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Comparison of Thermal Properties of PCB Photoresist Films Cured by Different Techniques

Piyachat Wattanachai [*,1]
Christian Antonio [2]
Susan Roces [3]
[1] *Department of Chemical Engineering, Faculty of Engineering, Burapha University, Sean Sook, Muang, Chonburi 20131, Thailand*
[2] *Julius Kruttschnitt Mineral Research Centre, Sustainable Minerals Institute, University of Queensland, Indooroopilly, Brisbane, QLD 4068, Australia*
[3] *Department of Chemical Engineering, De La Salle University, Manila 0922, Philippines*
[*] *e-mail:* piyachat.a@eng.buu.ac.th

The possibility of implementing microwave technology to photoresist film curing which is a major process in the production of electronic printed circuit boards (PCB) was investigated and compared with a conventional curing method, *e.g.* UV lithography. Since both techniques involved irradiation, hot plate curing which relies on thermal conduction was undertaken to study the effect of a heat transfer approach. Two film thicknesses were studied, *i.e.* 0.0012 and 0.002 inch, and the effects of curing power and time were investigated. Thermal properties, *i.e.* percent cure, glass transition temperature (T_g), composition and degradation temperature (T_d), were evaluated using a Differential Scanning Calorimeter (DSC) and Thermogravimetric Analysis (TGA) and it was found that the commercial UV irradiation was sufficient to completely cure the thin film but only reached 76% cure for the thicker film, resulting in a lower T_g. The results show that the required processing conditions using a conventional household microwave to obtain almost complete curing were 1,000 Watts and 10 minutes curing time. In addition, improved curing was achieved in the thicker film because microwave can transmit into polar materials whereas UV cannot penetrate very far into the material. The hot plate curing was observed to produce a higher degree of curing and T_g, however, the uniformity of heating was found to be a major limiting factor of this technique. Slight differences in decomposition profiles of the films cured by different techniques implied slight differences in molecular structures. Compared to UV and hot plate curing, microwave technology was demonstrated as a potential curing technique in the production of PCBs due to its ability to efficiently cure thick films resulting in a strong material with high T_g. To apply the technique to other processes, optimal conditions, *i.e.* power and time, should be further investigated as well as the prevention of hot spots.

Keywords : Microwave curing, UV curing, Photoresists, Glass transition temperature, Percent cure, PCB

INTRODUCTION

Microwave (MW) technology has gained significant attention from both research and industrial point of view. It has been adapted to a large number of applications (Leonelli and Mason 2010) including material drying (Hansson and

Antti 2003, Pereira et al. 2007), epoxy resin curing (Boey and Yap 2001, Yarlagadda and Hsu 2004, Kwank et al. 2015), dental acrylic resin polymerisation (Azzarri et al. 2004, Lai et al. 2004), organic synthesis (Wiesbrock et al. 2004), waste digestion (Nguyen et al. 2012, Nguyen et al. 2007) as well as curing of construction materials (Rattanadecho et al. 2008, Chowasakoo and Sombatsompop 2007). This is due to the fact that MW is a form of high frequency energy that is transferred to materials through molecular interaction with the electromagnetic field. Hence, it provides a rapid, volumetric and selective heating, unlike conventional heating that relies on heat conduction and convection through the surface of materials. Significant drying time reduction has been observed for MW drying compared to conventional thermal heating without affecting product qualities (Leonelli and Mason 2010, Hansson and Antti 2003, Pereira et al. 2007, Rattanadecho et al. 2008, Chowasakoo and Sombatsompop 2007). While some studies reported a decrease in wood strength obtained from MW drying compared to oven drying, Hansson and Antti (2003) pointed out that the differences might arise from natural variation in biological substances. However, with a very fast heating rate, runaway reaction must be considered (Pereira et al. 2007, Wiesbrock et al. 2004).

One MW application that has been particularly gaining attention is microwave-assisted polymerization and thermoset curing in Microelectromechanical Systems (MEMS) and Printed Circuit Board (PCB) manufacture where it could have substantial benefits. One crucial process in producing PCB is the curing of the photoresists where UV lithography is commonly used; however, poor penetration ability is a drawback for UV light. With the powerful penetration ability of MW, uniform temperature distribution throughout carbon fiber reinforced plastics (CFRPs) of different thicknesses has been previously achieved (Kwank et al. 2015, Li et al. 2014). As high as a 50% reduction in curing time for thermoset phenolic resin was achieved using MW curing compared to thermal curing and nearly four times increase in shear strength of the glue was observed (Zheng et al. 2014). Similar results of short curing time and enhanced mechanical strength using MW curing were reported with epoxy resins and polymer composites (Yarlagadda and Hsu 2004, Kwank et al. 2015, Li et al. 2014). Despite the benefits of MW curing, it has also been reported to cause thermal run away reactions resulting material degradation and lowering of mechanical properties (Zheng et al. 2014, Yarlagadda and Cheok 1999).

The improvement in mechanical properties of epoxy cured by MW technique was due to the crosslink density of the polymer. With well dispersed carbon nanotubes, MW curing resulted in a composite with better characteristics compared to that obtained by thermal curing due to the fact that carbon and graphite are excellent MW susceptors, thus are able to absorb electromagnetic energy and converts this into heat resulting in higher conversion of the cure (Sung et al. 2014, Rangari et al. 2011). As the percent cure increased, the glass

transition temperature, which represents the temperature that polymer starts to mobilize or change from a rigid and brittle state to become rubber-like state, increased along with an improvement in adhesive bonding strength as well as compressive and bending strength (Zhou et al. 2003). Boey and Yap (2001) reported that the maximum percent cure and T_g were strongly dependent on the curing agents; nevertheless, an increase in MW power resulted in an increase in percent cure as well as T_g. The high heating rate of MW caused a delay in the reactivity of amine function and hence inhibited the polymerization in epoxy resin. Similarly, even though hardener/epoxy resin compositions were found to be a crucial parameter in controlling polymerization, MW curing yielded a greater crosslink density compared to oven curing (Yarlagadda and Hsu 2004). Not only the improvement of photoresist films in terms of higher percent cure and Tg was achieved by MW curing, satisfactory microstructures at lower curing temperatures were also obtained as compared to the other conventional curing methods (Antonio and Wattanachai, 2014 and Wattanachai and Antonio, 2016). While it is suggested that a combination of MW power and curing time should be carefully considered in curing acrylic resins (Lai et al. 2004), it was concluded that exposure time was most relevant factor (Azzarri et al. 2004). Whilst the use of MW irradiation is largely beneficial to curing, MW technology has also been successfully used in degradation or digestion of CFRP wastes by weakening the bonding between cured epoxy resin and carbon fibers as revealed in (Nguyen et al. 2012, Nguyen et al. 2007).

The present study reported the feasibility of extending MW technology to photoresist curing as it has not been well established in the PCB industry. A discussion on thermal properties, i.e. percent cure and glass transition temperature, of the MW cured films was presented in comparison to films cured by commercial UV lithography. Thermal curing was also carried out as it is the conventional curing method for many thermosets.

EXPERIMENTAL

Material and Apparatus

Etertec negative photoresist films with the thickness of 0.0012 and 0.002 inches were studied in this research. The film compositions are 20% acrylic ester, 50% acrylic polymer, 23% urethane acrylic, 5% solvent, and 2% others. Co light UV-Exposure Model 1230 operated at 3 kW with UV wavelength of 320-380 nm was used for the UV curing technique whilst a Grand Fise household microwave oven Model WD900ASL23 operating at 2.45 GHz with a maximum power of 1000 W was used for the MW cure. Hotplate (HP) curing was carried out using a LMS hot plate Model HTS-1003. The degree of polymerization and the glass transition temperature (T_g) of the photoresist films were characterized using a Mettler Toledo Differential Scanning Calorimetry (DSC) Model DSC 1 whereas the degradation temperature (T_d) was determined by a Mettler Toledo Thermogravimetric Analyzer (TGA) Model 850.

Photoresist Curing

Three photoresist film curing techniques were investigated, i.e., UV exposure, microwave (MW) curing, and conventional hot plate curing. UV curing was performed at a PCB manufacturing company whist the other two processes were carried out at Chemical Engineering Laboratory, Burapha University. The company's standard procedure of the photoresist film preparation starts by attaching the film onto a PCB board using a Morton hot roll laminator. The inlet and outlet temperatures of the hot roll were 120 and 55°C, respectively, and the speed of the rollers was 2 m/s which is equivalent to 4 s contact time with the films. The films were then exposed under UV radiation for another 8 s. As there are incompatibilities between the PCB substrate which is metallic and the domestic MW oven used, the photoresist film cured by MW were not hot rolled but placed directly on glass slides for curing and hence the MW curing times were considerably longer compared to that of UV exposure. Prior to performing the experiments, an appropriate film position in the MW cavity was determined by an infrared camera in order to minimize hot spots. The films on the glass substrates were placed 3 cm from the base of the MW oven. Two beakers containing water were placed on two edges of the films to fix the position of the films as well as to absorb any excess radiation. Two variables regarding MW curing were examined, i.e. curing time and MW power. With a fixed curing time of 2 min, the MW power was varied from 200, 500, and 1000 W to determine the effect of power. Whilst the effect of curing time was examined for a fixed MW power of 1000 W for different curing times of between 1 to 10 min. To prevent a direct contact of the film with the HP, aluminum foil was wrapped around the photoresist films prior to placing on a 150°C hotplate for 2, 5, 8, and 10 min. Similarly, two beakers containing water were placed on two edges of the films in order to fix the position of the films.

Thermal Properties Characterization

Degree of curing and T_g were determined using a DSC. Approximately 2-10 mg of the films were wrapped by an aluminium foil in order to prevent the curling and non-contact of the films with the crucible under heating conditions. The films were heated from -70 to 260°C with a heating rate of 20°C/min under a nitrogen blanket. The degree or percent cure was determined by Eq. (1).

$$\%\ \text{cure} = \frac{\Delta H_{100\%} - \Delta H_s}{\Delta H_{100\%}} \quad (1)$$

where $\Delta H_{100\%}$ is the heat of reaction of complete curing and ΔH_s is the heat of reaction of samples. For T_d determination, 5-10 mg of the sample were tested by TGA using an alumina crucible. The samples heated from 25-800°C with a heating rate of 10°C/min under a nitrogen blanket.

RESULTS AND DISCUSSION

Percent Cure

Figure 1 shows the DSC thermogram of the photoresist films of thickness 0.0012

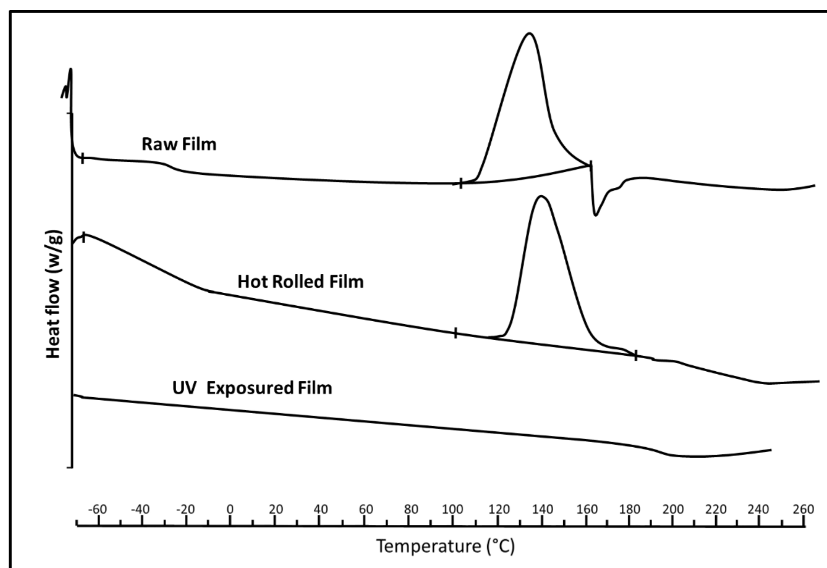

Fig. 1: DSC thermogram of 0.0012 in. films of raw, hot rolled and UV cured.

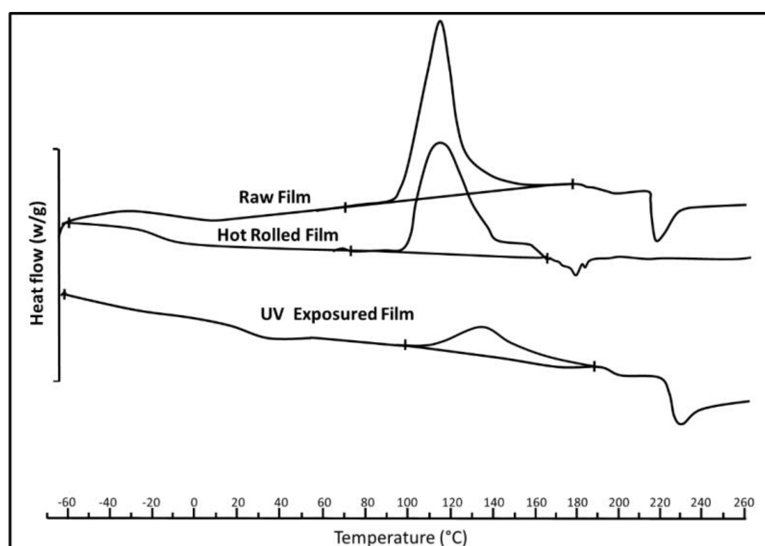

Fig. 2: DSC thermogram of 0.002 in. films of raw, hot rolled and UV cured.

in. at different states, i.e. raw film, hot rolled film, and UV cured film. It was found that the raw film started to polymerize at 123°C with a heat of reaction of 62.63 J/g whereas the hot rolled film polymerized at a slightly higher temperature of 124°C with a heat of reaction of 53.77 J/g. This is due to the fact that the film was slightly cured (14% calculated as in Eq. (1)) during the hot roll lamination process. As a result, $\Delta H_{100\%}$ was equal to 62.63 J/g for the 0.0012 in. film. It can be seen that after the 0.0012 in. film was exposed to UV radiation, it was completely cured and hence no heat of reaction was observed during the DSC experiment. On the other hand, the thick film of 0.002 in. was not completely cured with the UV curing as observed in figure 2, owing to the fact that UV cannot penetrate very far into the film. In addition, $\Delta H_{100\%}$ of the 0.002 in. film was

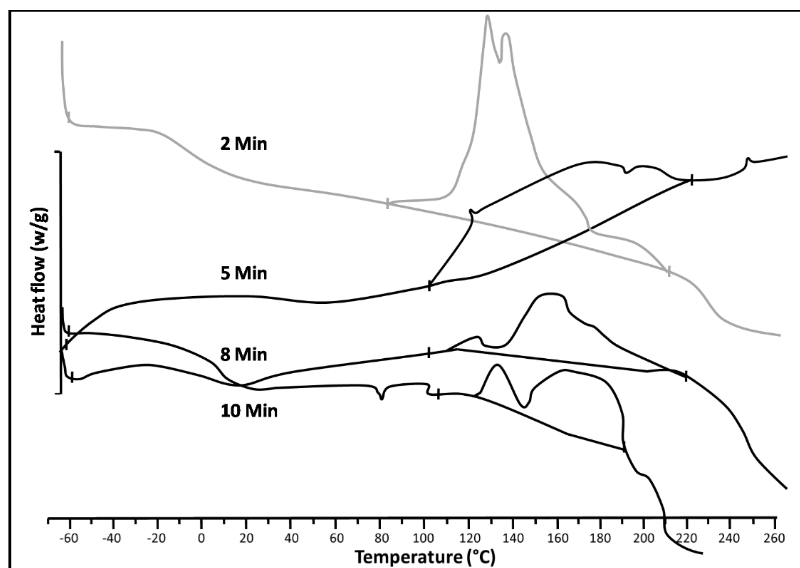

Fig. 3: DSC thermogram of 0.0012 in. HP cured (150°C) films at different curing time.

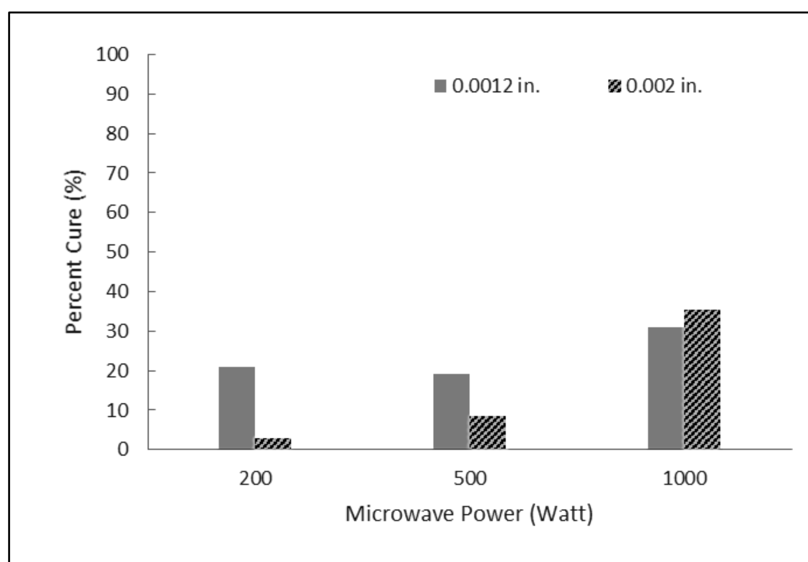

Fig. 4: Effects of MW power level on percent cure at a curing time of 2 minutes.

higher than that of the 0.0012 in. film due to the thicker film and was calculated as 87.25 J/g whereas the onset and peak polymerization temperatures were about 10°C lower. The hot roll lamination process also resulted in 14% curing of the film and the peak temperature was 1°C higher than that of the raw film while a percent cure of 76% and a 10°C increase in peak temperature were achieved after UV exposure. Similar DSC thermograms of the films cured by MW were observed; however, those of the films cured by HP were slightly different as illustrated in figure 3. The ranges of polymerization temperatures were considerably larger and there were two steps of polymerization shown as two peak temperatures, suggesting uneven film contact with the hotplate.

Figure 4 depicts the effects of curing power at a fixed curing time of 2 min. It

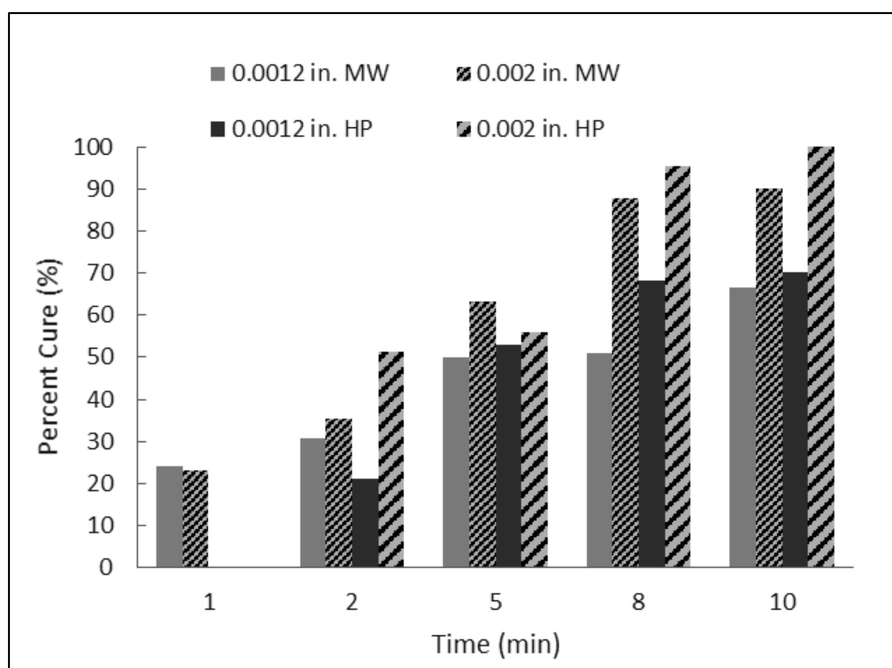

Fig. 5: Effects of curing time on percent cure of MW (1000 W) and HP (150°C) curing techniques.

was expected that the degree of curing would be higher in the thinner film due to the smaller volume of material and that curing will increase as MW power increases. The results indicates that this is the case and the improvement in the degree of polymerization with increasing MW power was observed, especially for the thick film (0.002 in.). It also shows that the degree of cure is not linearly related to the increase in power as found by other studies [20-23]. The greater change in percent cure observed in the thicker films suggest that it is more sensitive to changes in MW power. The MW power level of 1000 W was then chosen to study the effects of MW curing time on the films. Similar to UV curing, it was found that the onset temperature for polymerization of the thick film was about 10°C lower than that of the thin film at 200 and 500 W; however, at 1000 W only a slight difference of 1°C was observed for onset, peak, and endset temperatures of polymerization for the two thicknesses.

Figure 5 shows the effect of curing time for both MW and HP curing on the percent cure. MW curing was undertaken at 1000 W while HP curing was performed at 150°C. The results show that as the curing time increased the degree of polymerization for both film thickness improved for both curing methods. For MW curing, increasing curing time translated to better curing for the thicker films. At a curing time of 8 min, complete curing of the thicker film was close to being achieved, whereas the percent cure of the thin film at 10 min was still at 67%. It should be noted that when sufficient MW power (1000 W) and time (> 5 min) were used, the percent cure of the thick films were substantially greater than that of the thin films when in fact the film thickness should not be a factor in the MW curing mechanism. The MW

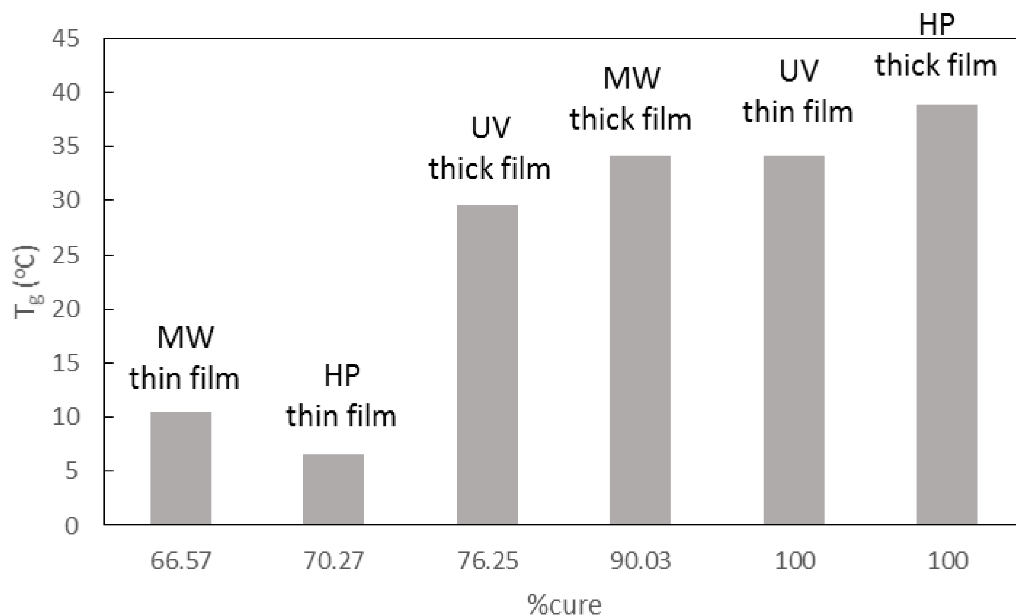

Fig. 6: Relationship between percent cure and Tg of 0.0012 and 0.002 in. films cured by UV, MW (1000 W, 10 min) and HP (150°C, 10 min) techniques.

frequency of 2.45 GHz should penetrate through the thickness of the films to provide volumetric heating. It is hypothesized that these results maybe because the thick films contain a larger amount of solvent which has a significantly higher dielectric constant than that of the polymer [1]. As a result, more energy were absorbed in the thick films, producing a higher degree of polymerization. In addition, as the percent cure increased, the onset, peak, and endset polymerization temperatures increased. The onset and peak temperatures of the 0.002 in. film cured at 10 min were as high as 162 and 177°C, respectively. As the cure progressed on the HP, it was observed that the film was contracting and losing good contact with the surface thus resulting in non-uniform curing as observed by color differences of the films after curing. The HP technique relies on traditional heat transfer for proper curing therefore proper contact with the heat source is required converse to the MW technique which heats volumetrically. Nevertheless, the HP technique yielded higher percent cure than those of MW cured films and this may be due to a non-optimized MW setup. It should be noted that a domestic MW, which are prone to non-uniform heating within the cavity, was used for the experiments thus with proper optimization the MW results could potentially be improved significantly.

Glass Transition Temperature (T_g)

Figure 6 illustrates the relationship between percent cure and T_g obtained from both film thicknesses cured by three different curing techniques, i.e. UV, MW (1000 W and 10 min), and HP (150°C and 10 min). For the thin film, UV curing resulted in a complete curing (100% cure) and highest T_g of around 34°C while a T_g

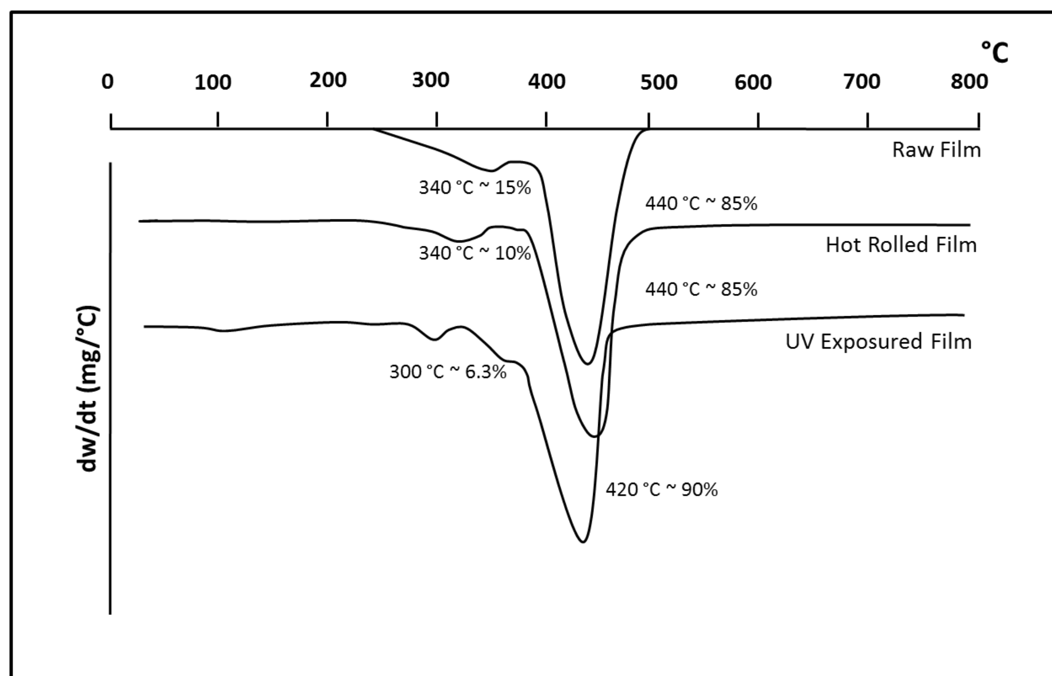

Fig. 7: DTG of 0.0012 in. films of raw, hot rolled and UV cured.

of approximately 10°C was achieved by the MW and HP curing with similar percent cure of approximately 70%. As one can expect, a higher degree of polymerization would result in a higher T_g due to stronger bonds. Since UV was not able to completely cure the thick film, the T_g was lower at 29°C for the 0.002 in. film. On the other hand, MW and HP curing techniques were efficient in curing thick film and hence T_g increased higher at 34 and 39°C, respectively.

Degradation Temperature (T_d)

Derivative Thermogravimetric Analysis (DTG) curves of 0.0012 in films were obtained from the TGA as shown in figures 7-9 for UV, MW, and HP curing, respectively. Figure 7 shows a slight weight loss at ~150°C in the raw film due to solvent evaporation. A slight degradation equivalent to 15% weight loss started around 250°C with a peak at 340°C followed by a second degradation step starting around 360°C and ended at 500°C. This second step was a major material decomposition in which the remaining content (85%) was degraded. With a low percent cure (14%) by hot roll process, changes in film molecular structure seemed insignificant and hence the DTG pattern appeared to be unchanged except the solvent evaporation step less noticeable. The photoresist cured by UV was completely cured and produced a different DTG pattern. The degradation of the UV cured photoresist was delayed to 280°C with a first peak at 300°C equivalent to 6.3% weight loss followed by a second peak at 420°C equivalent to 90% weight loss. The small degradation peak prior to the main degradation peak is believed to be a result of smaller molecular sizes. The shift in degradation onset is due to the fact that the material was more crosslinked, resulting in stronger

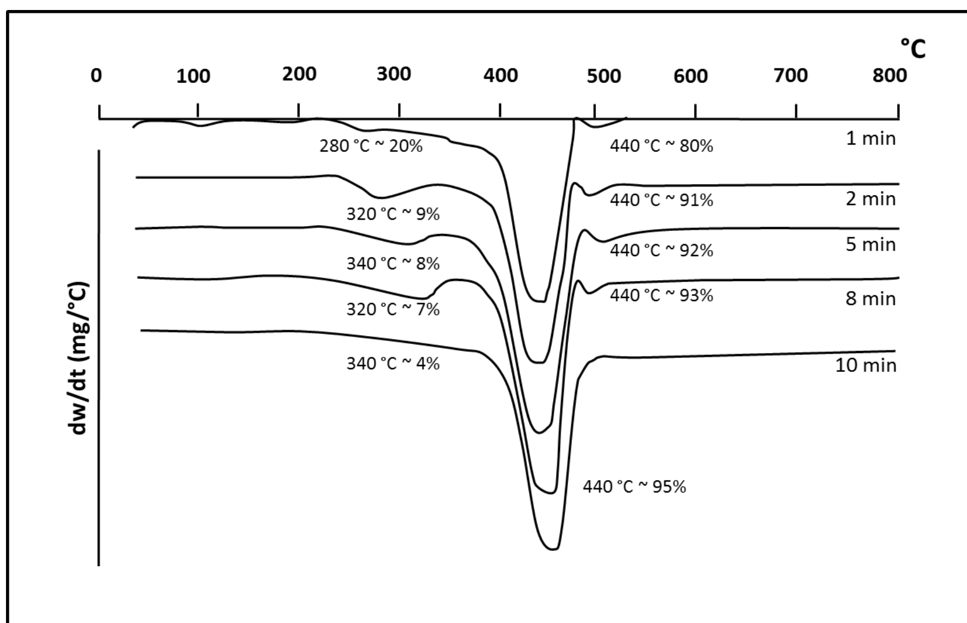

Fig. 8: DTG of 0.0012 in. MW cured (1000 W) films for different curing times

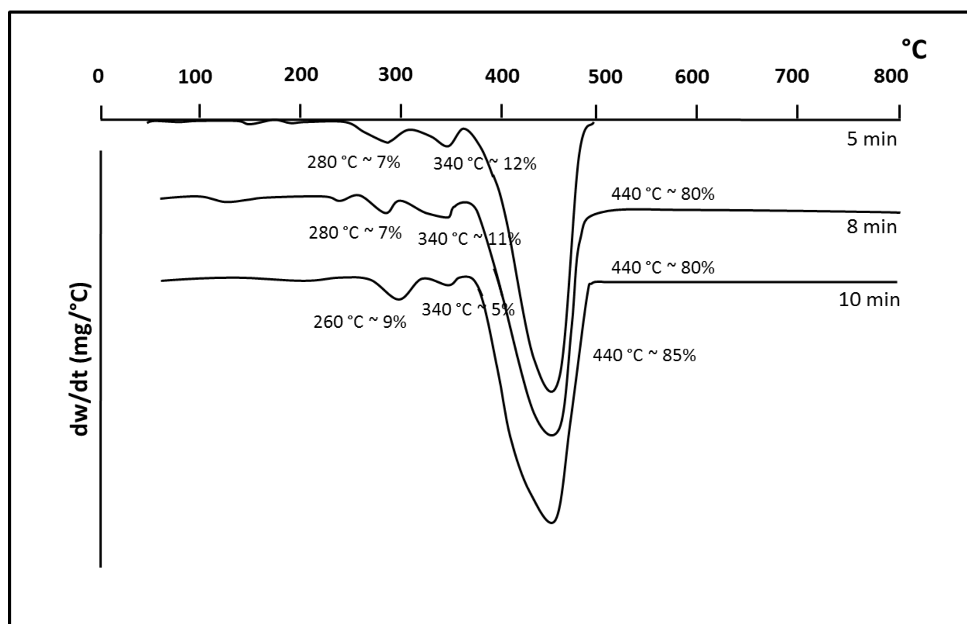

Fig. 9: DTG of 0.0012 in. HP cured (150°C) films for different curing times

molecular bonds. In addition, the range of decomposition temperatures was narrower as the decomposition endset was at 470°C, implying better molecular structure uniformity.

The effect of percent cure on molecular structure of the photoresist film is evident in the MW cured samples especially for 0.0012 in films as can be seen in figure 8. It was found that with low crosslink densities obtained from a short MW curing time, the DTG patterns were nearly identical to those of the raw and hot roll films except that there was an additional small peak at approximately 500°C. However, at a curing time of 10 min which

resulted in a percent cure of 67%, the small peaks at around 320°C and 500°C have disappeared and leaving only a peak at 440°C, suggesting a more homogeneous molecular structure. In addition, it should be mentioned that MW curing resulted in a higher degradation temperature of 440°C compared to 420°C from UV curing. By considering the DTG of both film thicknesses, it can be concluded that as the percent cure was higher than 60% (curing time of 10 min for 0.0012 in. film and of 8 and 10 min for 0.002 in. film), the decomposition occurred at a single temperature of 440°C.

Figure 9 illustrates the decomposition at temperatures lower than 380°C was more pronounced for HP curing. In fact, it was obvious that there were two small peaks, one between 260-280°C with a corresponding weight loss of 7-9% and one at 340°C with a weight loss of 5-12%. This is because HP curing may not have been homogeneous as discussed in the previous section. Two important features can be concluded on the degradation behavior of the photoresist films. Firstly, it can be concluded that MW and HP curing resulted in stronger molecular bonds as the degradation temperature was 20°C higher than that of UV curing. Secondly, MW curing achieved the most homogeneous molecular structure as demonstrated by a single decomposition peak.

CONCLUSION

The fact that microwave technology has showed a high potential in curing photoresist films is beneficial for the PCB industry as it means that a faster, cheaper, and less energy intensive process can be achieved. The study revealed that MW was more suitable and has a greater effect on polymerization of thick films. Relatively low energy power of 200 and 500 W resulted in a low degree of cure especially for a thick film whereas a nearly complete cured film could be obtained with higher MW power of 1000 W, provided sufficient exposure time was used. Although the conventional thermal cure (hot plate) seemed to be able to produce comparable percent cure with the MW technique, it was limited by the heating uniformity as can be obviously seen from the degradation behavior. As it is well known that a domestic household MW works at a single frequency in a multimode cavity, it is inherently prone to 'hot spots' which causes heating inhomogeneity within the cavity and is a limitation of using these type of equipment in these experiments. However, with the proper MW apparatus (*i.e.* single mode cavity, proper tuning, *etc.*) it is envisaged that a more efficient MW process could be developed resulting in higher degrees of polymerization with a more uniform molecular structure.

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