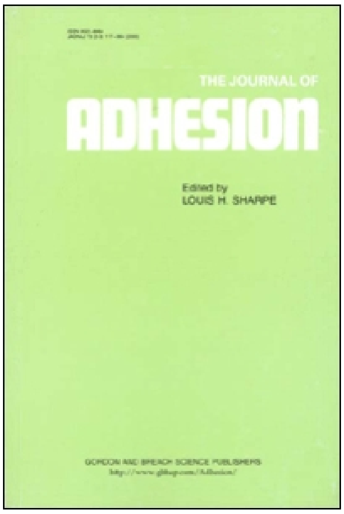

The Journal of Adhesion
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

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To cite this Article Xi, Xiangrong , Ye, Zhenming , Yu, Chaosheng , Lin, Wei , Qi, Xin , Wang, Lin and Xiong, Qi(2010) 'Preparation and Characterization of Electroconductive Adhesives of NanoG/Polyurethane-Epoxy IPNs', The Journal of Adhesion, 86: 8, 793 — 806

To link to this Article: DOI: 10.1080/00218464.2010.498306

URL: http://dx.doi.org/10.1080/00218464.2010.498306

The Journal of Adhesion, 86:793–806, 2010

ISSN: 0021-8464 print/1545-5823 online
DOI: 10.1080/00218464.2010.498306

Preparation and Characterization of Electroconductive Adhesives of NanoG/Polyurethane-Epoxy IPNs

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Polyurethanes (PU) based on toluene diisocyanate (TDI) and polypropylene glycol 2000 (PPG) were reacted with an epoxy resin (EP) to prepare interpenetrating polymer networks (IPNs). Three kinds of electroconductive adhesives were prepared by dispersing nano-graphite (NanoG) into different matrices, i.e., *pure PU, crosslinked PU/EP, and pure EP. The effects of epoxy content on morphological structure, conducting properties, thermal stability, and adhesive properties of the electroconductive adhesives were investigated by Fourier transform infrared spectroscopy, scanning electron microscopy, standard digital multimeter, dynamic mechanical thermal analysis, and lapshear tests. The results indicate that epoxy in the polyurethane-epoxy IPN adhesives plays an important role in clanging the morphological structure and improving conductivity properties, thermal stability, and adhesive properties of the electroconductive adhesives of PU.*

Keywords: Electroconductive adhesive; Epoxy resin; IPNs; Nano-graphite; Polyurethane resin

1. INTRODUCTION

Electroconductive adhesives have attracted attention extensively since they exhibit both higher adhesion and electrical conduction capabilities. They are available for numerous potential applications because electroconductive adhesives can provide antistatic behavior, dissipate or shield charge, and connect circuits [1,2]. Unfilled adhesive systems inherently

Received 22 September 2009; in final form 10 February 2010.

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have very low electrical conductivities due to their organic nature. Applications of polymeric materials are typically limited in many applications due to their inherent low thermal conductivity, low thermal stability, and high electrical resistivity. However, polymers are attractive because of their high strength-to-weight ratios, low cost, and easy processing. One way of producing electroconductive adhesives is by adding fillers such as silver, gold, nickel, aluminum, carbon black, and graphite to the matrices [3]. Usually, addition of nanometric additives to polymers may greatly enhance their properties and, consequently, their functionality [4,5]. Graphite is frequently used as an electrically conductive filler due to its moderate cost and good conducting properties. Graphite also has a positive influence on the thermal, dimensional stability, and adhesive properties apart from imparting high electrical conductivities to adhesives. Additionally, the remarkable structural, mechanical, and electronic properties of nanoscale graphite have extended the use of graphite [6].

Recently, interpenetrating polymer networks (IPNs) have attracted great attention and have constituted a rapidly developing branch of polymer composite materials, which may combine the properties of the components forming networks [7–9]. The main advantage of these IPNs is that they are less affected by external stresses than polymer blends composed of less compatible, physically mixed polymers [10,11]. Owing to the properties exhibited, network structures of IPNs are superior to those of the original constituting polymers. In particular, mechanical properties of interpenetrating polymer networks (IPNs) are often better than those of the respective constituent polymers. Therefore, many valuable systems have been developed by taking advantage of the IPN structure [12,13]. In order to improve the adhesive mechanical properties of a polyurethane resin (PU), an epoxy resin (EP) is introduced to partially crosslink the polyurethane resin to prepare the polyurethane-epoxy IPNs.

In this study, we synthesized polyurethane-epoxy IPNs and subsequently prepared NanoG (nano graphite)/polyurethane-epoxy IPN electroconductive adhesives, together with NanoG/pure PU and NanoG/pure EP electroconductive adhesives for comparison. Electrical conductivities, adhesive properties, and thermal stabilities of electroconductive adhesives were investigated.

2. EXPERIMENTAL

2.1. Materials

Polypropylene glycol 2000 (PPG) and 1,1,1-Tris(hydroxymethy) propane (TMP) used in this study were obtained from Sinopharm

Chemical Reagent Co., Ltd. Shanghai, China; toluene diisocyanate (TDI) was supplied by the Sixth Factory of Tianjin Chemical Reagents, Tianjin, China; dibutyltin dilaurate (DBTDL) was purchased from the Tianjin Guangfu Fine Chemical Research Institute, Tianjin, China; DGEBPA epoxy resin E-51 (Epoxide value: 0.48~0.54, Technical grade) was obtained from Wuxi Resin Factory of Blue Star New Chemical Materials Co., Ltd. Wuxi, China; Triethanolamine (Analytically pure, TEA) was purchased from Tianjin Basifu Chemical Trade Co., Ltd. Tianjin, China; the expandable graphite (Technical grade, EG) was obtained from Hebei Maoyuan Chemical Co., Ltd. Jinzhou, China. All of the chemicals were used as received.

2.2. Methods

2.2.1. Preparation of Nano-Graphite

The expandable graphite was heat-treated at 1050°C for 30 sec in a muffle furnace (SX2-10-12, Wuhan Jangyu Electrical Furnace Manufacture Co., Ltd., Wuhan, China) to obtain wormlike expanded graphite (EG). Then, EG suspended in absolute ethyl alcohol was shaken in an ultrasonic bath (ultrasonic cleaning, CQ25-6, Shanghai Hongxing Ultrasonic Electronic Instrument Co., Ltd., Shanghai, China) for 72 h to be fragmented into nanometer-sized graphite fragments, that then were filtered and dried in a vacuum oven (thermostatic vacuum dryer, ZKF030, Shanghai Experiment Instrument Plant, Shanghai, China) at 120°C to get nanoscale graphite (NanoG).

2.2.2. Synthesis of Matrices

The synthesis of polyurethane resin was carried out in a four-necked flask equipped with a stirrer, thermometer, nitrogen gas inlet, and a condenser with a drying tube. PPG was dried and degassed at 80°C under vacuum for 3 h prior to use. The pre-polymer was prepared by reacting PPG with TDI in the presence of dibutyltin dilaurate (DBTDL) (0.1~0.2% of the total mass) for 4~6 h at 70°C with stirring under nitrogen flow.

The synthesis of polyurethane-epoxy IPN resin was carried out in a four-necked flask equipped as above. The polyurethane-epoxy IPN was prepared by reacting polyurethane with epoxy in the presence of DBTDL (1% of the total mass) for 12 h at 70°C with stirring under nitrogen flow. The amounts for each reactants were listed in Table 1.

2.2.3. Preparation of Electroconductive Adhesives

In this study, three kinds of electroconductive adhesives were developed using the following polymeric matrices, namely, polyurethane,

TABLE 1 The Amounts of Reactants for Each Polymeric Matrix

Adhesives	Constituents	Amounts/g
PU	PPG-2000	10.0 ± 0.1
	TDI	4.4 ± 0.1
	DBTDL	0.014 ± 0.005
PU/20EP	PU	8.0 ± 0.1
	EP	2.0 ± 0.1
	DBTDL	0.10 ± 0.01

polyurethane-epoxy IPN, and epoxy resin E-51. The amounts for each ingredient are tabulated in Table 2. The electroconductive adhesives were prepared by thoroughly mixing electrically conductive filler and respective polymeric matrices and hardener at room temperature for 20 min. The NanoG/PU electroconductive adhesive, which was comprised of polyurethane resin, filler (NanoG), and hardener (TMP), was cured at room temperature for 2 days and subsequently at 100°C for another 3 h prior to characterization. The NanoG/PU/EP IPNs electroconductive adhesive, which was comprised of polyurethane-epoxy IPNs resin, filler (NanoG), and hardener (TMP:TEA = 1:3), was cured at 80°C for 5 h and subsequently at 120°C for another 2 h prior to characterization. The NanoG/EP electroconductive adhesive, which was comprised of epoxy resin E-51, filler (NanoG), and hardener (TEA), was cured at 120°C for 180 min and then was ready for characterization.

TABLE 2 The Amounts for Each Ingredient for Electroconductive Adhesives

Electroconductive adhesives	Constituents	NanoG%					
		1%	3%	6%	9%	12%	15%
NanoG/PU	PU/g	1.0010	1.0050	1.0060	1.0100	1.0080	1.0110
	TMP/g	0.0101	0.0105	0.0108	0.0103	0.0105	0.0109
	NanoG/g	0.0102	0.0308	0.0638	0.0990	0.1369	0.1771
NanoG/PU/ 20EP IPNs	PU/20EP IPNs/g	1.0040	1.0020	1.0100	1.0120	1.0090	1.0100
	TMP + TEA (1:3)/g	0.0366	0.0361	0.0364	0.0363	0.0366	0.0360
	NanoG/g	0.0101	0.0309	0.0638	0.0989	0.1364	0.1765
NanoG/EP	EP/g	1.0030	1.0060	1.0090	1.0020	1.0070	1.0090
	TEA/g	0.1401	0.1409	0.1402	0.1406	0.1408	0.1402
	NanoG/g	0.0105	0.0310	0.0636	0.0984	0.1366	0.1761

2.2.4. Measurement of Electrical Properties

Electroconductive adhesive layers were prepared, sandwiched between two acetone-degreased copper sheets, to allow electrical resistance characterization. The adhesive joint dimension was 2 cm in diameter and 0.1–0.3 mm in thickness. Electrical resistances were measured using a standard digital multimeter [UT56, Uni-Trend Group Limited (Shenzhen) Co., Ltd., Shenzhen, China]. Based on the data obtained, the electrical resistivities (ρ_v) were calculated using Eq. (1) below, where ρ_v is volume resistivity ($\Omega \cdot$cm), R is electrical resistance (Ω), S is the area of the adhesive layer (cm^2), and L is the thickness of the electroconductive adhesive layer (cm). The electrical conductivity, G (5/cm) can be obtained using Eq. (2).

$$\rho_v = R \times S/L \tag{1}$$

$$G = \frac{1}{\rho_v} \tag{2}$$

2.2.5. Measurement of Lap-Shear Strengths of Adhesive Joints

The lap-shear strengths of adhesive joints were measured by tensile testing of single overlapped adhesive joints situated between two aluminum strips with dimensions of 60 × 10 × 2 mm using a 5-KN universal testing machine (T-20A, Shenzhen Ruige Instrument Co., Ltd., Shenzhen, China) at a constant cross-head speed of 5 mm/min. The overlap area of aluminum was about 3 cm^2. Five groups of samples were measured and the measured data were statistically evaluated using computer software.

2.2.6. Measurement of Thermal Properties

Thermal degradation behaviour of the electroconductive adhesives was investigated using TGA (Pyris 1, Perkin-Elmer, Norwalk, CT, USA) from 50 to 600°C with a heating rate of 8°C/min under high purity nitrogen flow of 20 mL/min. The measurements were conducted on 2–5 mg samples. The plots of weight loss *versus* temperatures were recorded.

2.2.7. Infrared Spectroscopy (FTIR)

The formation of PU/EP IPNs was detected by Fourier transform infrared spectroscopy (FT-IR, E360, Nicolet Co., Ltd., USA). The samples of PU, EP, and PU/EP IPNs were measured before curing. Sample spectra were subtracted by the background collected before each sample.

2.2.8. Scanning Electron Microscopy (SEM)

Samples were frozen in liquid nitrogen and fractured with a sharp blow to obtain the fracture surfaces. Due to the electrically conducting capability of samples, the fractured surfaces were not sputtered with gold. Morphology of the fractured surfaces of the samples was observed using a scanning electron microscope Quanta-200 (FEI Co., Ltd., USA) with a device resolution of 10 nm at a vacuum of 0.83 Torr.

3. RESULTS AND DISCUSSION

3.1. Measurement of Polyurethane-Epoxy IPNs

3.1.1. Adhesive Mechanical Properties of Polyurethane-Epoxy IPNs

Figure 1 reveals the influence of EP content on adhesive mechanical properties of PU/EP IPN adhesives. The weight ratios of PU/EP are 100/0, 90/10, 80/20, 70/30, and 60/40. As shown in Fig. 1, with the increase of EP content from 0 to 20%, the lap-shear strength of PU/EP IPN adhesives increases from 2.10 to 4.32 MPa; however, it decreased as EP content continued to increase. Thus, the maximum lap-shear strength of PU/EP IPN adhesives appeared at about 20% of EP content. The relationship of the adhesive properties with respect

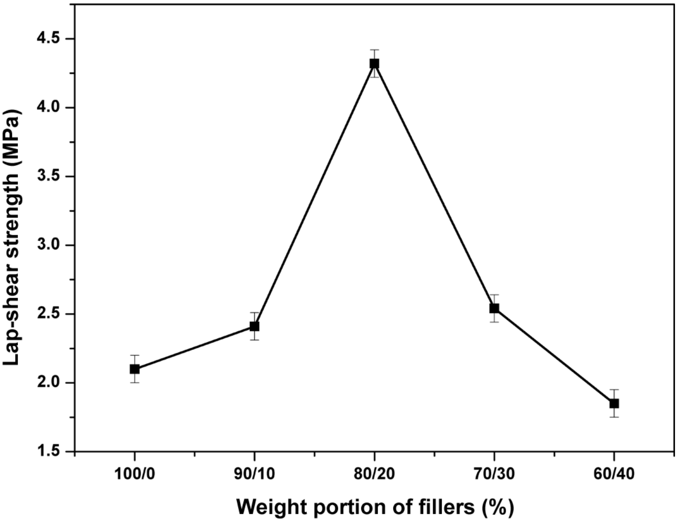


FIGURE 1 Variations of PU/EP IPNs lap-shear strength with different EP contents.

to the weight ratio of PU to EP showed that the formation of the polyurethane-epoxy network might affect their lap-shear strength in different ways. The incorporation of a small amount of the EP into the PU matrix could increase their shear strength because of intermolecular covalent chemical bonding and possible permanent interpenetration, leading to entanglement from the synergistic effect between the PU and EP resins. However, further increase of EP content (more than 20%) might decrease miscibility of the constituents of the PU/EP IPN adhesives, leading to phase separation between PU and EP. This eventually deteriorates the adhesives mechanical properties.

3.1.2. FTIR Spectroscopic Analysis

Figure 2 gives the Fourier transform infrared (FT-IR) spectra of the pure PU, crosslinked PU/EP (80/20), and pure EP before solidification. Peaks at 2900–3000 cm^{-1} are attributed to stretching of C–H bonds. According to the characteristics of EP and PU, we can use the intensity of the IR absorption of the hydroxyl groups –OH (3505 cm^{-1}) as a qualitative indication of the completion of the reaction between isocyanate groups (-NCO) of the PU prepolymer and the hydroxyl groups (-OH) of the epoxy resin. By monitoring the absorption strength at 3505 cm^{-1}, we can monitor the conversion extent of EP. Disappearance of the absorption at 3505 cm^{-1} for the hydroxyl group would indicate

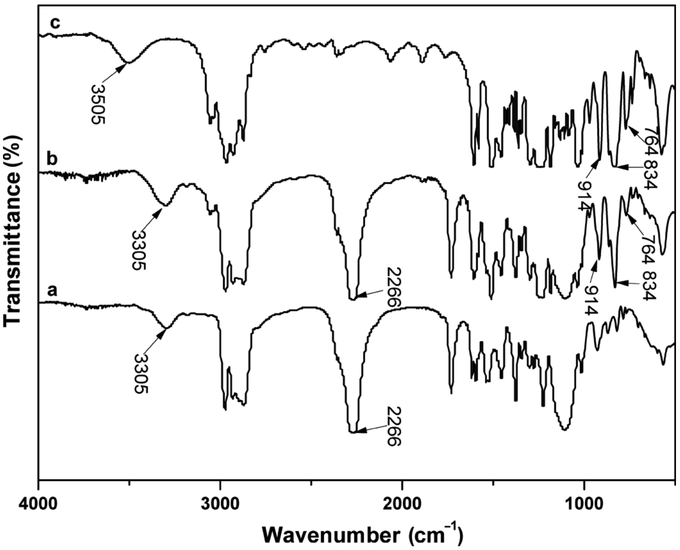


FIGURE 2 FTIR spectra of (a) pure PU, (b) PU/20EP IPNs, and (c) pure EP.

the complete conversion of the hydroxyl groups in EP as shown in Fig. 2. The above analysis can partially confirm the attainment of the PU/20EP IPNs. Besides the above comparison and analysis, the variation of the absorption band at 914 cm^{-1} is also usually used for quantitative analysis of EP [14]. The adsorption bands attributed to asymmetric stretch and symmetric stretch vibrations of epoxy substrates appeared at 914, 834, and 764 cm^{-1} in Fig. 2. Since the phenyl hydrogens were not involved in the reaction process, the absorption band at 834 cm^{-1}, attributed to out of plane deformation vibration of two adjacent phenyl hydrogens, did not change its absorption strength, which can be used as a standard. Comparing the characteristic peak at 914 cm^{-1} with that at 834 cm^{-1}, the slightly weakened absorption strength at 914 cm^{-1} implies the increasing crosslinking extent of PU by EP. From these two points of evidence, the FT-IR analysis indicates the formation of PU/20EP IPNs.

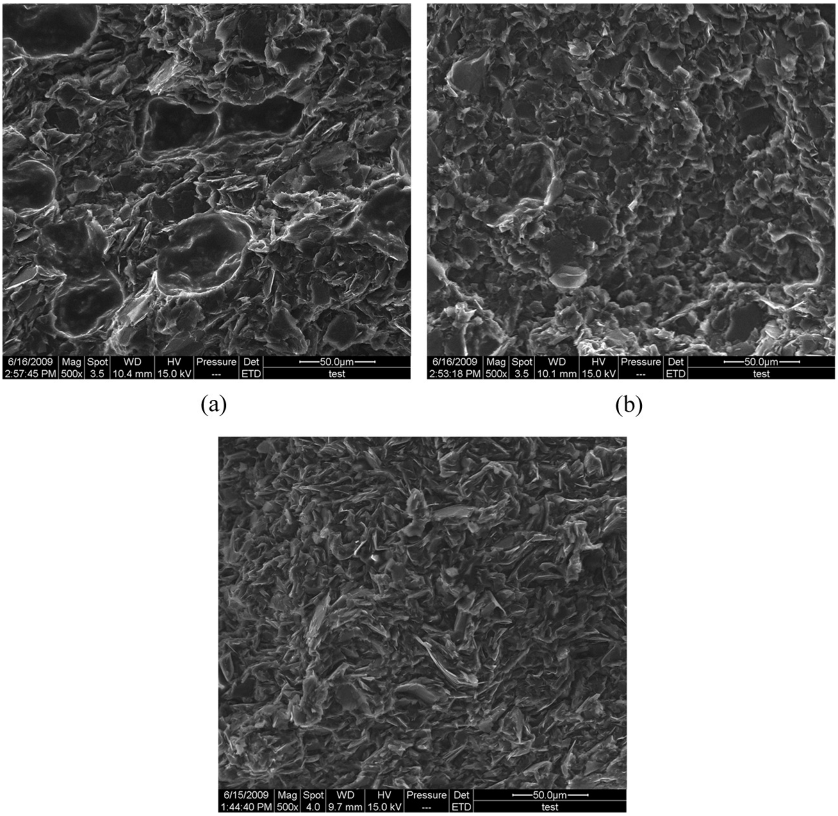
3.2. Electroconductive Adhesives

3.2.1. Microstructure of Electroconductive Adhesives

Figures 3(a–c) show the scanning electron micrographs (SEM) of NanoG/PU, NanoG/PU/20EP IPNs, and NanoG/EP electroconductive adhesives each with 15 wt% fillers. As shown in Fig. 3(a), the fractured surface of the NanoG/PU electroconductive adhesive was rough and porous. The formation of the pores in the matrix was likely triggered by the carbon dioxide generated by the reaction of the active –NCO groups of the PU prepolymer with the moisture in the air [15]. Apparently, the roughness and porosity of the adhesive's fractured surface decreased as the epoxy content was increased as shown in Fig. 3(b). The trend suggests that EP dispersed very well within the PU and formed covalent chemical bonding. The resulting permanent interpenetration and entanglement between the PU segments may effectively exclude the moisture. Thus, epoxy resin plays an important role both in ameliorating the morphological properties of the PU and possibly in decreasing its moisture susceptibility.

3.2.2. Electrical Conductivity

Table 3 shows the variations of electrical conductivities *versus* NanoG concentration for the individual electroconductive adhesives. Electrical conductivity for the adhesives increases with NanoG concentration and addition of NanoG significantly improved the conductivities of all the investigated adhesives. As shown in Table 3, NanoG/PU, NanoG/PU/20EP IPN, and NanoG/EP electroconductive adhesives containing more than 9, 6, and 3 wt% NanoG, respectively,



(a) (b)

(c)

FIGURE 3 Scanning electron micrographs of electroconductive adhesives: (a) NanoG/PU, (b) NanoG/PU/20EP IPNs, and (c) NanoG/EP [500×].

TABLE 3 The Volume Conductivities *versus* NanoG Content for NanoG/PU, NanoG/PU/20EP IPNs, and NanoG/EP Electroconductive Adhesives

NanoG wt%	NanoG/PU S/cm	NanoG/PU/20EP IPNs S/cm	NanoG/EP S/cm
1	–	–	–
3	–	–	0.228
6	–	0.152	0.414
9	0.011	0.474	1.251
12	0.027	0.631	2.197
15	0.035	0.932	3.381

have already shown electrical conductivities higher than the baseline adhesive. The electrical conductivities continued to increase with the increased content of NanoG, and eventually reached 0.035, 0.932, and 3.381 S/cm for the NanoG/PU, NanoG/PU/20EP IPN, and NanoG/EP electroconductive adhesives, respectively, with 15 wt% NanoG. The improvement of the conducting capability can be attributed to the formation of conducting networks *via* dispersion of graphite nanosheets within the adhesives. Among the three kinds of adhesives listed in Table 3, the NanoG/EP electroconductive adhesives exhibited the highest electrical conductivity. A possible reason for this behavior may be the good compatibility of epoxy resin with NanoG and the regular arrangement of NanoG in the epoxy resin, as also indicated in the SEM image in Fig. 3(c). On the contrary, NanoG/PU electroconductive adhesives showed lower electrical conductivities than two other adhesives. The poor electrically conducting performance of the NanoG/PU composites may be attributed to inhomogeneous distribution of NanoG in PU matrix. This might be from large amounts of pores generated by carbon dioxide produced by the reaction of the active –NCO groups of the PU prepolymer with the moisture in the air, as also suggested in the SEM image in Fig. 3(a). By comparison, we can find that of the electrically conducting capability of NanoG/PU/20EP IPN electroconductive adhesives falls between that of the NanoG/PU electroconductive adhesives and NanoG/EP electroconductive adhesives. The incorporation of epoxy resin to form the PU/EP IPN adhesives could reduce the amount of pores so as to improve the electrically conducting capabilities of PU/20EP IPN adhesives, which could be proved by morphological examination in Fig. 3(b). Thus, the epoxy resin plays an important role in enhancing the electrical conduction of PU/20EP IPN adhesives.

3.2.3. Adhesive Properties of Electroconductive Adhesives

The adhesive joints were prepared by overlapping two aluminum strips bonded together using the individual adhesives prepared. Figure 4 shows variations of lap-shear strengths of NanoG/PU, NanoG/PU/20EP IPNs, and NanoG/EP electroconductive adhesives with weight % of NanoG. As shown in Fig. 4, the increase in lap-shear strength with an increase in filler content in the whole concentration range was observed in NanoG/PU electroconductive adhesives, *i.e.*, from 2.10 MPa for the pure polyurethane to 3.55 MPa when filled with 15 wt% of NanoG. On the contrary, the lap-shear strength of the adhesive joint decreases with an increase in filler content in the whole concentration range for NanoG/EP electroconductive adhesives, *i.e.*, from 19.28 MPa for the pure epoxy to 7.22 MPa when filled with

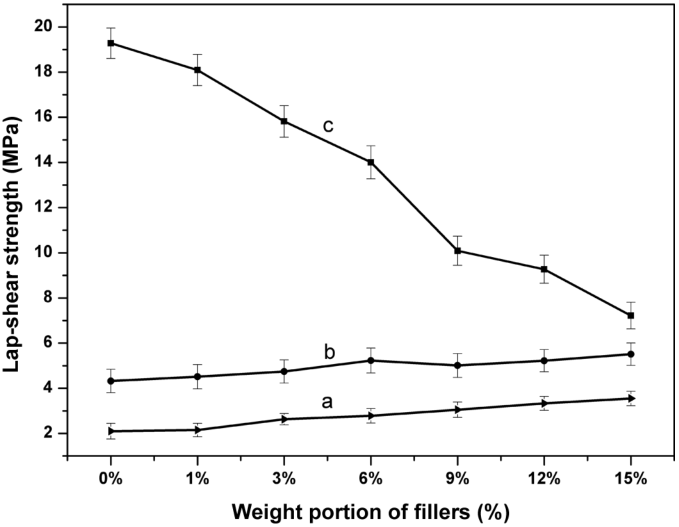


FIGURE 4 Relationship between lap-shear strength and the content of conductive filler for the matrices: (a) pure PU, (b) PU/20EP IPNs, and (c) pure EP.

15 wt% of NanoG. The lap-shear strength of the adhesive joint of NanoG/PU/20EP IPN electroconductive adhesive increases when increasing filler content, *i.e.*, from 4.32 MPa for the pure PU/20EP IPNs adhesive to 5.51 MPa when filled with 15 wt% of NanoG. From the comparison, it is clear that the NanoG/PU/EP IPNs perform better than the polyurethane-based system due to their modest cross-linking density with permanent physical entanglements and intermolecular covalent chemical bonding. This confirms the superiority of the IPNs over that of its constituent polyurethane polymer. Furthermore, that NanoG/PU/20EP IPNs electroconductive adhesives exhibited persistently higher lap-shear strength than NanoG/PU electroconductive adhesives suggests that NanoG plays an important role in enhancing the strength properties of adhesives with PU apart from increasing their electrically conducting capabilities.

3.2.4. Thermal Performance of the Electroconductive Adhesives

Figures 5 and 6 show thermogravimetry (TG) and derivative thermogravimetry (DTG) curves of the NanoG/PU, NanoG/PU/20EP IPN and NanoG/EP electroconductive adhesives with 9 wt% fillers. Seen from Figures 5 and 6 show that the NanoG/PU adhesives degraded in two steps. The first step of degradation occurred at

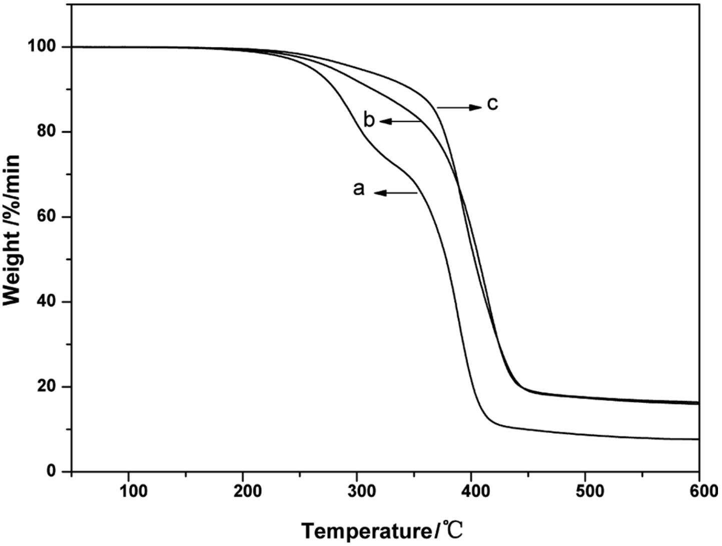


FIGURE 5 TG curves of electroconductive adhesives incorporating 9 wt% of NanoG for (a) PU, (b) PU/20EP IPNs, and (c) EP.

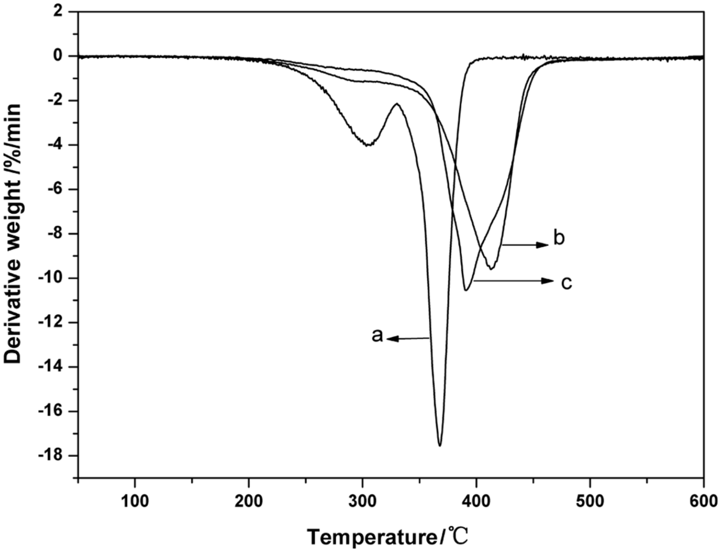


FIGURE 6 DTG curves of electroconductive adhesives incorporating 9 wt% of NanoG for (a) PU, (b) PU/20EP IPNs, and (c) EP.

temperatures ranging from around 230 to 330°C, and the second step of degradation occurred at temperatures ranging from 330 to 402°C. The first degradation is likely the PU decomposition. By comparison, the NanoG/PU/20EP electroconductive adhesives degraded in one apparent step with a weight loss at temperatures ranging from around 230 to 500°C. The weight loss rate at 230 to 340°C has slowed; that is to say, the incorporation of EP slowed the first step of PU thermal degradation. Moreover, the temperatures for maximum rate of weight loss for NanoG/PU, NanoG/PU/20EP IPN, and NanoG/EP electroconductive adhesives were 368.1, 423.8, and 392.1°C, respectively. For the NanoG/PU/20EP IPNs electroconductive adhesive, the temperature of maximum weight loss rate shifts to a higher temperature compared with that of NanoG/PU and NanoG/EP electroconductive adhesives. The shift to the higher temperature of thermal decomposition for NanoG/PU/20EP IPNs electroconductive adhesive indicated an enhancement of its thermal stability. This can also be explained by the fact that the incorporation of epoxy into the polyurethane resin resulted in forming covalent chemical bonding and permanent interpenetration and entanglement between the PU prepolymer and the epoxy networks can delay their degradation and, thus, elevate thermal degradation temperatures.

4. CONCLUSIONS

Polyurethane-epoxy IPNs were prepared and characterized as electroconductive adhesives comprised of NanoG and different matrices, *i.e.*, pure PU, PU/EP IPN, and pure EP. The lap-shear strength of the PU/EP IPN adhesives increased with the EP content from 0 to 20 wt%. Increase of EP content to 20 wt% reduced lap-shear strength of PU/EP IPN adhesives. The lap-shear strength of PU/EP IPN adhesives showed a maximum value at 20 wt% of EP. For all three kinds of adhesives, the electrical conductivity increased with an increase in NanoG content. The electrical conductivity of the PU/EP IPN electroconductive adhesives reached 0.932 S/cm when the content of the NanoG filler was 15 wt%. Addition of epoxy in the polyurethane-epoxy IPN adhesive also improved the thermal stability of the polymeric matrix so that the NanoG/PU/EP IPNs can be applied at comparatively high temperatures.

ACKNOWLEDGMENTS

The authors are grateful to Professor Li Bin (College of Science, Northeast Forestry University, Harbin, Heilongjiang, China) for his kind help and discussion during the research. This work was also supported by the Chinese College Students Innovative Projects (No:091022535).

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