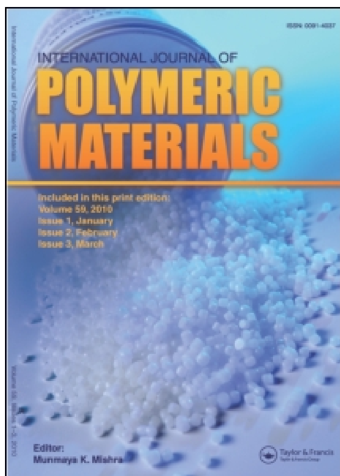
This article was downloaded by:
On: *19 January 2011*
Access details: *Access Details: Free Access*
Publisher *Taylor & Francis*
Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK




**International Journal of Polymeric Materials**
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713647664

# Adhesive and Thermal Properties of Epoxy-Imide Resins Obtained from Different Diimide-Diacids: Structure-Property Correlations

Ginu Abraham[a]; S. Packirisamy[a]; R. Ramaswamy[a]
[a] Polymers and Special Chemicals Division, Propellants and Special Chemicals Group, PCM Entity, Vikram Sarabhai Space Centre, Thiruvananthapuram, India

**To cite this Article** Abraham, Ginu , Packirisamy, S. and Ramaswamy, R.(2006) 'Adhesive and Thermal Properties of Epoxy-Imide Resins Obtained from Different Diimide-Diacids: Structure-Property Correlations', International Journal of Polymeric Materials, 55: 2, 121 — 134
**To link to this Article: DOI:** 10.1080/009140390916503
**URL:** http://dx.doi.org/10.1080/009140390916503

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: http://www.informaworld.com/terms-and-conditions-of-access.pdf

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

*International Journal of Polymeric Materials*, 55:121–134, 2006
Copyright © Taylor & Francis Inc.
ISSN: 0091-4037 print/1563-5333 online
DOI: 10.1080/009140390916503

Taylor & Francis
Taylor & Francis Group

# Adhesive and Thermal Properties of Epoxy-Imide Resins Obtained from Different Diimide-Diacids: Structure-Property Correlations

**Ginu Abraham**
**S. Packirisamy**
**R. Ramaswamy**
Polymers and Special Chemicals Division, Propellants and Special Chemicals Group, PCM Entity, Vikram Sarabhai Space Centre, Thiruvananthapuram, India

Downloaded At: 08:46 19 January 2011

*Different epoxy-imide resins were prepared through the reaction of epoxy resins namely, Araldite® GY 250 (difunctional; DGEBA) and Araldite® EPN 1138 (polyfunctional; novolac epoxy) with diimide-diacids such as 4,4′-bis(4-carboxyphthalimido)diphenylmethane, 4,4′-bis(4-carboxyphthalimido)diphenylsulphone, 3,3′-bis (4-carboxyphthalimido)diphenylsulphone, 4,4′-bis(4-carboxyphthalimido)diphenyl ether, 2,2-bis[4-(4-trimellitimidophenoxy)phenyl]propane, 1,13-bis(4-carboxyphthalimido)-4,7,10-trioxatridecane, and 1,6-bis(4-carboxyphthalimido)hexane in 1:1 carboxyl equivalent to epoxy equivalent ratio. The adhesive and thermal properties of these systems were evaluated to arrive at structure–property correlations. It is observed that epoxy-imides with more aliphatic moieties and (or) ether linkages give higher room temperature adhesive strength when compared to those with more aromatic moieties. But the latter systems give higher retention of room temperature adhesive strength at elevated temperatures when compared to that of the former systems. Thermogravimetric analysis shows that epoxy-imide systems are stable up to 360°C and char residues of GY 250- and EPN 1138-based systems at 800°C fall in the range of 14–38% and 23–48%, respectively, in nitrogen atmosphere. This has been attributed to the higher crosslinking possible for the latter systems due to the polyfunctional nature of EPN 1138.*

Received 16 November 2004; in final form 30 November 2004.
The authors are thankful to the Director, Vikram Sarabhai Space Centre for granting permission to publish this work. Thanks are due to Mr. K. S. Sastri and Dr. K. N. Ninan for their encouragement. Help received from the Analytical and Spectroscopy Division for the thermal, chemical, and spectral analysis of the samples, and from Dr. S. S. Bhagawan and his colleagues of the Propellant Engineering Division for adhesive strength measurements are gratefully acknowledged. One of the authors (G.A.) is thankful to the University Grants Commission, New Delhi for granting her a fellowship.
Address correspondence to Ginu Abraham, Laboratoire de chimie des Polymères Organiques (LCPO) CNRS (UMR 5629)-ENSCPB-Université Bordeaux 1 16, Avenue Pey Berland, 33607, Pessac, France. E-mail: abraham.ginu@gmail.com

**Keywords:** epoxy-imide resins, high temperature adhesives, stainless steel, lap shear, thermal analysis

Downloaded At: 08:46 19 January 2011

## INTRODUCTION

Epoxy resins modified with imide group or in other words, epoxy-imide resins have gained more attention in recent years, as they combine the versatility and ease of processability of epoxy resins [1–4] and the high temperature properties of imide groups [5–19]. The authors' laboratory has evaluated the adhesive strength and thermal properties of different epoxy-imide resins obtained by curing difunctional and polyfunctional epoxy resins with diimide-diacids [14–15,18–19]. While developing epoxy-imide adhesive systems attention was mainly focused on achieving maximum adhesive strength at room temperature and evaluating the percentage retention of room temperature adhesive strength at elevated temperatures for the optimum carboxyl equivalent (eqv) to epoxy eqv ratio. The optimum ratio for achieving maximum room temperature adhesive strength varied from one system to the other. It was noticed that the percentage retention of room temperature adhesive strength at elevated temperatures was influenced by the nature of diimide-diacids, nature of epoxy resins, aromatic content and imide content. Thus, it would be of interest to study the structure–property correlations of different epoxy-imide systems. In order to understand the dependence of adhesive and thermal properties on the structure of epoxy-imide resins, it is desirable to evaluate these properties for resins synthesized using the same carboxyl eqv to epoxy eqv ratio. Maximum amount of imide groups can be incorporated into epoxy-imide system without leaving behind any unreacted epoxy/carboxyl groups when the carboxyl eqv to epoxy eqv ratio is 1:1. Thus, in the present study, epoxy-imide resins were prepared by curing Araldite GY 250 (difunctional; DGEBA) and Araldite EPN 1138 (polyfunctional; novolac-epoxy) with diimide-diacids such as 4,4′-bis(4-carboxyphthalimido)-diphenylmethane, 4,4′-bis(4-carboxyphthalimido)diphenylmethane, 3,3′-bis(4-carboxyphthalimido) diphenylsulphone, 4,4′-bis(4-carboxyphthalimido)diphenylether, 2,2-bis[4-(4-trimellitimidophenoxy)phenyl]propane, 1,13-bis(4-carboxyphthalimido)-4,7,10-trioxatri-decane and 1,6-bis(4-carboxyphthalimido)hexane using 1:1 carboxyl eqv to epoxy eqv ratio. The adhesive and thermal properties of these epoxy-imide resins were evaluated and attempts were made to correlate the properties with the structure of these resins.

Downloaded At: 08:46 19 January 2011

# EXPERIMENTAL

## Materials

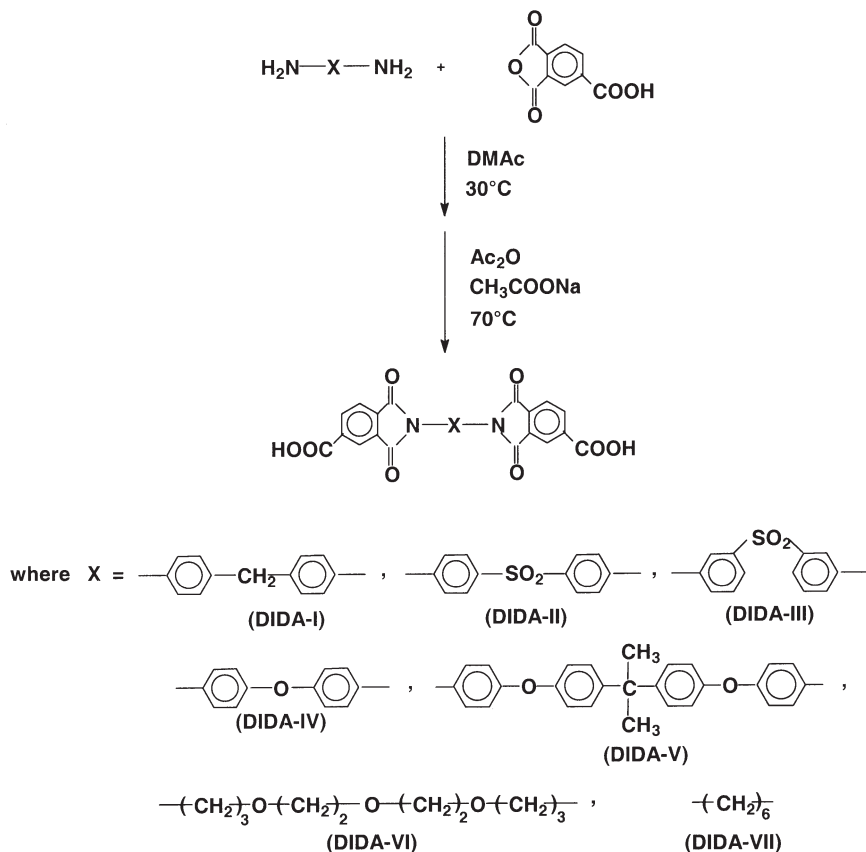
Trimellitic anhydride (Aldrich, Milwaukee, WI, USA) was purified by recrystallization from acetic anhydride. 4,4′-Diaminodiphenylmethane (DDM), 4,4′-diaminodiphenyl sulphone (p-DDS), 3,3′-diaminodiphenylsulphone (m-DDS) and 4,4′-diaminodiphenylether (DDE) (Merck, USA), 1,6-diaminohexane (Aldrich, Milwaukee, WI, USA) and 4,7,10-trioxa-1,13-tridecanediamine (Aldrich, Milwaukee, WI, USA) were used without further purification. 2,2-Bis[4-(4-aminophenoxy)phenyl]-propane was synthesized following the procedure described elsewhere [19]. Dimethyl acetamide (DMAc) (Spectrochem, Mumbai, India) was purified by distilling over phosphorous pentoxide (S.D. Fine Chemicals, Mumbai, India). Tetrahydrofuran (THF) (Qualigens, Mumbai, India) was refluxed over calcium hydride and distilled before use. Epoxy resins, Araldite GY 250 (DGEBA, epoxy value: 5.25 eqv/kg), and Araldite EPN 1138 (Novolac epoxy, epoxy value: 5.60 eqv/kg) manufactured and supplied by M/s. Hindustan Ciba-Geigy India Ltd., Mumbai, India were used as received.

## Synthesis of Diimide-Diacids

Diimide-diacids such as 4,4′-bis(4-carboxyphthalimido)diphenylmethane, 4,4′-bis(4-carboxyphthalimido)diphenylsulphone, 3,3′-bis (4-carboxyphthalimido)diphenylsulphone, 4,4′-bis(4-carboxyphthalimido)diphenylether, 2,2-bis[4-(4-trimellitimidophenoxy)phenyl]propane, 1,6-bis(4-carboxyphthalimido)hexane, and 1,13-bis(4-carboxyphthalimido)-4,7,10-trioxatridecane were synthesized through the reaction of DDM, P-DDS, m-DDS, DDE, 2,2-bis[4-(4-aminophenoxy)phenyl]propane, 1,6-diaminohexane, and 4,7,10-trioxa-1,13-tridecanediamine, respectively, with trimellitic anhydride in 1:2 mole ratio in DMAc followed by chemical imidization with acetic anhydride and sodium acetate at 70°C (Scheme 1) following the procedure reported elsewhere [14] and these diimide-diacids are referred to as DIDA-I, DIDA-II, DIDA-III, DIDA-IV, DIDA-V, DIDA-VI, and DIDA-VII, respectively. The diimide-diacids were characterized by IR, elemental analysis and acid value.

## Surface Preparation of Stainless-Steel Coupons

Surface preparation of stainless steel (AISI 304 SS cold roll) test pieces of dimensions 100 mm × 25 mm × 1.6 mm was carried out following the procedure described elsewhere [14].




**SCHEME 1** Synthesis of diimide-diacids.

Downloaded At: 08:46 19 January 2011

## Curing of Epoxy Resins with Diimide-Diacids

The cure reaction was followed by IR and DSC. The completion of the cure reaction is confirmed by the absence of the peak at 910 $cm^{-1}$ (corresponding to epoxy group) in the IR spectra of the cured epoxy-imide systems. The initial, maximum, and final cure temperatures ($T_{ic}$, $T_{maxc}$, and $T_{fc}$, respectively) of the epoxy-imide systems were determined from the DSC data. $T_{ic}$, $T_{maxc}$, and $T_{fc}$ of these systems fall in the temperature range 145–160°C, 170–200°C, and 230–250°C, respectively.

## Adhesive Formulation

Diimide-diacid and epoxy resin were taken in a pestle and mortar in 1:1 carboxyl eqv to epoxy eqv ratio and to this mixture the required

amount of tetrahydrofuran (2 ml for every 5 g of GY 250—diimide-diacid mixture and 5 ml for every 5 g of EPN 1138–diimide-diacid mixture) was added and thoroughly mixed. This mixture was applied on cleaned stainless-steel coupons and the coupons were dried in an air-oven at 100°C for 5 min. The coupons were then bonded in such a way that the bonded area was approximately 3 $cm^2$. The bonded coupons were kept in an air oven under a contact pressure of 3.3 $kg/cm^2$ applied by a lever press to facilitate the formation of a uniform thin adhesive layer. The temperature of the oven was then raised to 150°C over a period of 30 min and maintained at this temperature for 30 min. The temperature of the oven was further raised to 180°C over a period of 15 min and maintained at this temperature for 1 h and finally the oven temperature was raised to 250°C over a period of 15 min and the specimens were kept at this temperature for 30 min. The oven was allowed to cool to room temperature before the coupons were removed.

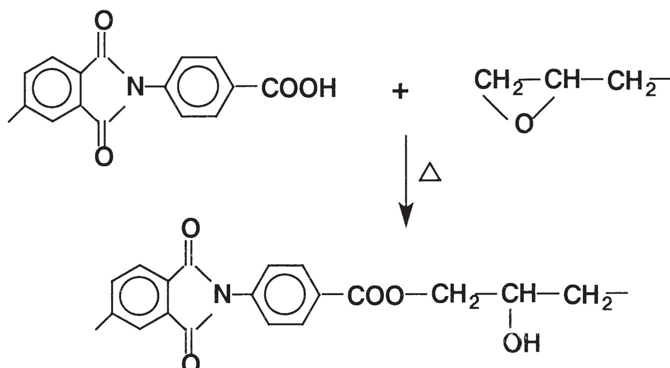
Downloaded At: 08:46 19 January 2011

## Measurements

Viscosity of Araldite® GY 250 and Araldite® EPN 1138 was measured using a Brookfield viscometer (Model: HBTD). Viscosity at 30°C of Araldite® GY 250 was measured using spindle 4 at 50 rpm and that of Araldite® EPN 1138 was measured using spindle 7 at 2.5 rpm. Thermograms of epoxy-imide resins were recorded with a DuPont 900 thermal analyzer in conjunction with a 951 thermogravimetric analyzer at a heating rate of 10°C/min in nitrogen atmosphere. The tensile lap shear strength of bonded stainless-steel coupons was measured using a Universal Testing Machine (Instron Model No. 4549) at a crosshead speed of 10 mm/min. The tensile lap shear strength at 150°C was measured after equilibrating the test specimens at the required temperature for a period of 10 min in a hot chamber attached to the Instron. To obtain the lap shear strength value at a particular temperature, five bonded coupons were tested. The lap shear strength value reported is an average of five measurements, which fell in the range of $\pm 1$ MPa.

# RESULTS AND DISCUSSION

Curing of epoxy resins, GY 250 and EPN 1138 with diimide-diacids proceeds through carboxyl-epoxy addition reaction at elevated temperatures as shown in Scheme 2 and the structures of epoxy-imide resins, obtained for carboxyl eqv to epoxy eqv ratio 1:1, are shown in Figure 1. The epoxy-imide resins obtained from GY 250 are linear




**SCHEME 2** Curing of epoxy resin with diimide-diacid.

polymers whereas the ones formed from EPN 1138 are crosslinked polymers resulting from the polyfunctional nature of EPN 1138.

Downloaded At: 08:46 19 January 2011

## Adhesive Properties

Adhesive lap shear strength at room temperature and at 150°C of GY 250-DIDA and EPN 1138-DIDA systems was evaluated and the results are compared in Figures 2 and 3, respectively. Percentage retention of room temperature adhesive strength at 150°C of GY 250-DIDA and EPN 1138-DIDA systems are given in Table 1.

### *Adhesive Properties of Araldite GY 250-DIDA Systems*

It is observed that the room temperature adhesive strength decreases in the following order: GY 250–DIDA-VI > GY 250–DIDA-VII > GY 250–DIDA-V = GY 250–DIDA-IV > GY 250–DIDA-I > GY 250–DIDA-III > GY 250–DIDA-II. The highest room temperature adhesive strength of 31 MPa is obtained for GY 250–DIDA-VI system. This is attributed to the presence of both aliphatic and ether linkages present in DIDA-VI. GY 250–DIDA-VII gives the second highest adhesive strength because it contains only aliphatic linkages and does not have ether linkages as in the case of DIDA-VI. Among the other epoxy-imide resins that are obtained from fully aromatic diimide-diacids, the ones that contain ether linkages give higher room temperature adhesive strength. This may be due to the flexibility imparted to the system by ether linkages. The epoxy-imide resins that are made from sulphone linkage containing diimide-diacid give the lowest room temperature adhesive strength and this may be due to presence of relatively rigid $-SO_2-$ linkages.

Downloaded At: 08:46 19 January 2011

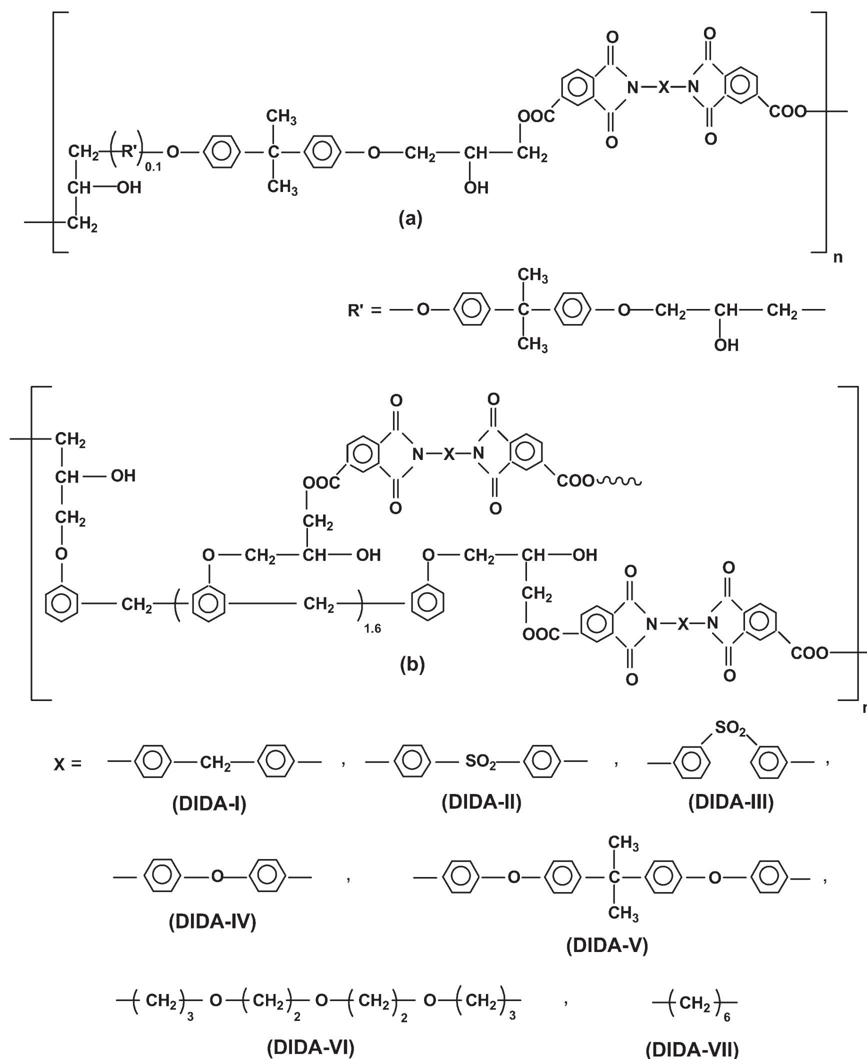



**FIGURE 1** Structures of different epoxy-imide resins: (a) GY 250-DIDAs and (b) EPN 1138-DIDAs.

The adhesive strength at 150°C of GY 250–based systems decreases in the following order: GY 250–DIDA-IV > GY 250–DIDA-III > GY 250–DIDA-II > GY 250–DIDA-V > GY 250–DIDA-I > GY 250–DIDA-VII > GY 250–DIDA-VI. It is interesting to note that GY 250–DIDA-VI system that gives the highest room temperature adhesive strength

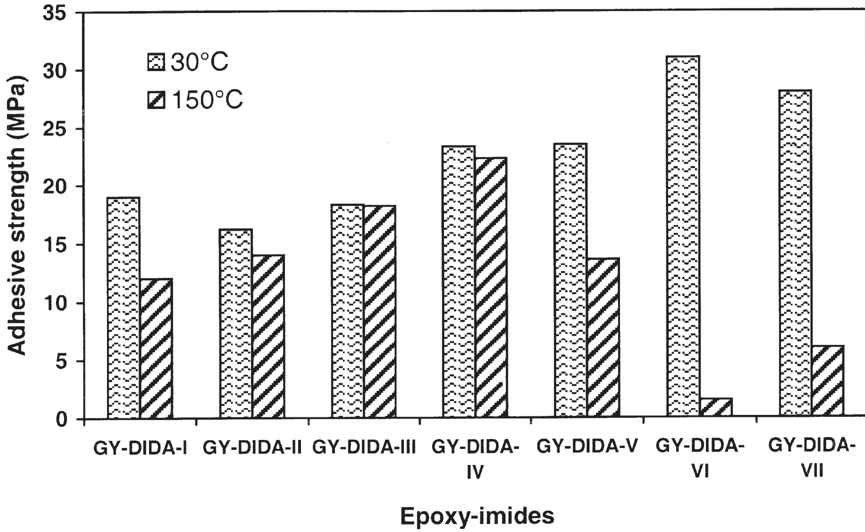



**FIGURE 2** Adhesive lap shear strength of different GY 250-DIDA systems at room temperature and at 150°C.

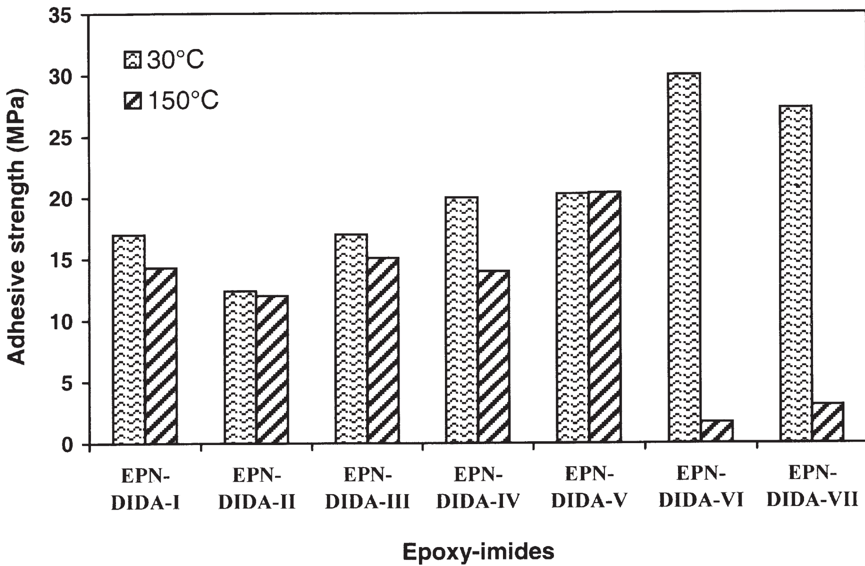



**FIGURE 3** Adhesive lap shear strength of different EPN 1138-DIDA systems at room temperature and at 150°C.

Downloaded At: 08:46 19 January 2011

**TABLE 1** Percentage Retention of Room Temperature Adhesive Strength at 150°C of GY 250/EPN 1138–DIDA systems

| | % Retention | |
|---|---|---|
| Diimide-diacids | GY 250 | EPN 1138 |
| DIDA-I | 63 | 84 |
| DIDA-II | 86 | 97 |
| DIDA-III | 99.5 | 89 |
| DIDA-IV | 96 | 70 |
| DIDA-V | 58 | 105 |
| DIDA-VI | 5 | 6 |
| DIDA-VII | 21 | 11 |

Downloaded At: 08:46 19 January 2011

gives the lowest adhesive strength at 150°C. This is attributed to the presence of flexible $-CH_2-CH_2-O-CH_2-CH_2-O-$ linkages. The second lowest adhesive strength is for the one derived from DIDA-VII containing methylene ($-CH_2-$) linkages. Although both DIDA-IV and DIDA-V contain ether linkages, epoxy-imide based on the former gives higher adhesive strength at elevated temperature when compared to that of the latter. In DIDA-V aliphatic linkage of the type $-C(CH_3)_2-$, similar to the one present in GY 250, is present and this may cause secondary relaxation that in turn would reduce the high temperature property of the system. Even though the room temperature adhesive strength is poor for DIDA-II– and DIDA-III–based epoxy-imides, they exhibit better retention of their room temperature adhesive strength at 150°C. This may be due to the presence of sulphone linkages [6,14]. Epoxy-imide systems derived from aliphatic diimide-diacids give very poor adhesive strength at elevated temperature. This is attributed to the presence of thermally less stable aliphatic linkages in the system.

## Adhesive Properties of Araldite EPN 1138-DIDA Systems

In general, the room temperature adhesive strength values of EPN 1138-based systems are lower than those of the GY 250-based systems. This is attributed to the brittleness of EPN 1138–based systems caused by the polyfunctional nature of EPN 1138. The room temperature adhesive strength of epoxy-imide resins based on DIDAs decreases in the following order: EPN 1138–DIDA-VI > EPN 1138–DIDA-VII > EPN 1138–DIDA-IV = EPN 1138–DIDA-V > EPN 1138–DIDA-I = EPN 1138–DIDA-III > EPN 1138–DIDA–II.

As observed in the case of GY 250–based systems, for EPN 1138–based systems also, aliphatic diimide-diacids give maximum room temperature adhesive strength. Aliphatic linkages impart flexibility to the system thereby improving the room temperature adhesive strength of EPN 1138–based systems. Among the epoxy-imide resins based on diimide-diacids obtained from aromatic diamines, DIDA-IV– and DIDA-V–based systems give the maximum adhesive strength (≈20 MPa). This is attributed to the presence of flexible ether linkages in these systems. Both DIDA-I– and DIDA-III–based systems give room temperature adhesive strength of 17 MPa. It is interesting to note that EPN 1138–DIDA-III–based system has higher adhesive strength when compared to that of EPN 1138–DIDA-II system (12.4 MPa). DIDA-II contains sulphone linkage in the *para* position and DIDA-III contains sulphone linkage in the *meta* position. The lower adhesive strength of EPN 1138-DIDA-II system is probably due to the brittleness caused by the rigidity of *para* sulphone linkage.

For EPN 1138–DIDA systems, the adhesive strength values at 150°C follow the trend: EPN 1138–DIDA-V $>$ EPN 1138–DIDA-III $>$ EPN 1138–DIDA-I $\cong$ EPN 1138–DIDA-IV $>$ EPN 1138–DIDA-II $>$ EPN 1138–DIDA-VII $>$ EPN 1138–DIDA-VI. The trend observed is somewhat different from that obtained for GY 250–based systems. The trend observed for adhesive strength at 150°C of EPN 1138–based systems can be better understood by considering the percentage retention of room temperature adhesive strength at 150°C. For GY 250–DIDA-IV, 96% retention of room temperature adhesive strength is observed at 150°C whereas only 70% retention of its room temperature adhesive strength is observed for EPN 1138–DIDA-IV. Earlier studies [14] on epoxy-imide resins obtained from imide-diacids [17] and diimide-diacids [14] suggest that EPN 1138–based systems exhibit higher retention of room temperature adhesive strength at elevated temperatures and this has been attributed to the higher crosslinking possible with EPN 1138–based system due to the polyfunctional nature of EPN 1138. In fact, the authors have observed [14] that the EPN 1138–DIDA-IV system is able to retain 100% of its room temperature adhesive strength at 150°C even for carboxyl eqv to epoxy eqv ratio of 0.75. For epoxy-imide systems [14,17] it is observed that the percentage retention of adhesive strength at elevated temperatures increases with the increase in aromatic content and imide content or in other words with the increase in carboxyl eqv to epoxy eqv ratio. Thus, for the system obtained by using carboxyl eqv to epoxy eqv ratio 1.0, the adhesive strength retention at 150°C is expected to be higher than that observed for the ratio 0.75. The lower percentage retention of adhesive strength of EPN 1138–DIDA-IV system when

Downloaded At: 08:46 19 January 2011

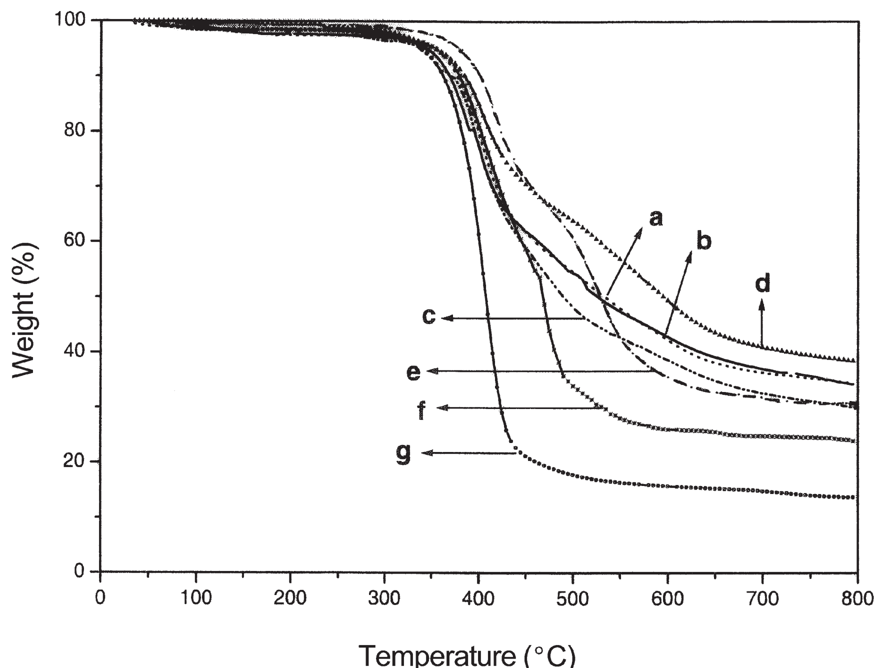



**FIGURE 4** Thermograms of different GY 250–DIDA systems (a) GY 250–DIDA-I, (b) GY 250–DIDA-II, (c) GY 250–DIDA-III, (d) GY 250–DIDA-IV, (e) GY 250–DIDA-V, (f) GY 250–DIDA-VI, and (g) GY 250–DIDA-VII.

Downloaded At: 08:46 19 January 2011

compared to that of GY 250–DIDA-IV system can be explained by considering the viscosity of GY 250 and EPN 1138 resins. EPN 1138 has viscosity of 1730 Pa·s whereas GY 250 has viscosity of 4 Pa·s. Hence, it is likely that EPN 1138–DIDA-IV system may be less homogeneous when compared to GY 250–DIDA-IV system. Thus, in the case of EPN 1138–DIDA-IV system, unreacted DIDA-IV or structural units rich in DIDA-IV moieties could be present at the adhesive interface, which acts as a weak boundary causing reduction in the percentage retention of room temperature adhesive strength at 150°C.

Interestingly, for the EPN 1138–DIDA-V system, the percentage retention of room temperature adhesive strength at 150°C is 105%, which is much higher than that observed for EPN 1138–DIDA-IV system (70%). As discussed earlier, THF is used in the adhesive formulation to improve the ease of application. DIDA-V is soluble in THF and hence, EPN 1138-DIDA-V is more homogeneous. On the other hand, DIDA-IV is insoluble in THF and for this reason, the EPN 1138–DIDA-IV system may not be as homogeneous as in the case

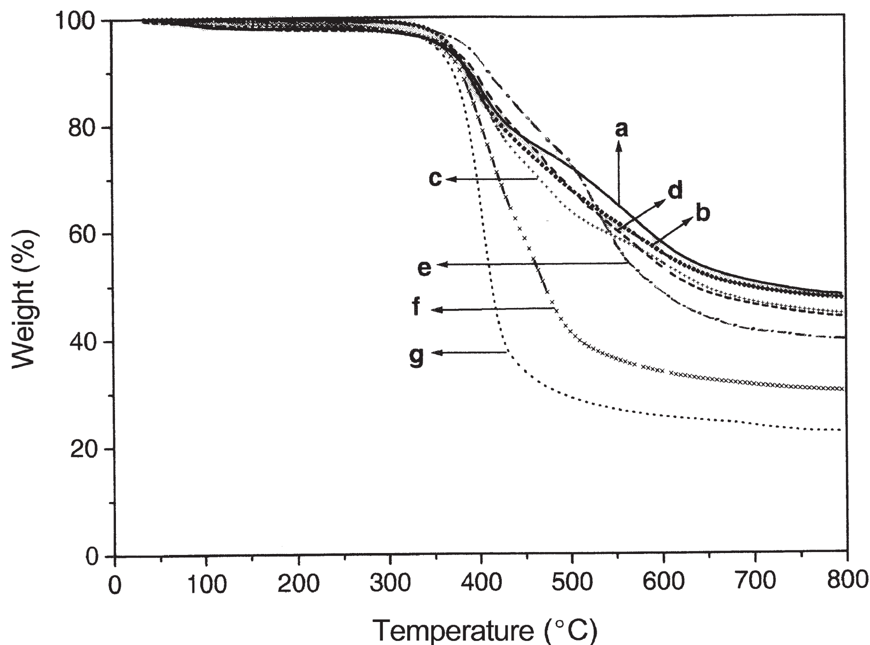



**FIGURE 5** Thermograms of different EPN 1138–DIDAs (a) EPN 1138–DIDA-I, (b) EPN 1138–DIDA-II, (c) EPN 1138–DIDA-III, (d) EPN 1138–DIDA-IV, (e) EPN 1138–DIDA-V, (f) EPN 1138–DIDA-VI, and (g) EPN 1138–DIDA-VII.

of EPN 1138–DIDA-V system. The explanation given above is also applicable to the EPN 1138–DIDA-III system. Among the other EPN 1138–DIDA systems, the one containing para sulphone linkage gives higher retention of its room temperature adhesive strength at 150°C. This may be due to the polar nature of $-SO_2-$ linkages. As in the case of GY 250–DIDA-VI/DIDA-VII systems, the corresponding ones made

**TABLE 2** Comparison of Thermal Properties of GY 250–DIDA systems

| System | $T_i$ (°C) | $T_{max}$ (°C) | $T_f$ (°C) | Char residue at 800°C (%) |
|---|---|---|---|---|
| GY 250–DIDA-I | 365 | 410 | 645 | 34.1 |
| GY 250–DIDA-II | 362 | 405 | 640 | 34.0 |
| GY 250–DIDA-III | 360 | 390 | 660 | 30.1 |
| GY 250–DIDA-IV | 360 | 400 | 660 | 38.3 |
| GY 250–DIDA-V | 376 | 420 | 580 | 31.0 |
| GY 250–DIDA-VI | 390 | 422 | 450 | 14.4 |
| GY 250–DIDA-VII | 375 | 411 | 485 | 24.0 |

Downloaded At: 08:46 19 January 2011

**TABLE 3** Comparison of Thermal Properties of EPN 1138–DIDA systems

| System | $T_i$ (°C) | $T_{max}$ (°C) | $T_f$ (°C) | Char residue at 800°C (%) |
|---|---|---|---|---|
| EPN 1138–DIDA-I | 360 | 400 | 650 | 48.0 |
| EPN 1138–DIDA-II | 360 | 390 | 670 | 47.4 |
| EPN 1138–DIDA-III | 375 | 405 | 690 | 44.5 |
| EPN 1138–DIDA-IV | 370 | 405 | 660 | 44.0 |
| EPN 1138–DIDA-V | 370 | 435 | 636 | 41.0 |
| EPN 1138–DIDA-VI | 390 | 419 | 460 | 23.0 |
| EPN 1138–DIDA-VII | 375 | 427 | 510 | 30.4 |

from EPN 1138 exhibit poor retention of adhesive strength at 150°C. This is due to the presence of thermally less stable aliphatic linkages.

## Thermal Stability of GY 250-/EPN 1138-DIDA Systems

The thermograms of GY 250-DIDA and EPN 1138-DIDA systems (in nitrogen atmosphere) are compared in Figures 4 and 5, respectively. The initial, maximum and final decomposition temperatures ($T_i$, $T_{max}$, and $T_f$ respectively) and char residues at 800°C of GY 250-DIDAs and EPN 1138-DIDAs are given in Tables 2 and 3, respectively. It is observed that the overall thermal stability and char residues are higher for EPN 1138–based systems than for GY 250–based systems. This is attributed to the higher crosslinking in EPN 1138–based systems arising from the polyfunctional nature of EPN 1138. It is noticed that the epoxy-imides synthesized from DIDA-I to DIDA-V give higher char residue when compared to the ones synthesized from DIDA-VI and DIDA-VII. This is attributed to the easy cleavage of aliphatic linkages present in DIDA-VI and DIDA-VII.

## CONCLUSIONS

In order to study the structure–property correlations, different types of epoxy-imides have been prepared by curing Araldite® GY 250 and Araldite® EPN 1138 with different diimide-diacids, and their adhesive and thermal properties have been compared. It is observed that the epoxy-imide resins based on diimide-diacids synthesized from aliphatic diamines give higher room temperature adhesive strength when compared to those synthesized from aromatic diamines. However, the adhesive strength at 150°C for the former systems is much lower than that of the latter systems and this has been attributed to the presence of aliphatic linkages in the former systems. The room temperature adhesive strength of epoxy-imide resins obtained from

Downloaded At: 08:46 19 January 2011

ether linkages containing diimide-diacids is higher than the other epoxy-imide resins. In general, the room temperature adhesive strength values of EPN 1138–based systems are lower than those of the GY 250–based systems. This is attributed to the brittleness of EPN 1138–based systems caused by the polyfunctional nature of EPN 1138. Among GY 250–DIDA systems, DIDA-IV–based one gives the maximum adhesive lap shear strength at 150°C. Among EPN 1138–DIDA systems, the DIDA-V–based system gives the maximum adhesive strength at 150°C. The overall thermal stability is higher for EPN 1138–based systems than for GY 250–based systems and this has been attributed to the higher crosslinking present in EPN 1138–based systems resulting from the polyfunctional nature of EPN 1138.

## REFERENCES

[1] Lee, H. and Neville, K. (1967). *Handbook of Epoxy Resins*, Mc Graw Hill, New York.
[2] May, C. A. and Tanaka, Y. (1973). *Epoxy Resins: Chemistry and Technology*, Marcel Dekker, New York.
[3] Houwink, R. and Solomon, G. (1965). *Adhesion and Adhesives*, Elsevier, Amsterdam.
[4] Skiest, I. (1978). *Handbook of Adhesives*, 2nd edition, VNR Company, New York.
[5] Gaw, K. O. and Kakimoto, M., *Adv. Polym. Sci.* **140**, 109 (1999).
[6] Ichino, T. and Hasuda, Y., *J. Appl. Polym. Sci.* **34**, 1667 (1987).
[7] Fuller, T. J., Marchetti, J. R., and Sanjana, Z. N. U.S. Patent 4, 760,105 (1988).
[8] Shau, M.-D. and Chin, W.-K., *J. Polym. Sci. Polym. Chem. Ed.* **31**, 1653 (1993).
[9] Gounder, R. N. and Geary, J. T. U.S. Patent 4, 467,100 (1984).
[10] Rao, B. S., *J. Polym. Sci. Polym. Lett. Ed.* **26**, 3 (1988).
[11] Serra, A., Cadiz, V., Martinez, P., and Mantecon, A., *Angew. Makromol. Chem.* **140**, 113 (1988).
[12] Chin, W.-K. and Shau, M.-D. (1996). In *Ency. Polym. Mat.* J. Salomone, Ed., CRC Press, Boca Raton, Vol. 5, p. 2210.
[13] Lee, C. J. U.S. Patent 4, 487,894 (1984).
[14] Adhinarayanan, K., Packirisamy, S., George, R. S., Rao, V. L., and Ramaswamy, R., *J. Appl. Polym. Sci.* **43**, 783 (1991).
[15] Adhinarayanan, K., Packirisamy, S., Suma, P. M., and Ramaswamy, R. (1991). In *Polymer Science, Contemporary Themes*. S. Sivaram, Ed., Tata McGraw-Hill, New Delhi, Vol. I, p. 76.
[16] Packirisamy, S., Adhinarayanan, K., Jameela, S. R., and Ramaswamy, R. (1991). In *Polymer Science, Contemporary Themes*. S. Sivaram, Ed., Tata McGraw-Hill, New Delhi, Vol. I, p. 150.
[17] Abraham, G., Packirisamy, S., Adhinarayanan, K., Feby, A. G., and Ramaswamy, R., *J. Appl. Polym. Sci.* **78**, 1729 (2000).
[18] Abraham, G. (2001). Ph.D. thesis, Mahatma Gandhi University, Kottayam, India.
[19] Abraham, G., Packirisamy, S., Vijayan, T. M., Ramaswamy, R., *J. Appl. Polym. Sci.* **88**, 1737 (2003).

Downloaded At: 08:46 19 January 2011