# Chemistry and properties of poly(arylene ether 1,3,4-oxadiazole)s and poly(arylene ether 1,2,4-triazole)s

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Poly(arylene ether)s containing 1,3,4-oxadiazole and 1,2,4-triazole units were prepared by the aromatic nucleophilic displacement reaction of bisphenol oxadiazole and bisphenol triazole compounds with activated aromatic dihalides. The polymers exhibited glass transition temperatures ($T_g$s) ranging from 182 to 242°C and several polymers exhibited melting transitions ($T_m$s) ranging from 265 to 390°C. Inherent viscosities ranged from 1.02 to 3.40 dl $g^{-1}$ indicating relatively high molecular weights. Thin films exhibited tensile strengths, moduli and elongations at 23°C of 90–110 MPa, 2.7–3.6 GPa and 4–7%, respectively. Titanium-to-titanium tensile shear specimens of a poly(arylene ether 1,3,4-oxadiazole) exhibited tensile shear strengths at 23 and 150°C of 22.1 and 17.9 MPa, respectively.

**(Keywords: poly(arylene ether); polyoxadiazoles; polytriazoles; high temperature polymers; adhesives; films)**

## INTRODUCTION

Poly(1,3,4-oxadiazole)s were first synthesized by the reaction of bis(tetrazole)s and diacid chlorides[1]. Since then several different reaction pathways have been developed to prepare these polymers[2–7]. The most popular route involved the preparation of a precursor polymer (i.e. polyhydrazide) by the reaction of a diacid chloride or derivative with a hydrazine or dihydrazine compound. This precursor polyhydrazide was subsequently cyclized to the poly(1,3,4-oxadiazole) by heating up to 300°C under vacuum or by heating in a high boiling dehydrating solvent such as sulphuric or polyphosphoric acid.

Poly(1,2,4-triazole)s were first synthesized by the reaction of bis(tetrazole)s with bis(imidoyl chloride)s[1]. Several different reaction pathways to prepare these polymers have since been established[2,5,6,8–10]. One of the more common methods to prepare poly(1,2,4-triazole)s involves reaching a precursor polyhydrazide with aniline in polyphosphoric acid at ~175°C. This synthetic approach leads to a poly(1,2,4-triazole) which contains a small amount of 1,3,4-oxadiazole.

As part of a programme on high performance/high temperature polymers for potential use as adhesives and composite matrices on aerospace vehicles, the preparation and evaluation of poly(arylene ether)s containing various heterocyclic units is in progress. The synthetic approach has involved the aromatic nucleophilic displacement reaction of novel bisphenol heterocyclic compounds with activated aromatic dihalides. The polymerization reactions are carried out in polar aprotic media using potassium carbonate at elevated temperatures under nitrogen. Heterocyclic units that have been incorporated into the backbone of poly(arylene ether)s using this approach include quinoxaline[11], phenylquinoxaline[11], imidazole[12–15], benzoxazole[16], benzimidazole[17], pyrazole[18], 1,3,4-oxadiazole[19] and 1,2,4-triazole[19]. Virtually any heterocyclic ring system can be used in this approach provided that it can survive the polymerization conditions. Heterocyclic units incorporated into the backbone of poly(arylene ether)s generally result in an increase in the glass transition temperature ($T_g$) and modulus over that of a poly(arylene ether) without heterocyclic units. The degree of increase varies from one system to another. Heterocyclic units such as imidazole[12–15], benzimidazole[17] and pyrazole[18] cause greater increases in properties than the other systems which do not contain hydrogen on a heteroatom in the ring.

## EXPERIMENTAL

### *Starting materials*

Diphenylsulphone (Aldrich Chemical Co.) was recrystallized from methanol, m.p. 122–123°C. 4,4'-Dichlorodiphenylsulphone (Aldrich Chemical Co.) was recrystallized from toluene, m.p. 147–149°C. 4,4'-Difluorobenzophenone (Chemical Dynamics Corp.) was recrystallized from ethanol, m.p. 104–105°C. Bis(4-fluorophenyl)phenyl phosphine oxide (American Hoescht

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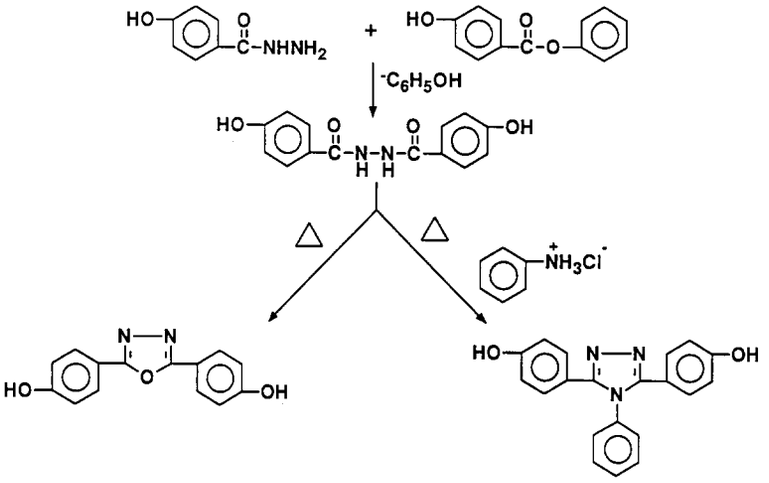


**Scheme 1**

Corp.) was recrystallized from ligroin, m.p. 126–128°C. 1,3- and 1,4-Bis(4-fluorobenzoyl)benzene were prepared as previously described[20], m.p. 178–179°C and 217–219°C, respectively. 4-Hydroxybenzoic hydrazide (Aldrich Chemical Co.) was used as received. Phenyl-4-hydroxybenzoate (K and K Laboratories) was recrystallized from toluene, m.p. 174.5–176.5°C.

*2,5-Bis(4-hydroxyphenyl)-1,3,4-oxadiazole*

The title compound was prepared as depicted in *Scheme 1* by the following procedure. Into a 250 ml three-neck round-bottomed flask equipped with a magnetic stirbar, nitrogen gas inlet, glass stopper and distillation head, were placed 4-hydroxybenzoic hydrazide (60.9 g, 0.4 mol) and phenyl-4-hydroxybenzoate (85.7 g, 0.4 mol). The mixture under nitrogen was heated to ~210°C by use of a Wood's metal bath. The solids melted and phenol began to evolve and was removed via the distillation head. After ~30 min the melt began to solidify. The temperature was subsequently increased to 300°C and held for 1 h under nitrogen. The mixture was allowed to cool and the tan solid was pulverized and stirred with methanol (200 ml). The solid was collected and dried at 150°C for 3 h in a forced air oven to give a white solid (56 g, 55% yield). The solid was recrystallized from a mixture of *N,N*-dimethylacetamide (DMAc, 225 ml) and water (105 ml) and after drying at 200°C for 8 h under vacuum, gave 46 g (45% yield) of white solid. The material exhibited a sharp endothermic peak at 347°C by differential thermal analysis (d.t.a.) (lit.[21] m.p. 338°C). Elemental analysis for $C_{14}H_{10}O_3N_2$. Calculated: C, 66.13%; H, 3.96%; N, 11.02%. Found: C, 66.17%; H, 4.05%; N, 11.12%.

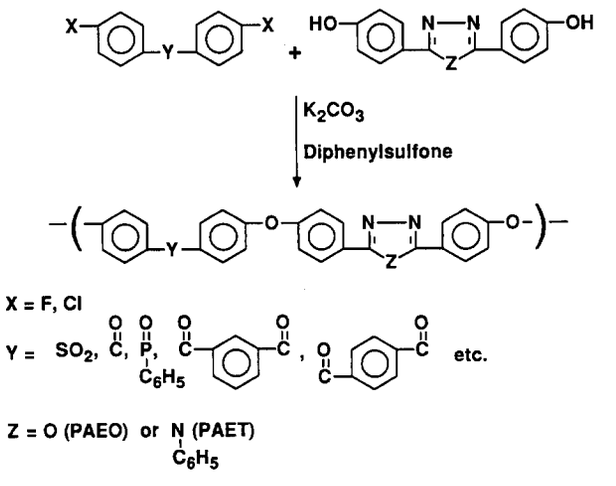
*3,5-Bis(4-hydroxyphenyl)-4-phenyl-1,2,4-triazole*

The title compound was prepared as depicted in *Scheme 1* by the following procedure. Into a 250 ml three-neck round-bottomed flask equipped with a magnetic stirbar, nitrogen gas inlet, glass stopper and distillation head, were placed 4-hydroxybenzoic hydrazide (60.9 g, 0.4 mol) and phenyl-4-hydroxybenzoate (85.7 g, 0.4 mol). The mixture under nitrogen was heated to ~210°C by use of a Wood's metal bath. The solids melted and phenol began to evolve and was removed via the distillation head. The temperature was maintained at 210°C for ~2 h and aniline hydrochloride (103.6 g, 0.8 mol) was subsequently added as a solid and the temperature was increased to 250°C and held for 2 h under nitrogen. The mixture was cooled and washed with methanol and after drying for 3 h at 150°C gave 66 g (50% yield) of white solid. The solid was recrystallized from DMAc (225 ml) and after drying for 8 h at 200°C under vacuum gave 50 g (37% yield) of white needles. The material exhibited a sharp endothermic peak at 420°C by d.t.a. Elemental analysis for $C_{20}H_{15}N_3O_2$. Calculated: C, 72.93%; H, 4.59%; N, 12.76%. Found: C, 72.97%; H, 4.63%; N, 12.84%.

*Polymers*

The following experimental procedure is representative of that used for the preparation of the poly(arylene ether 1,3,4-oxadiazole)s (PAEO) and poly(arylene ether 1,2,4-triazole)s (PAET) as depicted in *Scheme 2*. The only variable was the reaction temperature which ranged from 210 to 280°C depending on the solubility of the polymer.

Into a 500 ml three-neck round-bottomed flask equipped with a mechanical stirrer, thermometer, nitrogen gas inlet and reflux condenser, were placed 2,5-bis(4-hydroxyphenyl)-1,3,4-oxadiazole (19.068 g, 0.075 mol), diphenylsulphone (200 g, 20% solids) and pulverized anhydrous potassium carbonate (23.9 g, 0.17 mol, 15% excess). The mixture was heated to



**Scheme 2**

~180°C under nitrogen for ~0.5 h and 1,3-bis(4-fluorobenzoyl)benzene (24.167 g, 0.075 mol) was subsequently added. The temperature was increased to 210°C for 16 h under nitrogen. The viscous solution was cooled to ~175°C, diluted with *N*-methyl-2-pyrrolidone (NMP, 150 ml) and the mixture was precipitated into methanol/acetic acid mixture (20:1) in a high speed blender. The polymer was washed successively in hot water and hot methanol and dried for 8 h at 120°C under vacuum to give 39.4 g (98%) of white fibrous polymer. The polymer exhibited a $T_g$ of 182°C, a $T_m$ of 265°C and an inherent viscosity (0.5% solution in *m*-cresol at 25°C) of 1.53 dl $g^{-1}$.

*Films*

*m*-Cresol solutions of the polymers (15–20% solids) were centrifuged and deposited onto plate glass and dried to a tack-free form in a flowing dry air chamber. The films on glass were subsequently stage-dried in a forced air oven to a temperature ~50°C above the $T_g$ of the polymer and held there for 0.5 h. Thin film tensile properties were determined according to ASTM D882 using four specimens per test condition.

*Moulded specimens*

Powdered PAEO (~9 g) prepared from the bisphenol oxadiazole and 1,3-bis(4-fluorobenzoyl)benzene was compression moulded in a 32 $mm^2$ stainless steel mould by heating to 300°C under 2.1 MPa for 1 h. The unendcapped PAEO had an inherent viscosity ($\eta_{inh}$) of 1.53 dl $g^{-1}$ (0.5% solution in *m*-cresol at 25°C). Miniature compact tension specimens (15.7 mm × 15.7 mm × 9.5 mm thick) were machined from the moulding and subsequently tested to determine fracture toughness ($K_{Ic}$, critical stress intensity factor) according to ASTM E399 using four specimens per test. Critical strain energy release rate ($G_{Ic}$) was calculated using the mathematical relationship $G_{Ic} = (K_{Ic})^2/E$, where $E$ is the modulus of the material.

*Adhesive specimens*

Adhesive tape was prepared by multiple coating of 112 E glass with an A-1100 finish using *m*-cresol solution of the PAEO prepared from the bisphenol oxadiazole and 1,3-bis(4-fluorobenzoyl)benzene ($\eta_{inh}$ = 1.43 dl $g^{-1}$, *m*-cresol, 0.5%, 25°C) and subsequently dried after each coat. Titanium (Ti)–Ti tensile shear specimens with a Pasa-Jell 107 (Products Research and Chemical Corp.) were fabricated in a press by heating to 300°C under 1.4 MPa and holding for 1 h. Tensile shear strengths were determined according to ASTM D1102 using four specimens per test condition. Isothermal ageing was performed on unstressed adhesive specimens at 200°C in a circulating air oven.

*Other characterization*

D.t.a. was performed at a heating rate of 10°C $min^{-1}$ with the melting point taken at the endothermic peak temperature. Differential scanning calorimetry (d.s.c.) was performed at a heating rate of 20°C $min^{-1}$ with the $T_g$ taken at the inflection point of the $\Delta T$ *versus* temperature curve. Thermogravimetric analysis (t.g.a.) was performed on powder samples at a heating rate of 2.5°C $min^{-1}$ in air or nitrogen at a flow rate of 15 $cm^3$ $min^{-1}$. Inherent viscosities ($\eta_{inh}$) were performed on 0.5% solutions in *m*-cresol, chloroform or concentrated sulphuric acid at 25°C. Wide angle X-ray diffraction (WAXS) data were obtained on polymer powder, thin films and adhesive tape. The X-ray diffractometer was operated at 45 kV and 40 mA using a copper radiation source with a flat sample holder and a graphite monochromator. The density of 1 s counts was taken every 0.01° ($2\theta$) and was recorded on hard disc for the angular range of 10–40° ($2\theta$). An external $\alpha$ quartz standard was used in the goniometer alignment. Elemental analysis was performed by Galbraith Laboratories, Inc. (Knoxville, TN).

## RESULTS AND DISCUSSION

*Bisphenol compounds*

2,5-Bis(4-hydroxyphenyl)-1,3,4-oxadiazole and 3,5-bis(4-hydroxyphenyl)-4-phenyl-1,2,4-triazole were prepared in a one-pot synthesis as depicted in *Scheme 1* from a common intermediate [i.e. 1,2-bis(4-hydroxybenzoyl) hydrazine]. The 1,2-bis(4-hydroxybenzoyl) hydrazine was prepared from commercially available starting materials and was not isolated from the reaction mixture. The yields for the purified bisphenol oxadiazole and triazole were 45% and 37%, respectively. These yields can probably be improved considerably by optimizing the reaction conditions. The bisphenol compounds were white crystalline solids with high melting points and were relatively insoluble in most organic solvents.

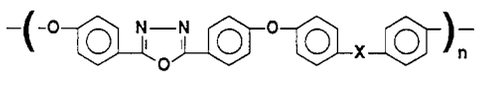
*Poly(arylene ether 1,3,4-oxadiazole)s*

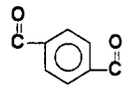
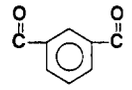
High molecular weight PAEOs were prepared as depicted in *Scheme 2* by the reaction of activated aromatic dihalides, the bisphenol oxadiazole and potassium carbonate in diphenylsulphone (15–20% solids) under nitrogen. The reaction mixtures were heated to 210–280°C for ~16 h. The reaction temperature was governed by the chemical structure of the polymer; some of the semicrystalline polymers required higher reaction temperatures to keep them in solution. Inherent viscosities of the PAEOs ranged from 1.02 to 1.71 dl $g^{-1}$, $T_g$s ranged from 182 to 242°C and $T_m$s ranged from 265 to 390°C (*Table 1*). The trend in the $T_g$s of the polymers in *Table 1* with phenylphosphine oxide > sulphone > carbonyl > terephthaloyl > isophthaloyl followed that found for other poly(arylene ether heterocyclic)s[12–16]. The polymers were soluble in *m*-cresol and/or concentrated sulphuric acid. Several of the as-isolated PAEOs which exhibited $T_m$s by d.s.c. were found to be semicrystalline by WAXS. A representative d.s.c. thermogram and a WAXS diffractogram for the same polymer are shown in *Figures 1* and *2*, respectively. Thin films of the PAEOs cast from *m*-cresol were amorphous. However, crystallinity could be induced into some films by annealing at elevated temperature. T.g.a. of the PAEOs showed no weight loss occurring below 300°C in air or nitrogen with a 5% weight loss occurring at ~440°C in air and at ~500°C nitrogen.

*Poly(arylene ether 1,2,4-triazole)s*

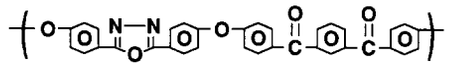
High molecular weight PAETs were prepared as depicted in *Scheme 2* by the reaction of activated aromatic dihalides, the bisphenol triazole and potassium carbonate in diphenylsulphone (15–20% solids) under

**Table 1** Polymer characterization (PAEOs)



| Polymer | $X$ | $\eta_{inh}$ (dl g$^{-1}$)[a] | $T_g$ (°C)[b] | $T_m$ (°C)[b] |
|---|---|---|---|---|
| P1 |  | 1.38 | 242 | – |
| P2 | $SO_2$ | 1.02 | 226 | – |
| P3 |  | 1.57[c] | 205 | 325 |
| P4 |  | 1.71[c] | 201 | 390 |
| P5 |  | 1.53 | 182 | 265 |

[a]Inherent viscosity determined on 0.5% solutions in *m*-cresol at 25°C
[b]Differential scanning calorimetry at a heating rate of 20°C min$^{-1}$
[c]Inherent viscosity determined on 0.5% solutions in concentrated sulphuric acid at 25°C



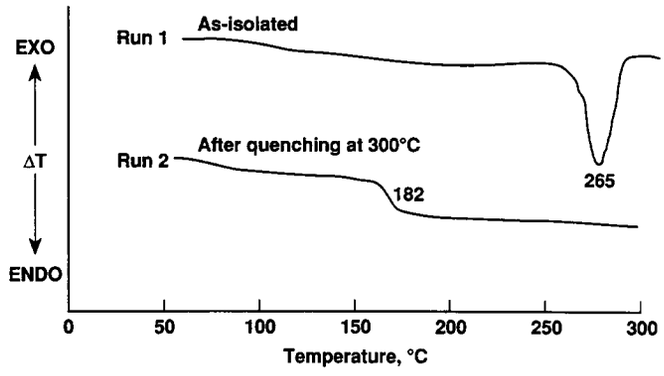
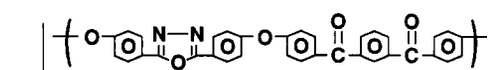



**Figure 1** Differential scanning calorimetric curve of PAEO powder in static air. Heating rate 20°C min$^{-1}$, sample size 4.3 mg, sensitivity 0.5 (mcal s$^{-1}$) in$^{-1}$



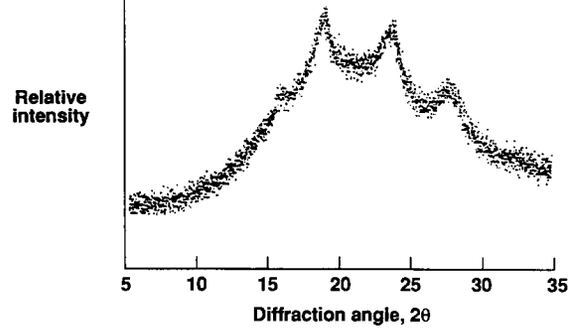


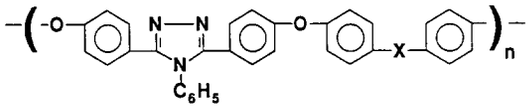

**Figure 2** X-ray diffractogram of as-isolated PAEO powder

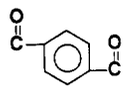
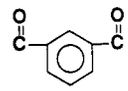
nitrogen. The reaction mixtures were heated to 210–280°C for ~16 h. One of the PAETs was semicrystalline and required a reaction temperature of ~280°C to keep it in solution. Inherent viscosities of the PAET ranged from 1.37 to 3.40 dl g$^{-1}$, $T_g$s ranged from 192 to 216°C and one polymer exhibited a $T_m$ of 377°C (*Table 2*). Although an $\eta_{inh}$ of 3.40 dl g$^{-1}$ is high, poly(1,2,4-triazole)s prepared via conventional synthesis have been reported[2] with viscosities of ~2.0 dl g$^{-1}$. The polymers were soluble in *m*-cresol and polymer P8 (*Table 2*) was soluble in chloroform. T.g.a. of the PAETs showed no weight loss occurring below 300°C in air or nitrogen with a 5% weight loss occurring at ~410°C in air and at ~475°C in nitrogen.

*Films*

Thin films of the PAEOs and PAETs were cast from *m*-cresol solutions and subsequently tested for tensile properties (*Table 3*). The films obtained from semicrystalline polymers were amorphous after casting and were tested without any post-treatment such as thermal annealing. The tensile strengths and moduli measured at room temperature ranged from 90 to 110 MPa and 2.7 to 3.6 GPa, respectively. Several of the films tested at 150°C exhibited good property retention, especially the tensile

**Table 2** Polymer characterization (PAETs)



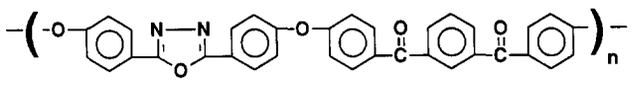
| Polymer | $X$ | $\eta_{inh}$ (dl g$^{-1}$)[a] | $T_g$ (°C)[b] | $T_m$ (°C)[b] |
|---|---|---|---|---|
| P6 |  | 2.94 | 207 | – |
| P7 |  | 3.40 | 216 | 377 |
| P8 |  | 1.37[c] | 192 | – |

[a]Inherent viscosity determined on 0.5% solutions in *m*-cresol at 25°C
[b]Differential scanning calorimetry at a heating rate of 20°C min$^{-1}$
[c]Inherent viscosity determined on 0.5% solutions in chloroform at 25°C

**Table 3** Thin film properties

| Polymer | Test temperature (°C) | Tensile strength (MPa) | Tensile modulus (GPa) | Elongation (%) |
|---|---|---|---|---|
| P1 | 23 | 93.1 | 3.09 | 6.6 |
| | 150 | 55.1 | 2.08 | 3.6 |
| P2 | 23 | 92.4 | 2.71 | 5.8 |
| | 150 | 53.8 | 1.89 | 4.3 |
| P5 | 23 | 114.4 | 3.55 | 4.4 |
| | 150 | 51.0 | 2.52 | 10.7 |
| P6 | 23 | 91.0 | 2.69 | 8.1 |
| | 150 | 55.1 | 1.91 | 9.2 |
| P7 | 23 | 87.6 | 2.72 | 7.8 |
| | 150 | 55.1 | 1.90 | 9.5 |
| P8 | 23 | 96.5 | 3.08 | 6.9 |
| | 150 | 46.9 | 2.38 | 5.4 |

**Table 4** Adhesive properties



$T_g$ = 182°C, $\eta_{inh}$ = 1.53 dl g$^{-1}$

| Test (°C) | Exposure | Ti–Ti tensile shear[a] strength (MPa) |
|---|---|---|
| 23 | None | 22.2 |
| 150 | None | 18.1 |
| 177 | None | 2.8 |
| 23 | 500 h at 200°C in air | 31.2 |
| 150 | 500 h at 200°C in air | 21.7 |
| 177 | 500 h at 200°C in air | 8.3 |
| 23 | 1000 h at 200°C in air | 34.1 |
| 150 | 1000 h at 200°C in air | 22.2 |
| 177 | 1000 h at 200°C in air | 7.6 |

[a]Processed at 300°C under 1.4 MPa for 1 h

modulus. For example, the tensile strength and modulus of polymer P5 (*Table 1*, $T_g$ 182°C) at 23°C were 114.4 MPa and 3.55 GPa and at 150°C were 51.0 MPa and 2.51 GPa, respectively. This material maintained 71% of its room temperature modulus when tested within 32°C of its $T_g$. Polymer P8 (*Table 2*) also exhibited a notable retention of room temperature tensile modulus when tested at 150°C. The tensile strengths and moduli of these polymers are somewhat higher than those of poly(arylene ether)s without heterocyclic units[20]. It is interesting to note that the polymers containing the isophthaloyl group in the backbone (P5 and P8, *Tables 1* and *2*) exhibited the highest tensile strengths and moduli.

*Fracture toughness*

Since the PAEO P5 (*Table 1*) exhibited relatively high film properties and preliminary work indicated good processability, a neat resin moulding was fabricated by heating to 300°C under 2.1 MPa and holding for 1 h. The moulding was well consolidated and void free. Flash from the moulding exhibited a $T_g$ of 182°C; however, no $T_m$ was detected. The moulding was machined into miniature compact tension specimens and tested at room temperature. A $K_{Ic}$ (critical stress intensity factor) of 6.25 mPa m$^{1/2}$ was obtained which calculates to a $G_{Ic}$ (critical strain energy release rate) value of 10 990 J m$^{-2}$. This number is very high. For example, Udel® P1700 polysulphone gave a $G_{Ic}$ of 3300 J m$^{-2}$ when tested in our laboratory. The fractured surface of the PAEO compact tension specimen showed an extensive amount of yielding, creating new surface area to dissipate the fracture energy.

*Adhesive specimens*

Preliminary adhesive properties were determined on PAEO P5 (*Table 1*) and are presented in *Table 4*. Adhesive tape was prepared by multiple coating of 112 E glass with an A-1100 finish using *m*-cresol solution of the polymer with subsequent drying after each coat. Ti–Ti tensile shear specimens were fabricated in a press by heating to 300°C under 1.4 MPa for 1 h. After adhesive panel fabrication, the adhesive in the bond area was amorphous, as indicated by d.s.c. and WAXS. The tensile shear strengths for the unexposed specimens tested at 23 and 150°C were ~22.1 and 17.9 MPa, respectively. The specimens tested at 177°C exhibited low strength (2.8 MPa) and thermoplastic failure. After isothermal ageing for 500 and 1000 h at 200°C in flowing air and testing at 23, 150 and 177°C, the tensile shear strengths increased significantly. Examination of aged adhesive tape by d.s.c. and WAXS again showed no sign of crystallinity. In addition, the aged polymer remained soluble in *m*-cresol, indicating that no crosslinking had occurred during isothermal ageing. Mass spectral analysis of unaged adhesive tape indicated that a small amount of *m*-cresol was present. The residual *m*-cresol present in the adhesive tape was apparently enough to plasticize the material, thereby lowering tensile shear strengths and causing thermoplastic failure at elevated temperatures in the unexposed specimens.

## CONCLUSIONS

Several poly(arylene ether 1,3,4-oxadiazole)s and poly(arylene ether 1,2,4-triazole)s were prepared by the aromatic nucleophilic displacement reaction of a bisphenol oxadiazole and a bisphenol triazole with activated aromatic dihalides. The polymers exhibited film properties higher than those of poly(arylene ether)s without heterocyclic units. One poly(arylene ether 1,3,4-oxadiazole) exhibited good adhesive properties and outstanding fracture toughness.

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