

## Dynamic Mechanical Properties of BPDA-based Polyimides

ChunHai chen, Rikio Yokota,

Institute of space and astronautical science, 3-1-1 yoshinodai, Sagamihara, Kanagawa  
229-0022, Japan

### Introduction

Aromatic polyimides have been widely applied in modern high-technology fields due to their excellent thermal stability, good mechanical properties, electronic properties and chemical resistance, etc. Many researches have been performed on structure-property relationships in polyimides to obtain information for practical application. In numerous polyimides, biphenyltetracarboxylic acid dianhydride (BPDA) based polyimides have been recognized because of its special asymmetric structure. A few reports have focus on structure-property research of isomeric BPDA-based polyimides. In addition, when the PIs films were prepared by two-step method, the properties of PIs films are not only relied on their chemical structure<sup>(1-6)</sup>, but also the thermal imidized condition. To obtain the chemistry, mechanical and physical properties of PIs films have to consider these influence factors, namely the crystal structure and dimensional orientation of PIs chains are different with changing of the thermal imidized conditions.

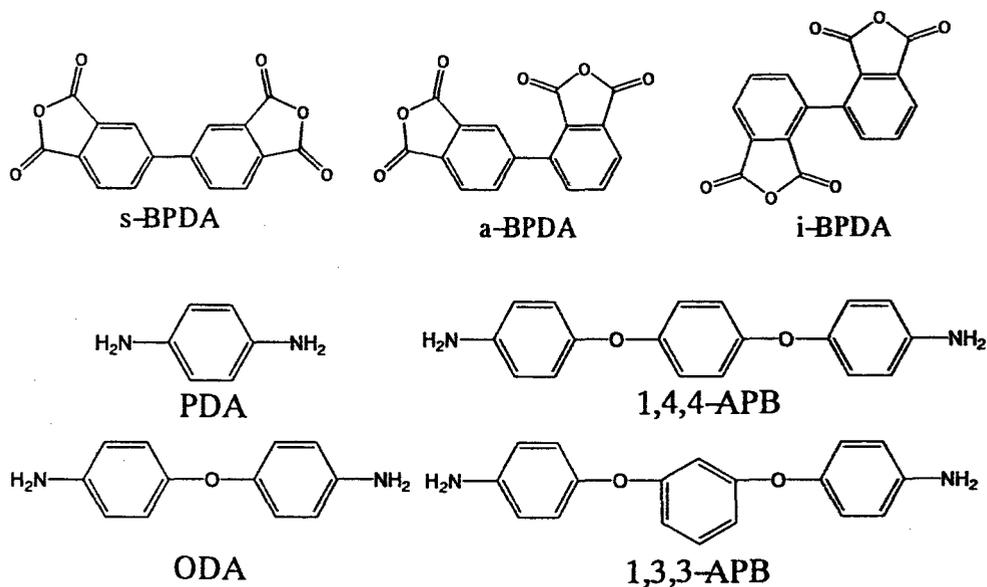
In this present paper, a series of polyimides films were prepared 2,3,3,4 -biphenyltetracarboxylic dianhydride (a-BPDA) and 2,3,2,3 -biphenyltetracarboxylic dianhydride (i-BPDA) with various aromatic diamines and were investigated in the different thermal imidization temperature using dynamic mechanical analysis (DMA) technique. The properties of the a-BPDA and i-BPDA based PIs were compared with those of polyimides delived from 3,4,3,4 -biphenyltetracarboxylic dianhydride (s-BPDA) with the same various diamines.

### Experiment

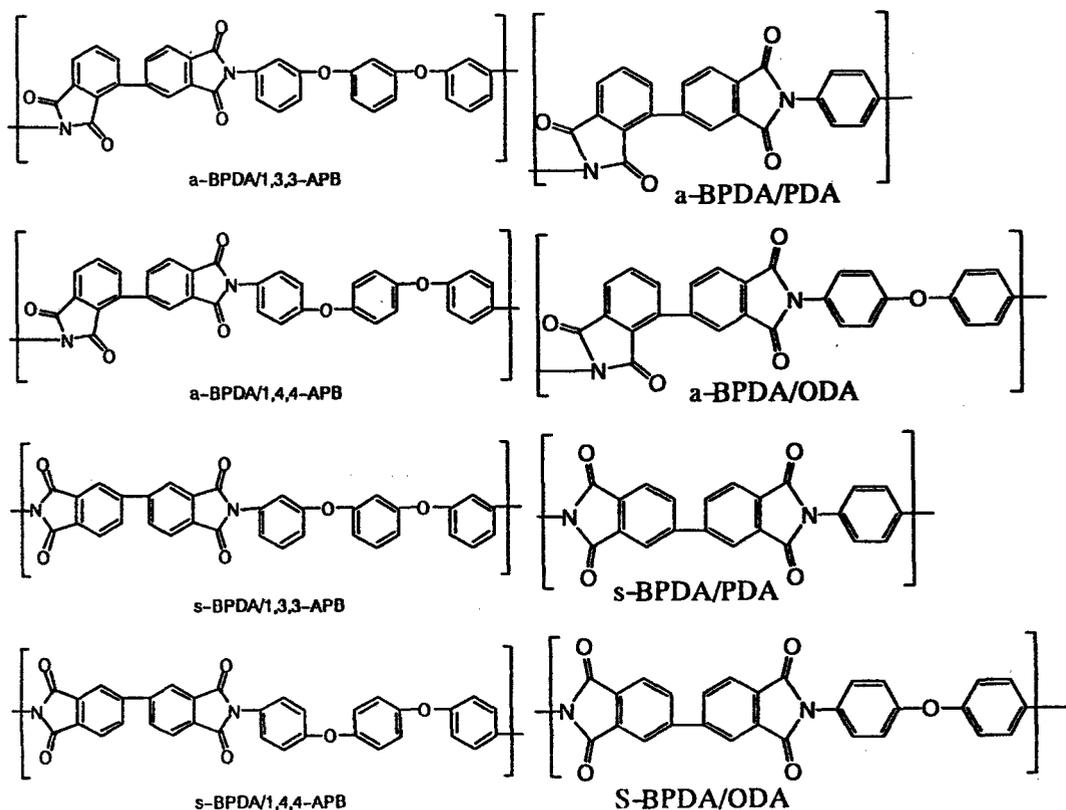
**Material** 2,3,3,4 -biphenyltetracarboxylic dianhydride (a-BPDA, Ube industries Inc., m.p.197°C), 2,3,2,3 -biphenyltetracarboxylic dianhydride (i-BPDA, JSR), 3,4,3,4 -biphenyltetracarboxylic dianhydride (s-BPDA, Allco Chemical Co., m.p.227°C), 1,4-bis(4-aminophenoxy)benzene (1,4,4-APB, Chriskev co.), 4,4 -Diaminodiphenyl (ODA, Tokyo kasei kogyo co.), 1,4-phenylenediamine (PDA, Tokyo kasei kogyo co.), 1,3-bis(3-aminophenoxy)benzene (1,3,3-APB, Mitsui chemicals co.), N,N-dimethylacetamide (DMAc, Tokyo kasei kogyo co.)

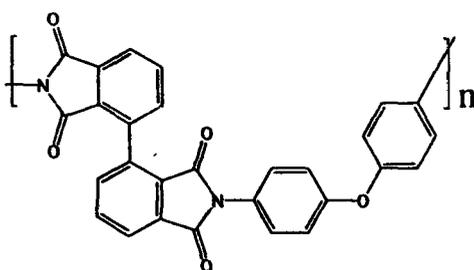
**Synthesis of PAAs** Diamine (0.015mmol) was dissolved in 30ml of DMAc under nitrogen at room temperature. To this solution (0.01499mmol) was added dianhydride group-by-group and maintained at 20°C. After the solution was stirred for 1h, it was added in anhydride for end capping and kept at that temperature for 2h. The appropriate amount of DMAc was added to the solution so as to keep the total volume of the solution constant at 10%. The polyamic acid (PAA) was obtained. Scheme 1 shows the chemical structure of dianhydrides and diamines.

**Preparation of PI Films** A clean and dry glass substrate was heated to 60°C and the DMAc solutions of PAA was cast onto the glass and then dried at 60°C for 2h.in an air convection oven. The semi-dried PAA film was removed from the substrate, and wrapped with at film then put into a rounded furnace. Film of s-BDA/1,3,3-APB gets from NASA Langley RC.



Scheme 1 The structures of dianhydrides and diamines monomer





i-BPDA/ODA

Scheme 2 The structures of PIs

## Results and Discussion

Dynamic mechanical analysis were performed by using RED-II.

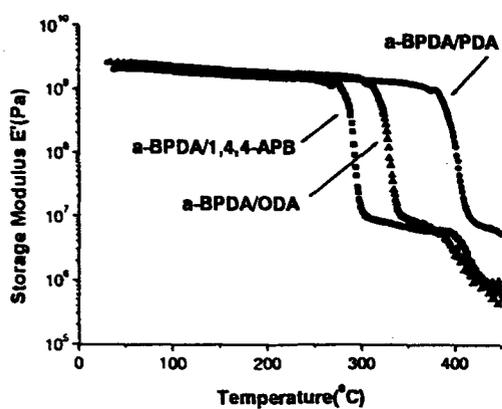


Fig. 1 E' curves for a-BPDA based PIs films

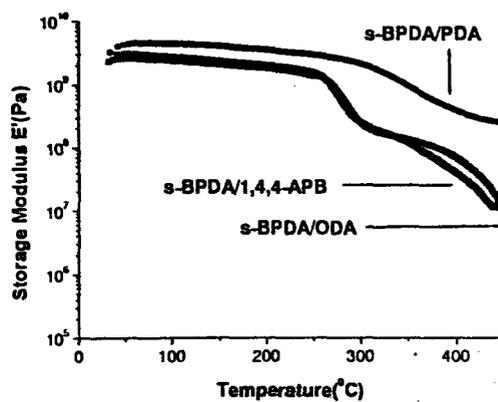


Fig. 2 E' curves for s-BPDA based PIs films

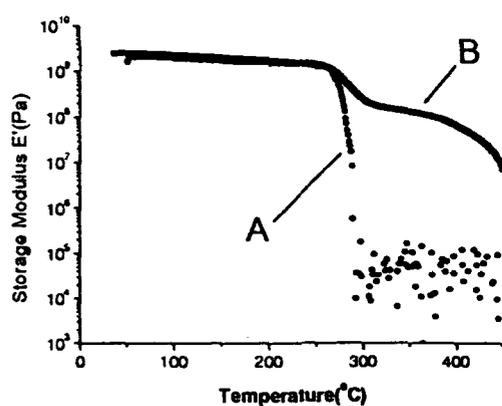


Fig.3 DMA Curves of s-BPDA/ODA Films:  
A cured at 150°C/1h + 250°C/2h,  
B cured at 150°C/1h + 250°C/2h + 350°C/1h

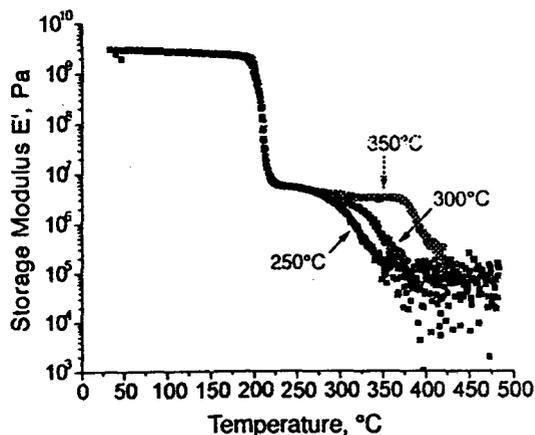


Fig. 4 DMA Curves of s-BPDA/1,3,3-APB Films

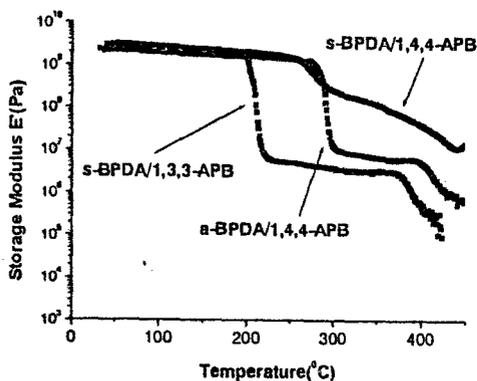


Fig. 5 E' curves for s-BPDA/1,4,4-APB, s-BPDA/1,3,3-APB and a-BPDA/1,4,4-APB films.

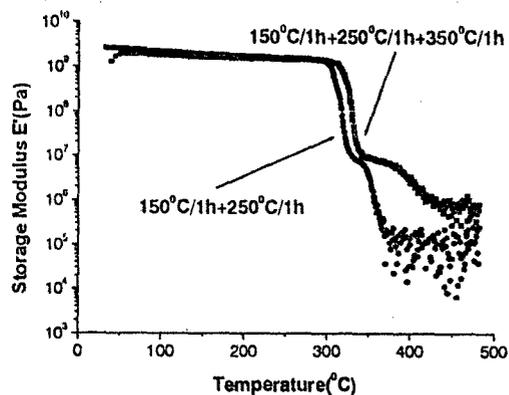


Fig. 6 DMA curves of a-BPDA/ODA films.

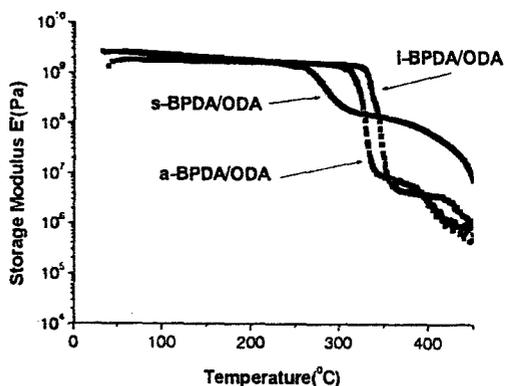


Fig. 7 E' curves for s-BPDA/ODA, i-BPDA/ODA and a-BPDA/ODA films.

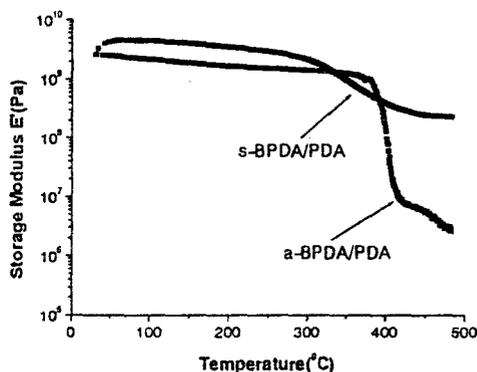


Fig. 8 DMA curves for s-BPDA/PDA and a-BPDA/PDA films

### References and Notes

- (1) Masatoshi Hasegawa, Nobuyuki Sensui, Yoichi shindo, and riki yokota, *Macromolecules*, Vol. 32, 387-396.
- (2) T.V.Holland, T.E.Glass, J.E.McGrath; *Polymer*, 2000, 41, 4965-4990.
- (3) Yuejin Tong, Wenxi huang, jun Luo, Mengxian Ding, *J polym Sci Part A: polymer chemistry*, Vol. 37, 1425-1433. 1999.
- (4) Kochi, M.; Uruji, T.;Iizuka, T.; Mita, I.; yokota, R. *J. Poly. Sci., Part C* 1987, 25, 441.
- (5) Hasegawa, M.; kochi, M.; Mita, I.; Yokota, R. *Eur. Polym. J.* 1989,25, 349
- (6) Inoue, H.; Okamoto, H.; Hiraoka, Y. *Radiat. Phs. Chem.* 1987, 29, 283.
- (7) Kochi, M.; Mita, I.; Horigome, T.; Yokota, R. In *Proceedings of 2<sup>nd</sup> International Conference on Polyimides*; Society of Plastics Engineers: Ellenville. NY, 1985; p 454.