Effect of Temperature-Pressure Cycles on Structural Properties of Epoxy-Based Composites for Printed Circuit Boards Applications

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ABSTRACT: During multilayer printed circuit board production, cyclic pressure–temperature (P–T) treatments can give rise to mechanical and thermal stresses with subsequent inner delamination phenomena. These are mainly related to the thermomechanical properties of epoxy resins used in the manufacturing process. In this study, the physical and chemical changes of commercial epoxy-based prepregs were investigated as a function of P–T treatments by means of thermogravimetric analysis, differential scanning calorimeter, and Fourier transform infrared-photoacoustic spectroscopy. It was verified that the thermal properties start to decrease after the first P–T treatment. In connection, absorbance spectra indicated that the peaks at 2273 cm^{-1} and 2235 cm^{-1} (cyanate group) disappeared after this treatment. Heat transfer as a function of laminate thickness (consequently, as a function of P–T treatment) is one variable that probably contributes to the findings in the work. The results of this study indicated that the analytical methods were satisfactory to determine the beginning of the deterioration of the printed circuit as a function of the P–T treatments. They can be used as a reference for quality controls and to prevent mechanical problems such as interlaminar fractures in the relevant printed circuits. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 110: 1606–1612, 2008

Key words: polymer matrix composites; curing of polymers; thermal properties; laminates; photoacoustic infrared spectroscopy

INTRODUCTION

Prepreg is a partially cured sheet of reinforced thermoset resin. For the printed circuit board (PCB) production, it is made of woven fabric of fiberglass coated with epoxy-based resins and then partially cured ("B staged" state). A multilayer PCB consists of up to 24 conductive layers separated by dielectric material (prepreg). Each layer comprising prepreg with outer layer(s) of a conductive foil (e.g., copper) is exposed to heat and high pressure ("C staged" state) in a laminating press. This processing will be repeated several times (P–T cycles—relaminating layers of B-staged prepreg and C-staged core) until it obtains the multilayer thickness.[1]

During prepreg manufacturing, there are several variables that will contribute to the quality of a good PCB multilayer lamination. For example, the reinforcement architecture, the amount of resin applied to the fiberglass fabric, the degree to which the resins have been precured, the resin composition, the uniformity of the fiberglass fabrics, the fiber/matrix interface, and the amount of heat transfer from fiberglass to resin.[2,3] PCB manufacturers by means of P–T cycles can optimize the last variable. However, the other variables are under the control of prepreg producers.

The new generation of resins for PCB applications with superior thermal, mechanical, dimensional, and electrical performance belongs to bismaleimide (BMI), cyanate ester (CE), and bismaleimide-triazine (BT) resin families.[4] In particular, BT resins are excellent candidates for this application because of their low dielectric constant, low dissipation factor, and the possibility to model the glass transition temperature (T_g) to high values from 200 up to 350°C. BMI resins have a good thermal stability that is better than that of epoxies. However, this family of resin presents poor adhesiveness. CE resins, on the other hand, give excellent performance on electrical properties, but their thermal stability is inferior to that of BMI resins.[5] Some authors have proposed

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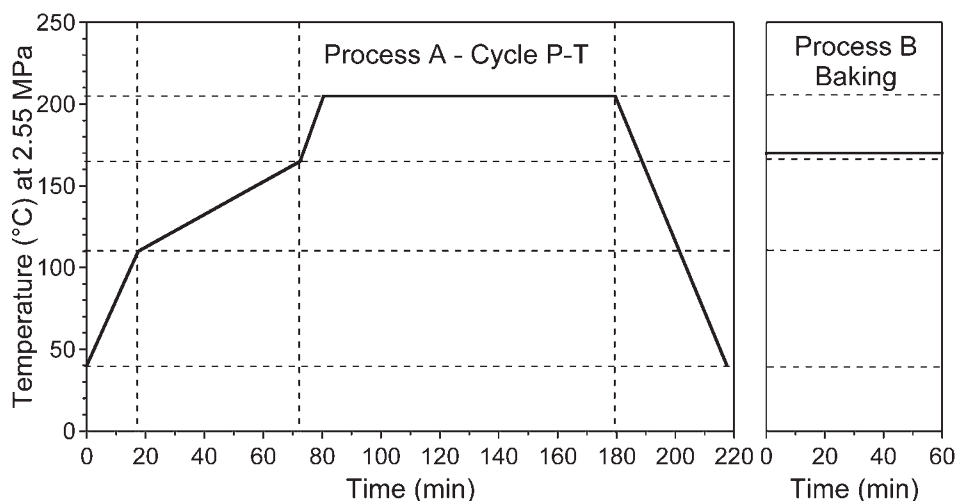

Figure 1 Temperature profile applied to the prepreg for the curing process (P–T cyclic treatment).

resin formulations containing at least two specimens from different families of resins to offset the individual drawbacks.[6–9]

One of the most common drawbacks on multilayer PCB production is the interlaminar cracking (delamination) of the system. This can be originated from cyclic loads, temperature, and moisture stresses as well as interface adhesion strength. In general, this kind of failure is analyzed on the basis of mechanical and numerical simulations studies.[2,3,10,11]

This article aims at assessing the physical and chemical changes of epoxy-based composites as a function of P–T cyclic treatments. Thermogravimetric analysis (TGA), differential scanning calorimeter (DSC), and Fourier transform infrared-photoacoustic spectroscopy (FTIR-PAS) have been used to monitor the occurrence of structural changes during the P–T curing treatments. The laminated prepreg is covered by a patent.[12] The thermosetting resin composition comprises several chemicals including resins, crosslinking agent, flame retardant, toughener, filler, accelerator, and solvent. The characterization methods will focalize principally the resins that comprise epoxy family with BMI, CE, and/or BT.[12]

EXPERIMENTAL

Material and lamination process

Laminates were prepared by using E-glass fabric/epoxy-based prepreg containing about 33 wt % of glass fiber and with a thickness of about 70 μm. The resin matrix is a proprietary epoxy blend system, which might contain BT, bromine epoxy, epoxy prepolymers, BMI resin, CE, and oligomeric styrene-*co*-maleic anhydride (SMA) as crosslinking agents. The more relevant characteristics of this resin system is the fairly high glass transition temperature (T_g) that is comprised between 170 and 220°C depending upon BT amount, reduction of the brittleness problem (although it is used SMA), and composition that permit its application to high speed and frequency circuits.[12]

The lamination process was performed according to the P–T cycle reported in Figure 1, which is equivalent to that applied in an industrial scale of PCB production. A temperature treatment spanning from 40 to 205°C with a gradient profile as sketched in Figure 1 was applied to the specimen under the pressure of 2.55 MPa. This cycle was sequentially replicated four times. After each treatment, a baking of the specimen at 170°C for 60 min was also carried out (Fig. 1). At each cycle, two further prepreg sheets were applied. Specimen codes after each one of the four cycles and relative thickness recorded as a function of the applied treatments are reported in Table I. The number and letter in sample code mean the cycle and applied process, respectively. For

TABLE I
Specimen Codes and Thickness as a Function of the P–T Cycle and Number of Prepreg Sheets

Sample (code)[a]	Prepreg sheets (no)	Process (type)	Thickness (μm)
1A	10	First cycle P–T	480
1B	10	Baking of 1A	480
2A	12	Second cycle P–T	630
2B	12	Baking of 2A	630
3A	14	Third cycle P–T	700
3B	14	Baking of 3A	700
4A	16	Fourth cycle P–T	770
4B	16	Baking of 4A	770

[a] Numbers refer to the cycle sequence; "A" refers to the process type P–T; "B" refers to baking process of the sample derived from relevant process A.

example, the sample 3A corresponds to the laminate at which two new prepreg (B-Stage) were added at each side of 2B (C-stage) and collected after the process A (Fig. 1) of the third cycle.

Characterization

Sampling was performed after each P–T cycle using a cork borer to cut round samples of 3 or 5 mm for TGA, DSC, and PAS-FTIR, respectively. In this way, each sampled specimen contained all prepreg layers as indicated in Table I.

TGA was carried out by means of a TA Q500 thermal analysis system with Thermogravimetric Analyzer Software from TA Instruments. TGA traces were obtained from samples of ~ 10–15 mg scanned at 10°C min^{-1} from 25 to 700°C under air flow rate of 60 mL min^{-1}. Two or three replicates were performed.

A Mettler TA 4000 System instrument, consisting of DSC-30 differential scanning calorimeter and TA72 GraphWare software, were used to evaluate the thermodynamic characteristics of the laminates. Samples of 10–15 mg were weighed in 40-μL aluminum pan, and an empty pan was used as a reference. The DSC temperature calibration was performed using three standard materials (In, Pb, and Zn) and the energy calibration using indium standard. Measurements were performed under nitrogen flow rate of 80 mL min^{-1} according to the following protocols: (i) first heating scan from −50 to 300°C at 10°C min^{-1}; (ii) first cooling scan from 300°C to 0°C at 100°C min^{-1} and 4 min of isotherm at the end; and (iii) second heating scan from 0 to 300°C at 10°C min^{-1}.

A Bruker IFS 66 FTIR spectrometer equipped with a Bio-Rad demodulator and a helium-purged photoacoustic cell (MTEC Model 200) was used (FTIR-PAS). A carbon black reference was used to collect the corresponding reference spectra. The stainless steel PA cell was purged by helium to provide CO_2 and moisture-free environment. The spectra were collected in the 4000–400 cm^{-1} wavenumber range at a resolution of 8 cm^{-1} and 128 scans/sample. Other settings were a speed of 2.2 kHz scan; acquisition mode of double-sided, forward–backward; opening 12 mm; source setting Globar (MIR); and Blackman-Harris three term as apodization function.

RESULTS AND DISCUSSION

Commercially available prepreg is used as bonding sheets in the multilayer PCB production. Its electronic and mechanical characteristics are due to a proprietary resin composition. This means a mixture of epoxy resin containing one of the following functionalities: epoxy, cyanate, imide, and triazine.

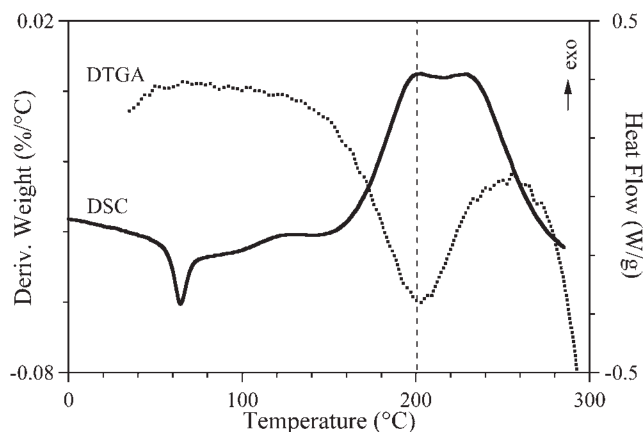

Figure 2 DTGA and DSC traces of pristine prepreg.

Crosslinking agents can be more than one, being at least one of them a copolymer of styrene and maleic anhydride.[12] Taking into consideration the described composition, one can suppose a complex chemical and physical structure. Anyway, an analytical approach was proposed to gain some insight about the physical and/or chemical origin of PCB delamination problem as an alternative to the mechanical and simulation tests.[2,3,10,11] The following sections deal with the characterization of the specimen by thermal analysis (TGA and DSC) and spectroscopic analysis (PAS-FTIR).

Thermal analysis (DSC-TGA)

Figure 2 correlates DSC thermodynamic changes of prepreg resin with TGA weight loss. DSC trace shows a glass transition with endothermic relaxation at temperature (T_g) of ~ 59°C followed by three overlapped exothermal peaks corresponding to crosslinking reactions with three different mechanisms involved. The first peak, appearing at ~ 127°C, is characterized by an enthalpy of 12 J g^{-1}, which counts for about 5% of the total reaction energy (~ 254 J g^{-1}). The second DSC peak, centered at 202°C, begins at about 140°C. These two temperature values correspond to the derivative peak of TGA (DTGA) (194°C) and to the onset temperature of the first weight loss step. The weight loss of this step is ~ 3%. Therefore, it can be supposed that a polycondensation reaction occurs in this range of temperature. The last exothermal DSC peak is centered at ~ 229°C and is highly overlapped to the second one. The proportion of energy of both peaks is equivalent to ~ 121 J g^{-1} by calculation of the partial area of the exothermal DSC peak.

Figure 3 shows the DTGA traces of the pristine untreated prepreg sample and of the laminates after the P–T treatments. The first DTGA peak of prepreg disappears in the laminates confirming its

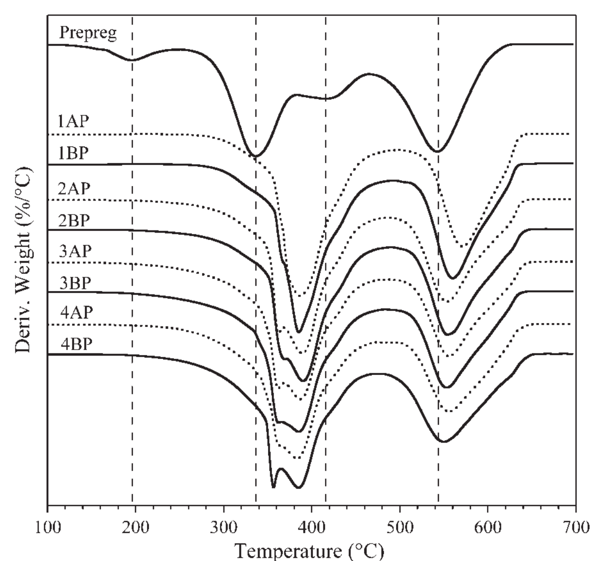

Figure 3 DTGA traces of laminates as a function of P–T treatments compared to pristine prepreg.

relationship with volatiles from polycondensation reaction after each P–T treatment. The second DTGA peak recorded for prepreg (336°C) decreased in intensity, forming a tail in the two new overlapped peaks that appeared between 350 and 420°C after P–T treatment. This means that the molecular structure in prepreg, contributing to this weight loss step, decreases in amount during crosslinking reaction. After each P–T cycle, its residual amount gives rise to a tail at the left side of the first DTGA peak of the laminates. In the first P–T treatment, the first DTGA peak of the laminate (1A and 1B) is not well resolved appearing as a shoulder on the peak at around 385°C. With the following P–T treatments, this peak begins to be resolved with the increase in its intensity. The corresponding peak temperature (T_p) slightly decreases when moving from the first to the last cycle treatment (4B), which is centered at around 356°C. Inverse behavior is observed for the second DTGA peak (385°C) of the laminates. This peak presents a shoulder at the right side of the laminate DTGA profile, which corresponds to the third peak of prepreg. The last peak of prepreg is centered at ∼ 540°C and shifts toward higher temperature with P–T treatments. However, in the laminates, it is observed that the presence of a shoulder at high temperature side broadens with the P–T treatments. This peak shifting, associated with the emerging of a broad shoulder, can be the consequence of an increased diffusion barrier to the degradation products to go beyond the laminate surface. This larger barrier is probably due to the presence of further glass fabric layers from laminate 1A to laminate 4B.

The DTGA peak corresponds to the maximum of volatilization rate of the decomposition product. Its intensity is proportional to the amount of the volatilized chemical molecules, and the width is related to the chemical mechanism of decomposition (consequently, the reaction kinetic) and/or the barrier to the mass transport of the volatile chemicals from the bulk, under the same scanning rate. The four DTGA peaks of prepreg correspond to partially crosslinked resins (B stage). As was shown earlier (Fig. 2), only the first DTGA peak of the prepreg is related to polycondensation reactions. The other three peaks result from decomposition of the partially crosslinked resins. In the thermal degradation study performed by Hong and Yeh[13] for equivalent blend system of this work (epoxy-CE-BMI), only two peaks were found for scanning up to 500°C. The first peak recorded under air atmosphere and centered at 309 and 327°C was attributed to the degradation of epoxy resin at a heating rate of 2 and 8°C min^{-1}, respectively. The peak temperature at a higher scanning rate is equivalent to the value obtained in this work. The second peak at 478°C (at 2°C min^{-1}) was not totally formed due to the low temperature limit for the end of the experiment. Anuradha and Sarojadevi[14] synthesized CE and blended with BMI at different ratios. Their integral TGA traces recorded under nitrogen atmosphere presented only one step for both pristine and blend samples. The more stable component used in the blend was BMI with a maximum decomposition rate temperature (T_p) at 485°C (corresponding to DTGA peak of this work). The value of T_p for CE was 325°C. However, the equiponderant blend BMI : CE showed a better thermal stability with T_p value of 490°C.

From data of literature, the second, third, and fourth peaks of the laminate samples studied can be attributed to epoxy, cyanate, and BMI components, respectively, of almost totally crosslinked resins. Although lamination conditions were always the same, the different DTGA profiles of the laminates shown in Figure 3 can be assigned to the different heat transfer from treatment to treatment. The shifting of the DTGA peak of 4B to slightly lower temperature than that of 1A suggests that the resin crosslinked in the previous P–T treatment is probably partially degraded. So, this will contribute to the DTGA bulk signal of the 4B. Besides, this can contribute to the broadening of the DTGA peaks of the samples from the P–T later treatment rather than the effect of the increased thickness on the mass transfer.

The residue at 700°C under air atmosphere (∼ 33%) corresponds to the amount of glass fiber specified by the prepreg producer.

Figure 4 shows how the decomposition temperature (T_d) varies with the number of carried out treatments. All data errors are within a confidence interval of 95% of significance, which were

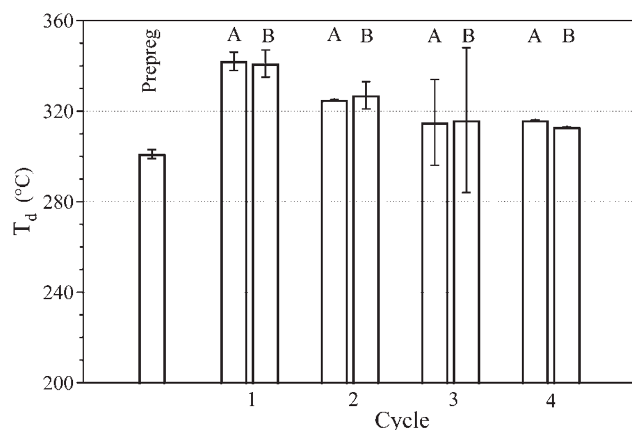

Figure 4 Changes on laminates onset decomposition temperature (T_d) as a function of P–T treatments compared to pristine prepreg.

evaluated through Student's t test. In general, the T_d mean deviation was lower than 2% with exception for samples after the third-cycle treatments that presented a mean deviation of 6 and 10% for samples from A and B processes, respectively. The higher data dispersion observed after cycle 3 (3A and 3B) does most likely originate from an irregular pressure and/or temperature distribution during the sample preparation. After the first P–T cycle (laminate 1A and 1B), T_d increases ~ 40°C due to the occurrence of the crosslinking reaction. After cycles 2–4, T_d was observed to increase only ~ 25°C, 15°C, and 15°C, respectively. Such a phenomenon can be ascribed to the contribution that the mechanical stress has on the resin degradation.

Glass transition temperature (T_g) measured for pristine prepreg and laminated after the first and second heating are reported in Table II. During the first DSC heating, prepreg (B stage) shows a T_g of ~ 59°C that increases up to 212°C in the second heating. Laminate 1A presented 238°C T_g during the first heating and 241°C T_g during the second heating. A slight decrease of these values has been observed after the backing process (sample 1B). Residual exothermic peak corresponding to ~ 0.5% of the total reaction enthalpy was observed in laminates 1A and 1B. The difference of 5°C between measured T_gs from the first and second heating means that the resins are almost totally crosslinked during the P–T treatment. Therefore, the residual reaction is completed in the second heating. Although the T_g of 1A sample increased with the thermal treatment performed in the second DSC heating, the same was not observed with that sample from baking treatment (sample 1B). A decrease of 2°C in the 1BT T_g value (first DSC heating) suggests that holding the laminate at 170°C for 60 min at 2.55 MPa might promote not only the residual crosslinked reaction but alongside some chain scission of the less stable epoxy resin. This degradation can decrease its molecular weight (MW), which can plasticize internally the system, contributing to the decreasing of T_g but not sufficient to decrease its thermal stability (see Fig. 4).

Two T_g values were instead detected in samples 2A and 2B during the first DSC heating, whereas specimen analyzed upon further P–T treatments displays three T_g values. Besides, after the first P–T treatment, T_g decreases in both first and second heating. The observation of more than one glass transition can be related to several factors, connected to macromolecular mobility. The thermosetting system is a mixture of resins with different chain flexibility and consequently different T_g. Moreover, the number of glass transitions depends on the compatibility between them. For incompatible blend, the number of glass transitions will be equivalent to the number of components. But, if this kind of blend is a system with interpenetrating crosslinked chains (IPN), the T_gs values will be distinct even though the components are more intimately mixed than in incompatible blends.[15] The decrease in MW can contribute to the decrease of T_gs values too. As suggested earlier, this can be due to a partial thermodegradation occurred during lamination process. Furthermore, the thermosetting system partially crosslinked is composed of chains with MW near to that of pristine resin and of that crosslinked with higher MW. These two levels of chain MW present different mobility and consequently different T_g. Finally, there is an instrumental factor to be considered. The change in the heat capacity (ΔC_P) at the glass transition decreases with increasing of the crosslinking degree. A low ΔC_P value can be difficult to be detected during a DSC scan that could have been possible to be detected with a higher sample mass. This could explain why samples from the first P–T treatment (1A and 1B) presented only one T_g in the first DSC heating.

TABLE II
Glass Transition Temperatures (T_g) of Laminates and Pristine Prepreg as a Function of P–T Cycles[a]

	Heating				
	First			Second	
Sample code[b]	T_{g_1} (°C)	T_{g_2} (°C)	T_{g_3} (°C)	T_{g_1} (°C)	T_{g_2} (°C)
Prepreg	59	ND	ND	ND	212
1A	ND	ND	238	ND	241
1B	ND	ND	236	ND	237
2A	ND	159	225	ND	227
2B	ND	155	235	ND	233
3A	51	147	215	ND	214
3B	34	164	224	169	206
4A	35	143	221	ND	176
4B	44	143	224	ND	224

[a] ND stands for "not detected."
[b] For code recording see Table I footnotes.

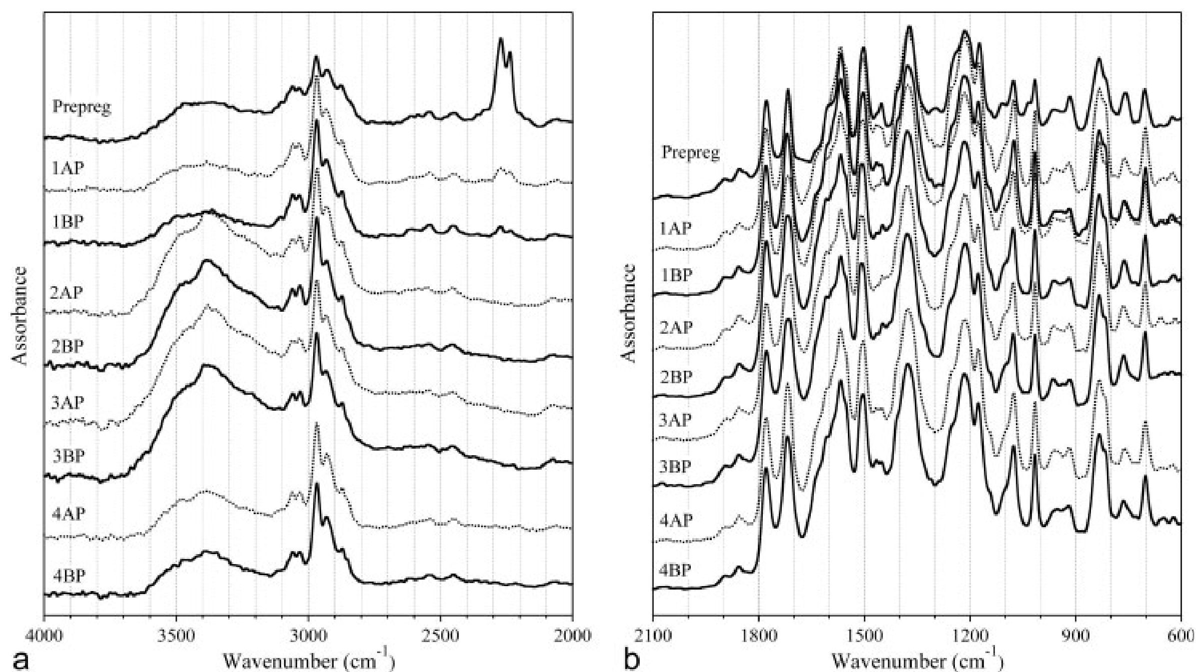

Figure 5 FTIR-PAS spectra of laminates as a function of P–T treatments compared to pristine prepreg.

As formerly observed in the TGA analysis, samples from the third treatment (3B) present a different behavior showing two glass transitions in the second DSC heating. Furthermore, the T_g values observed are lower than those observed on laminates from the others cycles. This suggests a heterogeneous heat transfer inside the sample during the P–T treatment associated to some variables that cannot be controlled; for example, moisture. In general, laminates after baking ($\times$B) present slightly higher T_g values (first DSC heating) indicating that this treatment promotes a residual crosslinking reaction. As mentioned earlier, further P–T treatments can change chemically the less stable epoxy resin, and this effect will be more significant with the increasing of thickness at each lamination. Because epoxy resin in the blend system is responsible for the adhesion, its degradation will influence the delamination phenomena.

Fourier transform infrared analysis

To follow any chemical structural changes that can occur during each P–T treatment, samples have been analyzed by PAS-Fourier transform infrared (FTIR). This spectroscopic technique is a solution to the analysis of opaque samples with better resolution than the other approaches (e.g., DRIFTS and ATR). The PAS signal is proportional to the thermal diffusion depth in the sample. Because all the samples surfaces analyzed are made of equivalent material, it can be supposed that absorption bands intensities are proportional to the P–T treatment. Figure 5 shows the absorbance spectra as a function of the wavenumber for the different P–T treatments. In Figure 5(a,b), spectra ranging from 2000 to 4000 cm^{-1} and from 600 to 2100 cm^{-1}, respectively, are reported. Aromatic band (1502 cm^{-1}) was used as reference to normalize spectra. Peaks at 2273 cm^{-1} and 2235 cm^{-1} appear for partially cured prepreg (B stage) and correspond to vibration modes associated to the absorptions of the cyanate groups. Their occurrence in laminates from the first P–T treatment (1A and 1B) is in accordance with DSC residual exothermic peaks of unreacted groups. Besides, in the successive P–T treatment, these peaks completely disappear accompanied by the appearance of a shoulder at 1554 cm^{-1} and the intensity increasing of the bands at 1373 cm^{-1} and 835 cm^{-1} (all corresponding to triazine ring) that can be supposed to be due to a polycyclotrimerization of an aromatic dicyanate.[6] At the same time, the absorptions band located at 917 cm^{-1} attributable to the epoxy group decreases, thus confirming the occurrence of a crosslinking process. On the other hand, a new peak appeared at 3468 cm^{-1} overlapping that one at 3383 cm^{-1} after the first P–T cycle (1A and 1B). Its intensity and resolution were higher for laminates from the second and third cycles. In connection, the absorption band centered at 1716 cm^{-1} is wider than that of prepreg and of the other cycles. This can be due to the formation of a new kind of carbonyl groups. Studies on the effect of moisture in the BMI[16,17] and CE[17–20] resins have suggested hydrolysis degradation with different reaction mechanism

for both resins, which explain the problem of delamination or blistering. The degradation products can be, for example, phenol, carboxylic acid, amide, carbamate, and amine. So, the intensity of peak increase in the range from 3200 to 3600 cm^{-1}, observed for samples from the second and third cycles, can be attributed to both the O—H and the N—H absorptions, and the enlargement of carbonyl band can be attributed to carbamate moiety. Hydrolytic degradation will decrease MW and chain flexibility of resins, which explain the lower values of T_g for samples after the first cycle (see first heating column in Table II).

CONCLUSIONS

This physicochemical study has enabled us to show in which way successive P–T cyclic treatments affect the chemical structures of epoxy-based composites and how trapped volatiles from polycondensation and/or moisture can be some of the relevant factors that influence the reaction mechanism. Consequently, these changes can deteriorate the mechanical and electrical properties of the material. Evidences have been collected that the cure P–T treatments in the PCB manufacturing process can be responsible for the failure of the circuit. The moisture present in the ambient and/or in the prepreg will have an important hole in the quality of the obtained laminate. Besides, heat transfer is a variable to be taken into account as a function of laminate thickness.

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