

Properties of Highly Soluble Addition-Type Imide Oligomers Containing Fluorenylidene Groups and Cured Resins

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In previous works, in order to improve the solubility of the imide oligomer while maintaining high thermal resistance, fluorenylidene groups were introduced to asymmetric thermosetting polyimide "TriA-PI" [1, 2]. In this work, an imide oligomer based on symmetric 3,3',4,4'-biphenyltetracarboxylic dianhydride (*s*-BPDA), a diamine containing fluorenylidene diphenyl ether group (9,9-bis(4-(4-aminophenoxy)phenyl)fluorene, BAOFL), and 4-phenylethynylphthalic anhydride (PEPA) was synthesized through thermal imidization in NMP. The calculated degrees of polymerization of the imide oligomer were 4. After imidization, the reaction solution maintained homogeneity without precipitation. The imide oligomer had excellent solubility of more than 33 wt% in aprotic polar solvents. The minimum melt viscosity was 326 Pa·s, measured by a rheometer. The imide oligomer could be molded easily by using a hot press. The glass transition temperature (T_g) of the cured resin exhibited 321 °C, judged by DSC. The tensile modulus, tensile strength, and elongations-at-break of the cured resin film were 2.78 GPa, 110 MPa, and 10.2 %, respectively. Properties of the imide oligomers and cured resins prepared from *s*-BPDA and several diamines are summarized in Table 1. The imide oligomer based on BAOFL has high solubility and good processability as compared with other diamines. Furthermore, T_g was higher than that of other cured resins. It suggested that fluorenylidene groups affect solubility and heat resistance, and ether linkages affect low viscosity and processability.

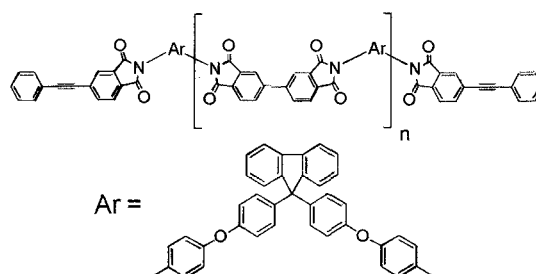


Fig. 1 Chemical structure of the imide oligomer containing fluorenylidene diphenyl ether groups ($n = 4$).

Table 1 Properties of the imide oligomers and cured resins based on *s*-BPDA and several diamines

	diamines ^{a)}	imide oligomers		cured resins ^{c)}	
		solubility in NMP ^{b)}	processability	T_g (°C) ^{d)}	T_{d5} (°C) ^{e)}
PI resin-1	BAOFL	soluble (33 wt%)	good	321	551
PI resin-2	4,4'-ODA	insoluble	poor	296	552
PI resin-3	1,3,4-APB	insoluble	good	250	548

a) BAOFL: 9,9-bis(4-(4-aminophenoxy)phenyl)fluorene, 4,4'-ODA: 4,4'-diaminodiphenyl ether, 1,3,4-APB: 1,3-bis(4-aminophenoxy)benzene b) Measured in NMP at r.t. c) Cured at 370°C for 1h. d) Determined by DSC at a heating rate of 10°C /min under argon. e) Determined by TGA at a heating rate of 10°C /min under argon.

References

- [1] Y. Ishida, T. Ogasawara and R. Yokota, *The Sixth Proceeding of China-Japan Seminar on Advanced Aromatic Polymers*, 184 (2004)
- [2] Y. Ishida, T. Ogasawara and R. Yokota, *Preprints of the 8th SPSJ Intl. Polym. Conf. (IPC 2005)*, 572 (2005)

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