Since each sample contains exactly the same amount of 1173, the rate limiting factor can be easily the explained by the concentration of 1173. Compared to other research of UV curing, the reaction durations are almost the same.

Regarding those two curing methods, the branching of the resin does decrease the curing temperature and this implies faster curing speed at the same temperature. For UV curing, since the limiting step is not the concentration of active group, the curing speed has no difference between branched resin and linear resin. However, the curing durations were all under 10s, which was already fast enough to satisfy the requirement of manufacturing process.

Morphologies of Solder Masks

Figure 6 shows the morphologies of solder masks printed by Epson R230 piezoelectric printer. To demonstrate the advantages of hyperbranched polymer, patterns in Figure 6(A,B) were printed with traditional linear solder resist ink containing E51 epoxy resin while patterns in Figure 6(C,D) are generated by

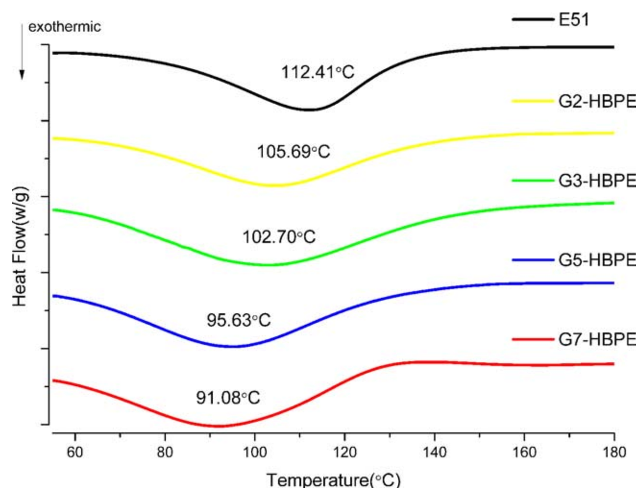


Figure 4. DSC results of E51 and various HBPEs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

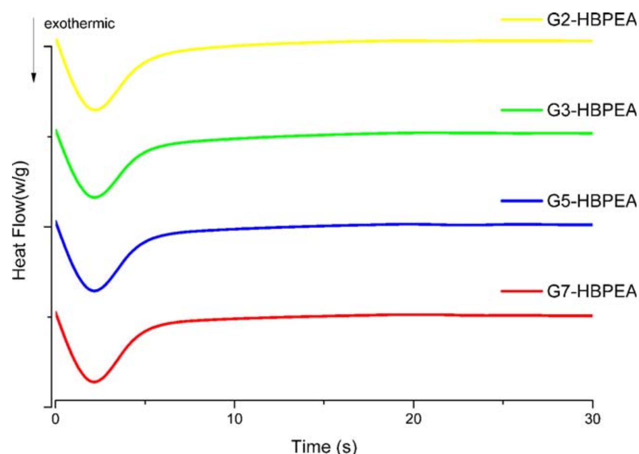


Figure 5. Photo-DSC results of various HBPEAs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

G7-HBPE. All the patterns were printed onto PET films which were placed on a preheated platform. Other parameters of printing were kept exactly the same and the ink could be continuously jetted through a 50 μm nozzle. The linewidth of the traditional solder resist ink is over 100 μm while that of hyperbranched polymer is just about 60 μm as Figure 6(C,D) show. Because hyperbranched polymer reacted faster than linear polymer, the G7-HBPE ink jetted on the substrate could not spread before being cured. However, traditional solder resist ink may spread on the substrate because of its slower curing speed, which causes the relative wider linewidth under same condition.[22] And in Figure 6(A), the initial part of the pattern shows an acute triangular shape because of linear polymer's shear-thinning nature. That is in the very beginning, the viscosity of ink is relative high, and with the flowing of ink, the viscosity decreases, which strongly reduces the accuracy of the start part. Compared with Figure 6(C), the pattern printed with hyperbranched ink shows excellent accuracy and consistency of linewidth.

Thermostability of Solder Masks

Thermostability of solder masks was tested by thermal gravity analysis and the results were shown in Figure 7. From Hult's research, the thermal stability of hyperbranched polymers is related to the chemical structure in the same manner as for linear polymers.[23] However, as Figure 7(B) shows, compared with

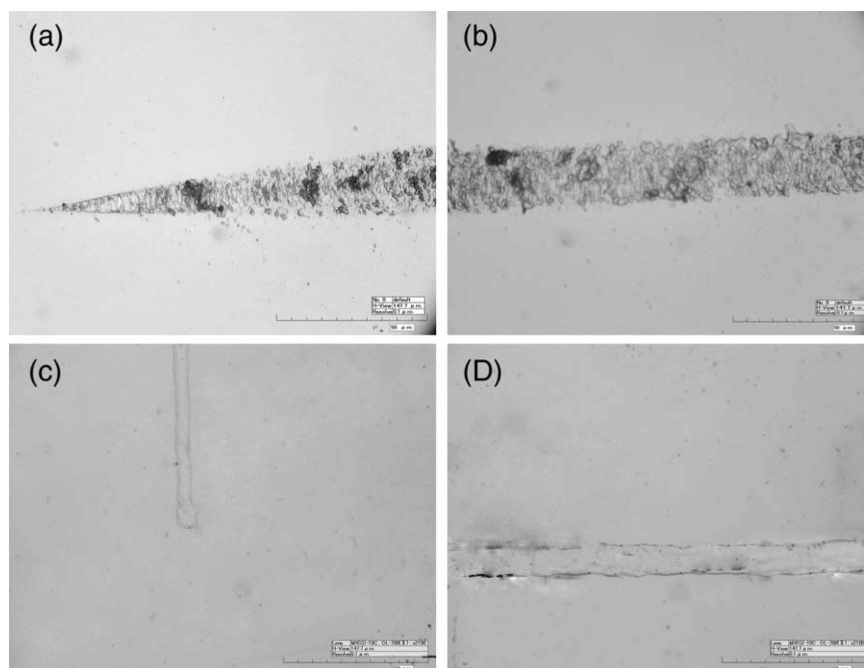


Figure 6. Morphologies of solder masks: (a) initial printing pattern of linear polymer solder resist ink, (b) pattern printed with linear polymer, (c) initial printing pattern of hyperbranched polymer solder resist ink, and (d) pattern printed with hyperbranched polymer.

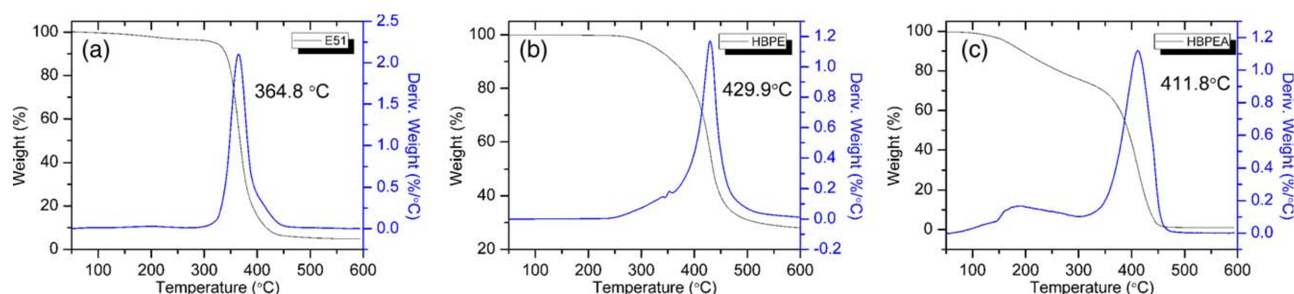


Figure 7. TGA of (a) E51, (b) HBPE, and (c) HBPEA. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

traditional E51 epoxy resin, HBPE has more stable thermal properties due to its higher content of phenyl structure. The maximum decomposing temperature of HBPE is nearly 430°C, about 50°C higher than E51, which could easily satisfy the requirements of SMT and other PCB manufacture procedures. The structure of HBPEA contains no phenyl group because of its UV curing condition. The phenyl group could absorb UV radiation and may cause degradation of solder masks, which weaken the heat resistance property of solder masks. As Figure 7(C) shows, the thermal stability of the UV curing HBPEA decreased. However, it can still maintain mask structure at the temperature around 350°C, which also meets the PCB soldering conditions.

Performances of Solder Mask

The solder masks containing HBPE and HBPEA were also tested under CPCA/JPCA 4306-2011 standard, and the results were listed in Table V. All generations of HBPE except G2 show excellent test results, which meet all the requirements of the standard. However, for HBPEA, G2 and G3 show relatively poor chemical resistance than traditional solder resist ink. This may cause by some uncured C=C, which could be reacted in some chemical atmosphere. But with the generation of HBPEA grows, the end unit could be cured more easily and the whole structure of curd mask could be more stable, which improves the stability of mask. And also the insulation resistance of masks containing HBPE is much better than that of HBPEA. This could also prove the uncured C=C exists in HBPEA. As the generation grows, the uncured C=C decreases, which improves the insulation resistance property.

CONCLUSIONS

Hyperbranched polyester epoxy resin and hyperbranched polyester epoxy acrylate resin were synthesized while the reactions were monitored by FT-IR. The chemical structures of HBPE and HBPEA were also proved by FT-IR. The resins were mixed with chosen active dilutes and curing agents which ensured the inks could continuously be jetted by piezoelectric ink-jet printer. The printed pattern with HBPE and HBPEA could be cured by heat and UV radiation, respectively. The curing temperature of HBPE decreased dramatically with the generation increasing and G7-HBPE's curing temperature was 91°C, almost 20°C lower than traditional solder resist ink. The curing duration of HBPEA was less than 10 s, which was already fast enough to satisfy the requirement of manufacturing process. The pattern printed with solder resist inks containing hyperbranched polymers shows excellent accuracy and consistency of linewidth than traditional inks with linear polymers because of hyperbranched polymer's no shear-thinning nature. And also the novel inks could print better linewidth because the curing reactions of HBPE could be so fast that the jetted inks were already cured before spread. In fact, under same printing condition, the linewidth of hyperbranched polymers was almost half of which printed with traditional inks. The masks generated by HBPE showed good heat resistance property as the content of thermal stable group (i.e. phenyl group) was much higher than linear polymer, while masks produced by HBPEA exhibited relatively weaker thermal stability but also could satisfy the requirements of PCB soldering conditions. Both masks could easily maintain their structure at the temperature of 350°C. From the test under

Table V. The Performance of the Solder Masks

Sample	Pencil hardness	Chemical resistance	Lead free soldering resistance	Insulation resistance (MΩ)
HBPE-G2	3H	Qualified	Disqualified	310
HBPE-G3	3H	Qualified	Qualified	349
HBPE-G5	3H	Qualified	Qualified	370
HBPE-G7	4H	Qualified	Qualified	380
HBPEA-G2	2H	Disqualified	Disqualified	107
HBPEA-G3	2H	Disqualified	Disqualified	120
HBPEA-G5	3H	Qualified	Qualified	150
HBPEA-G7	3H	Qualified	Qualified	173

CPCA/JPCA 4306-2011 standard, the high generation of HBPE and HBPEA show better performances in hardness, chemical resistance, soldering resistance and insulation resistance. It is highly recommend using highly branched polymer to replaced current linear polymer in solder resist ink.

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