Curing Behavior of the Plate Produced by Nonmetallic Materials Recycled from Waste Printed Circuit Boards

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ABSTRACT: A plate was produced by nonmetallic materials (NMs) from waste printed circuit boards (WPCBs) as reinforcing filler. In the whole preparation process of the plate, molding is most important because during molding, the bonding agent would cure with the crosslinking agent in the presence of initiator and the properties of the plate is significantly dependent on the extent of cure. This article used differential scanning calorimetry (DSC) to study the curing behavior of the plate. The results showed that filling NMs lowered down the reaction rate and reduced the activation energy E_a to 87.58 kJ/mol. Optimum curing temperature ranges were determined based on DSC data and two

types of plates were made under this temperature range. The mechanical properties of the plates were tested and the results showed that the plate of NMs was of moderate quality, which verified the feasibility of reusing NMs to substitute for the conventional fillers such as glass fiber, calcium carbonate, etc. This paper offers theoretical basis for producing good quality plate by NMs from WPCBs and resolving the environmental pollution caused by NMs. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 122: 1829–1837, 2011

Key words: plate of NMs; nonmetallic materials; curing behavior; DSC

INTRODUCTION

Printed circuit boards (PCBs) are the basic component of various electronic products, which is also known as the biggest industry in the electronic component industry area. According to the statistics, the average growth rate of PCBs over the world reaches by 8.7% in recent years.¹ It's obvious that the amount of waste printed circuit boards (WPCBs) also increases fast along with this trend due to the short service life of electronic products. As a result, dealing with those WPCBs has become a big problem and needs solutions urgently. Among those different treatments of WPCBs, recycling is the optimal choice. At present, researches on the recycling of waste PCBs mainly focus on the recovery of metals such as Cu, Al, Sn, etc.,^{2,3} while studies on the reusing of nonmetallic materials (NMs) are rare because of their relatively low economic interest and especial difficulties in treatment.⁴ The main treatments of NMs are still incineration and landfill, which have caused serious secondary pollution to the environment.5,6 However, NMs from WPCBs, which account

about 70 wt % of the whole and mainly contain glass fibers and resins, are of good thermal stability and excellence chemical resistance. NMs have a high recycling value.

At present, the main recycling methods of NMs from WPCBs include incineration, pyrolysis and physical recovery. Incineration is on the purpose of recycling thermal energy from NMs. However, it is not the best method for recycling NMs because the inorganic fillers contained in NMs, such as glass fibers, could significantly reduces the fuel efficiency.⁷ Pyrolysis is a chemical recycling technique that has been widely researched as a method of recycling synthetic polymers including polymers that are mixed with glass fibers. Pyrolysis of NMs from WPCBs could lead to the formation of gases, oils, and chars which can be used as chemical feedstock or fuels.^{4,8–10} Physical recovery directly reuses NMs as a filler to produce various products, like inorganic constructional material,^{8,11–13} composite material,^{14–17} and asphalt.¹⁸ For its advantages of relatively easy processing, low cost and less secondary pollution, physical recycling is a promising recycling method in recycling NMs.

In our studies, NMs reclaimed from WPCBs were reused as reinforcing filler to make a plate.^{7,19} The plate is a kind of composite plate, comprising NMs, bonding agent, reinforcing materials and other additives. It can be used as building decoration materials, molds, well covers, etc. For the various advantages of high chemical resistance, favorable process

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Figure 1 The flow chart of the preparation process of the plate of NMs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

condition and low cost,²⁰ unsaturated polyester (UP) was used as bonding agent. When UP was used, styrene was added as crosslinking agent and *tert*-butyl perbenzoate (TBPB) was used as initiator. Besides, glass fibers and calcium carbonate (CaCO₃) were added as reinforcing materials to improve the mechanical properties of the plate. The whole preparation process of the plate of NMs is described in Figure 1. This provides an environmental friendly and economic way to recycle NMs from WPCBs.

In the whole preparation process of the plate of NMs, molding is especially important because during molding, UP will cure with styrene in the presence of initiator and the properties of the cured resin significantly depended on the extent of cure²¹. In addition, adding NMs into UP would make the curing reaction more complex due to its complicated compositions. To get a good understanding about the impact that NMs had on the preparation of the plate and determine the optimum curing condition temperatures, this paper used differential scanning calorimetry (DSC), which was known as a powerful thermal analysis technique to determine cure kinetics of thermoset resins,²² to study the curing behavior of the plate of NMs. This could offer theoretical basis for producing good quality plate by NMs from WPCBs.

EXPERIMENTAL

Preparation of nonmetallic materials

WPCBs used in this article were the type of glass fiber cloth laminate without electronic components. WPCBs were firstly crushed by a high-speed shearing machine as the crude crusher and then a hammer grinder as the second crusher into powder with diameter less than 0.3 mm²³. Then the powder was through a corona electrostatic separator to separate NMs from metals, as showed in Figure 2. After separation, NMs were screened and the ones with particle size less than 0.07 mm were used in this study.

Field emission scanning electron microscopy (SEM), FEI SIRION 200, was employed to observe the microstructure of NMs. Prior to the analysis, the surfaces of NMs were sputter coated with a thin layer of gold under vacuum condition. After selected a micro zone of NMs by SEM, energy dispersive spectrometer (EDS) was used to detect the elements it comprised.

Preparation of curing systems

Simplified mixtures were employed to study the curing behavior. UP used in this article was *m*-phthalic acid unsaturated polyester resin, type of DS-801*N*-1 in the market. To get a better understanding of the curing behavior of the plate of NMs, UP without reinforcing filler and UP filled with CaCO₃ were used to compare with UP filled with NMs. These three curing systems were respectively, named "UP," "UP/CaCO₃," and "UP/NMs." As excessive content of CaCO₃ and NMs would increase the viscosity of the resin and result in a poor dispersion,



Figure 2 Nonmetallic materials and metals from waste PCBs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Mass Ratio of Each Component of UP, UP/CaCO ₃ , and UP/NMs					
	Content (wt %)				
Raw materials	UP	UP/CaCO ₃	UP/NMs		
Unsaturated polyester	18	18	18		
CaCO ₃	-	20	_		
Nonmetallic materials	_	_	20		
Styrene	6	6	6		
TBPB	0.2	0.2	0.2		

TABLE I

the adding content of CaCO₃ and NMs is suitable respectively, at 20 wt %. Ingredients of each curing system were added according to proportions listed in Table I. First, UP, styrene, and the initiator TBPB were mixed homogeneously, stirring for 15 min by magnetic stirrer. Then, CaCO₃ and NMs were respectively, added into the mixture and mixed for another 15min.

DSC measurement

The thermodynamic properties of each curing system were detected by DSC instrument (DSC Q2000 V24.4 Build 116). The curing systems were heated constantly from 90 to 200°C, respectively, at heating rates of 5, 10, 15, and 20° C min⁻¹, with nitrogen flow at 20 mL min⁻¹. UP, UP/CaCO₃ and UP/NMs reacted at the heating rate of 10°C min⁻¹ were detected by SEM to observe the microstructures.

The kinetic parameters of each curing system were evaluated respectively, by Kissinger method²⁴ and Flynn-Wall-Ozawa method.25-27 The two methods used the relationship between the heating rate β and the peak temperature T_p of the exothermic curve observed by DSC to calculate the value of activation energy E_a and pre-exponential factor A.

The Kissinger equation can be expressed as eq. (1)

$$-\ln\left(\frac{\beta}{T_p^2}\right) = -\ln\left(\frac{AR}{E_a}\right) + \frac{E_a}{RT_p} \tag{1}$$

where R is the mol gas constant. The value of A and E_a can be obtained by graphing $-\ln(\beta/T_p^2)$ versus $1/T_{p}$.

The Flynn-Wall-Ozawa equation can be described as following,

$$\ln\beta = \ln A - 1.056 \frac{E_a}{RT_P} \tag{2}$$

A plot of $\ln(\beta)$ versus $1/T_p$ is a straight line. The slope of the line is the value of $-1.056 E_a/R$, and the increment is the value of A.

TABLE II Raw Materials of the Raw Plate and the Plate of NMs

	Content (wt %)			
Raw materials	Raw plate	Plate of NMs		
Unsaturated polyester	18	18		
CaCO ₃	64	44		
Nonmetallic materials	_	20		
Glass fibers	10	10		
Styrene	6	6		
TBPB	0.2	0.2		
Zinc stearate	1	1		
Pigment	0.8	0.8		

Mechanical property test of the plate

A plate of NMs was made under the temperature deduced from DSC data. To get compared with, a raw plate without filling with NMs was made under the same condition. In consideration of the surface smoothness of the plate, NMs only substituted a part of CaCO₃. The raw materials of the raw plate and the plate of NMs were listed in Table II. Besides the materials of simplified mixture used in DSC measurement, zinc stearate, and pigment were added as coloring agent. NMs, CaCO₃ and glass fibers were premixed in a double Z-kneader. Other components included UP, polystyrene, TBPB, zinc stearate, and pigment were stirred for 10 min with a high shear mixer. Then, the resin paste was added to the double Z-kneader and kneaded for 15 min. After the fillers were saturated with resin, the whole mixture was molded into plate using a self-made hot-press former. The specimens of the plates were shown in Figure 3.



Figure 3 The specimens of the plate of NMs. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

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Figure 4 SEM diagram of NMs.

The mechanical properties of the plate of NMs, including flexural strength, flexural modulus, Charpy impact strength, and Rockwell hardness, were tested. In flexural test and impact test, the plates were cut into normalized size of 80 mm \times 10 mm \times 4 mm and measured respectively, according to the method of GB 9341-2000 and GB/T 1043-93 in China. Rockwell hardness was determined in accordance with GB 9342-88 using a ball indenter.

RESULTS AND DISCUSSION

Nonmetallic materials analysis

NMs recovered from waste PCBs mainly comprise resin powders and glass fiber yarns. As is known, glass fibers can be used as conventional filler of UP to enhance the strength, corrosion resistance, and electrical insulation. But before being filled, glass fibers should be pretreated by size to increase the compatibility with UP. The SEM diagram of NMs (Fig. 4) shows that resins have peeled off from glass fiber yarns. On the surface of glass fiber yarns there are some residual resins. This may help NMs present a fine wetting property and lead good adhesiveness with UP because UP can spread to cover most surface of resin powders.²⁸

The EDS detecting results of elements contained in NMs were shown in Figure 5. Besides elements of carbon, hydrogen and oxygen, NMs also comprise some metal elements, such as copper and aluminum, which are residual metals after the separation process. Limited to the separation efficiency, it is impossible to separate metals completely from NMs.^{29,30} Among all kinds of metals, copper accounts most as it was used to clothe PCBs plate. To analyze the content of residual copper in NMs, NMs were



Figure 5 EDS detect result of NMs ((a) resins on glass fiber yarn; (b) scattered resins).



Figure 6 Decomposition of TBPB.

preconditioned by chloroazotic acid, and then the digestion solution was detected by Inductively Coupled Plasma (ICP). The result showed that the weight percent of residual copper in the NMs was 2.7%. The affect of residual copper on the curing process should not be neglected.

Curing process of UP resins

The cure of UP resins is a free radical polymerization between UP resin and styrene initiated by initiators. The cure is complex because many reactive processes occur simultaneously. When the internal thermal energy is large enough, the initiator TBPB decomposes chemically to give free radicals, as showed in Figure 6. These free radicals are generally very unstable and react readily to open the C=C double bonds of unsaturated oligomers and styrene monomer. Launched by these free radicals, the functional C=C bonds of UP and styrene break and forms a bridge between the two polymer chains.

The crosslinking of C=C bonds could be mainly divided into three reactive processes: styrene-polyester copolymerization, styrene homopolymerization and polyester homopolymerization.^{31–33} The general reaction can be expressed as Figure 7. In these reactive processes, the resin temperature increases caused by the exothermic reaction, which accelerates the curing reaction of UP resins. The molecular weights of polystyrene and polyester increases and three-dimensional networks are finally formed after the curing process fulfilled. The fillers, NMs and



Figure 7 Curing process of UP-Styrene (x, y, z, n, m are the degree of polymerization).

CaCO₃, insert among the resin matrix and improve the mechanical properties.

Thermokinetics analysis

A single exothermic peak can be seen in DSC curve of each curing system, as shown in Figure 8. The



Figure 8 DSC curve of (a) UP, (b) UP/CaCO₃, (c) UP/ NMs.



Figure 9 Conversion degree α as a function of temperature *T* at different heating rates β during the curing process of (a) UP, (b) UP/CaCO₃ and (c) UP/NMs.

peak temperature ascends with the increase of heating rate β . A similar result has been found in other works.^{34,35}

The conversation degree α of the curing is a function of temperature *T* at a certain heating rate β . It is postulated that UP cured completely under this condition, so the relationship between α and *T* can be got, as shown in Figure 9. The slope of α and *T*, marked as $\frac{d\alpha}{dT}$ in isoconversional method, is used to

describe the reaction rate.³⁶ It can be seen from Figure 9 that the reactions of UP and UP/CaCO₃ respectively, finished within the increasing 10° C and 5° C. The decreases in curing time by the presence of CaCO₃ could be explained using Arrhenius theory, which is referred to that the reaction rate constant is dependent on reaction temperature, collision







Figure 10 SEM diagram of (a) UP, (b) UP/CaCO₃, and (c) UP/NMs after curing.

Sample	$\beta/^{\circ}C \cdot min^{-1}$	$T_i / {^{\circ}C}$	$T_f / ^{\circ}C$	$T_p/^{\circ}C$	$\Delta H_{\rm cure}/{ m J~g^{-1}}$
UP	5	129.53	137.72	133.53	-333.56
	10	140.36	149.11	144.77	-320.09
	15	145.70	156.05	150.92	-312.78
	20	148.20	158.63	153.65	-329.08
UP/CaCO ₃	5	140.45	145.91	142.81	-193.76
	10	143.97	155.35	149.39	-207.81
	15	148.01	161.49	154.72	-220.67
	20	149.45	164.92	157.54	-240.81
UP/NMs	5	108.54	134.01	124.03	-205.66
	10	113.37	145.45	133.90	-202.22
	15	115.68	150.82	138.68	-212.30
	20	124.78	155.09	144.75	-147.33

 TABLE III

 T_{ir} T_{fr} T_{pr} and ΔH_{cure} of the Three Curing Systems Obtained from DSC at Heating Rates of 5, 10, 15, and 20°C/min

factors.³⁷ When CaCO₃ filler was added into the UP, the diffusion of the reactive species for curing was restricted due to filler-filler interaction in the CaCO₃. This implied that CaCO₃ caused an increase in the collision factor of the reactive species around the neighborhoods in the curing system. Therefore, this resulted in an auto-acceleration reaction of the UP/ CaCO₃ curing system. Compared with UP and UP/ CaCO₃, the exothermic peak of UP/NMs was much gentler. The reaction temperature range of UP/NMs was broadened, with more than 20°C. And the slope of the curve decreased obviously, which reflected the reaction rate of UP/NMs lowered down. When the conversation degree α was at 0.5, the value of $\frac{d\alpha}{dT}$ of UP, UP/CaCO₃ and UP/NMs curing system respectively were 9448.4, 11821.0, and 8944.7°C⁻¹. It can be known that the reaction rate of UP/NMs was 5.3% lower than UP and 24.3% lower than UP/ $CaCO_3$. In relation to this investigation, the presence of NMs blocked the spreading of molecules and reduced the fluidity of the resin, which affected the crosslinking polymerization between molecules of UP and styrene. This can also be seen from SEM diagrams of the cured resins. As shown in Figure 10, the surface of UP and UP/CaCO₃ curing products is much smoother than that of UP/NMs. There are a lot of glass fiber yarns inside the cured UP/NMs, which impeded the reaction progressing.

Thermodynamic parameters of each curing system from DSC measurement were reported in Table III. T_i , T_f , and T_p respectively, represent the onset temperature, final temperature and peak temperature of the curing process, and ΔH_{cure} presents the exothermal enthalpy.³⁸ An average value of 323.9, 215.8, 191.9 J/g are respectively, assigned to the heat of curing of UP, UP/CaCO₃, and UP/NMs curing system, in accordance with some other reported researches.^{34,35} Compared with UP curing system, all values of T_i , T_{f_i} and T_p of UP/CaCO₃ increased, which meant the presence of CaCO₃ enhanced the difficulty of the initiation of cure. However, in comparison with the former two, all the thermodynamic parameters of UP/NMs decreased, especially the T_i which reduced by 20–25°C. This indicated that NMs could decrease the curing temperature and make the reaction easier to occur.

As preciously stated, the reaction between UP resin and styrene is a free radical polymerization. The occurrence of the cure is launched by the decomposition of initiator. It has been reported by some researchers that some metallic salts, such as cobalt, manganese, and copper salts can promote the decomposition of the initiator at low temperature, leading a decrease in curing time and curing temperature.^{39,40} Copper contained in NMs may act as promoter for the decomposition of initiator at lower temperature.

The values of T_p and β listed in Table III were used to evaluate activation energy E_a according to eqs. (1) and (2), respectively. The results were reported in Table IV and Figures 11 and 12. In all case, the value of E_a showed good reproducibility.

 TABLE IV

 Kinetic Parameters of the UP, UP/CaCO₃, and UP/NM Curing System

	Kissinger model		Ozawa model			
Sample	$E_a/kJ mol^{-1}$	lnA	R^2	$E_a/kJ mol^{-1}$	lnA	R^2
UP	89.61	25.35	0.9867	91.79	30.19	0.9867
$UP/CaCO_3$	130.37	37.01	0.9937	130.95	30.13	0.9943
UP/NMs	87.58	25.44	0.9903	89.72	41.45	0.9903

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2.8

1.4

0.00232

(_sχ) 2.1 βul



 $1/\text{Tp} \ (\text{K}^{-1})$ **Figure 11** $-\ln(\beta/T_p^2)$ versus $(1/T_p)$ plotting according to Kissinger method.

0.00240

0.00248

The activation energies calculated by the two methods are consistent with each other. Filled with CaCO₃, E_a of UP/CaCO₃ curing system increased substantially. As is known, CaCO₃ is of high surface energy and strong polar, which makes it need more energy to disperse well in the resin matrix.^{41,42} However, the value of E_a of UP/NMs curing system decreased. In Kissinger method, it was 2.03 kJ mol⁻¹ less than UP curing system's and 42.79 kJ mol⁻¹ less than UP/CaCO₃ curing system's. This reflected the presence of CaCO₃ enhanced the curing energy barrier while NMs reduced the activation energy and descended the energy barrier. The reason might be that some residual metals contained in NMs, such as copper and aluminum, could catalyze the reaction, resulting in reducing the reaction energy.

Optimum curing temperature range

In the producing process of the plate, the modeling temperature was constant. To eliminate the impact of heating rate on curing parameters, T_i , T_p , and T_f



Figure 12 $\ln(\beta)$ versus $(1/T_p)$ plotting according to Ozawa method.

TABLE V Curing Process Temperatures

Sample	$T_{gl}/^{\circ}C$	$T_{\rm cure}/^{\circ}{\rm C}$	$T_{\text{treat}} / ^{\circ} \text{C}$
UP UP/CaCO ₃ UP/NMs	125.6 137.7 102.8	129.1 138.7 118.6	133.0 141.1 129.2

were respectively, extrapolated at the heating rate $\beta = 0^{\circ}$ C/min by the method of T- β extrapolation (Table V).⁴³ In the process, T_{i} , T_{p} , and T_{f} , respectively, represent apparent gelatinization temperature T_{gl} , curing temperature T_{cure} , post-treat temperature T_{treat} when the heating rate β is at 0°C min⁻¹. To ensure the completeness of the cure, temperature range from T_{cure} to T_{treat} was determined as the optimum curing temperature range.

The optimum curing temperature range could provide references for the practical molding temperature for producing plate. In theory, the optimum curing temperature range of UP/CaCO₃ and UP/NMs are respectively, $138-141^{\circ}$ C and $120-130^{\circ}$ C. Obviously, UP/NMs needs lower temperature than UP/CaCO₃ does, which optimizes the process condition as well as reduces the consumption of energy.

Mechanical properties of the plate of NMs

According to the proportion of $CaCO_3$ and NMs in the plate of NMs, the molding temperature would be appropriate when at 135–140°C on the basis of the determined optimum curing temperature range. So the top/bottom temperature of the self- made hot-press former to make plates was finally determined at 140/135°C. The test results of mechanical performance of the plates were listed in Table VI.

Charpy impact strength is defined as the amount of energy absorbed in fracturing a specimen at high velocity. Flexural strength is maximum bending stress developed in a specimen just before it cracks or breaks. Rockwell hardness is a measurement of the nonrecoverable deformation of the material under a compressive stress. These measurements are the basic indexes for evaluating the mechanical performance of a material. The tests show the indexes of the two kinds of plates are similar and both of them are of moderate quality. The adding of NMs as

TABLE VI Mechanical Properties of the Raw Plate and the Plate of NMs

Sample	Raw plate	Plate of NMs
Charpy impact strength/kJ m ⁻²	4.02 59.85	4.34
Flexural modulus/GPa Rockwell hardness HRM	9.70 86.34	9.13 93.60

filler enhanced Charpy impact strength and Rockwell hardness of the plate to some extent but leaded a little decline in flexural strength and flexural modulus.

Although the mechanical performance of the plate of NMs is not competitive with those high performance materials, it's better than those composites using fly ash and silica as alternative filler reported by some researchers.^{44,45} The plate of NMs is qualified for using as models, manhole covers, and so on. This also verifies the feasibility of reusing NMs from waste PCBs as filler to produce recycled materials.

CONCLUSIONS

The results of DSC measurement showed that adding NMs into the resin lowered down the reaction rate. NMs blocked the spreading of molecules and reduced the fluidity of the resin, which affected the crosslinking polymerization between the molecules of UP and styrene. In addition, NMs also decreased the curing temperature by 10–20°C and reduced the activation energy E_a to 87.58 kJ mol⁻¹. The residual metals contained in NMs could act as promoter of the cure and descend the energy barrier.

The optimum curing temperature range deduced from the DSC data of UP/CaCO₃ and UP/NMs was respectively, around 140°C and 120–130°C. In the process, filling NMs into UP could optimize the process condition and reduce the consumption of energy. The mechanical properties tests showed that the plate of NMs was of moderate quality. NMs can be reused to substitute for the conventional fillers, which realized comprehensive utilization of NMs from WPCBs.

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