

일반강연 I-6

## 폴리술폰/NMP/물 계의 고화 현상에 관한 연구

김 제 영, Toshiyuki KANAMORI\*, 이 환 광\*\*, 백 기 전, 김 성 철  
한국과학기술원 화학공학과, 물질공학공업연구소\*, 충남산업대학교\*\*

### Vitrification Phenomena in Polysulfone/NMP/water system

Je Young Kim, Toshiyuki KANAMORI\*, Hwan Kwang Lee\*\*,  
Ki Jun Baik, and Sung Chul Kim

Dept of Chem. Eng., KAIST, Dept. of Polymer Eng., NIMC, Japan\*  
Dept of Ind. Chemistry, Chungnam Sanup University\*\*

#### 1. INTRODUCTION

Since the knowledge of vitrification phenomena can lead to a better understanding of the mechanism of membrane formation, it is desirable to include vitrification line into the phase diagrams. While the final morphology obtained during phase inversion depends upon the kinetics as well as the thermodynamics of the phase separation, the equilibrium phase diagram and vitrification line for amorphous polymers are still a good tool for controlling the morphology and interpreting the membrane structure.

#### 2. EXPERIMENTAL

Samples were prepared by weighing the appropriate amount of polysulfone, NMP and water in silver sample pans using a micro-syringe. Pans were hermetically sealed and they were stored in vacuum oven at least two days at 210°C to obtain a homogeneous solution. The sealed silver pan can endure 50 atm of inner pressure and there was no measurable weight loss after heat treatment. The DSC apparatus was a SEIKO model DSC 120 equipped with cooling accessories for subambient operation. All DSC runs were carried out with a heating rate of 10°C per minute.

#### 3. Results and Discussion

The vitrification line for the ternary system of PSf/NMP/water was determined by a DSC measurement with varying the compositions. In the

case of polysulfone, the  $\alpha$ -transition temperature is observed at 187.5°C and

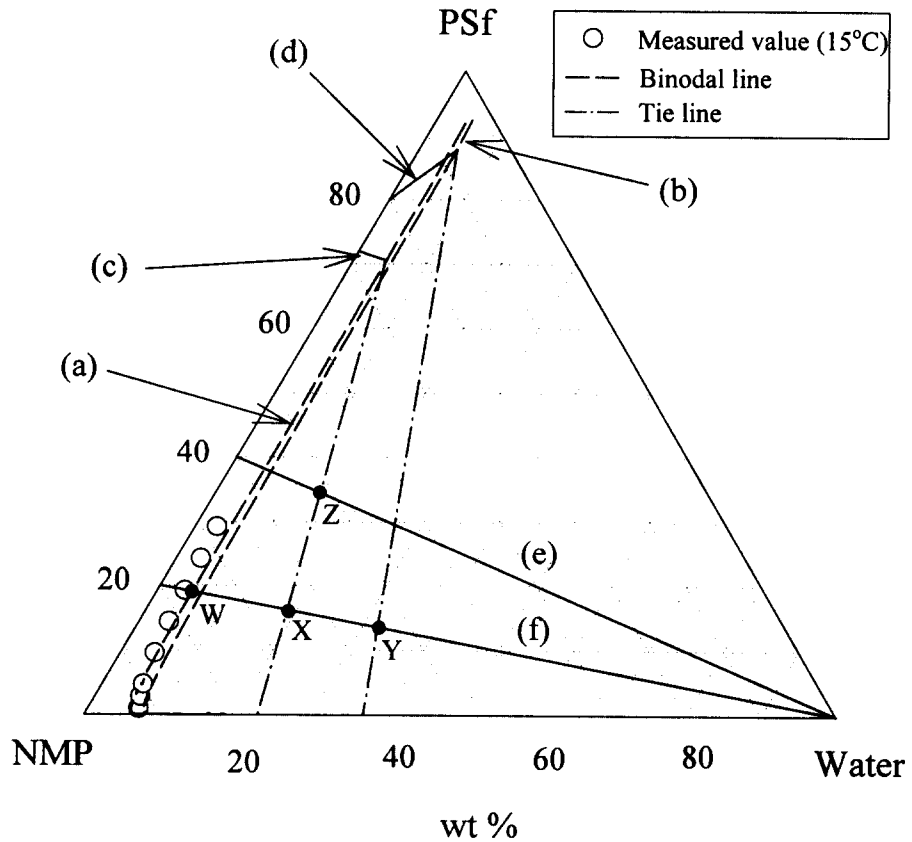


Figure Ternary phase diagram for PSf/NMP/water system showing calculated binodal line at 15°C (a) and at 60°C (b). Experimental binodal line at 15°C, vitrification line at 15°C (c) and at 60°C (d), tie line, and simplified mass transfer path (e), (f) are also included.<sup>4</sup>

the  $\beta$ -transition at -21.4°C thought to be the result of phenylene group

rotation. The  $T_g$  drop with the addition of solvent is more pronounced in the concentrated region and less in the dilute region and there is linear relationship between  $T_{g,a}$  of polysulfone and solvent content in the range of interest which is between 10 ~ 30 wt % of solvent.

We also draw vitrification line in the phase diagram for ternary system of PSf/NMP/water at 15°C and 60°C. As temperature goes up, the vitrification line moves upward which means higher polymer concentration is needed to reach vitrification condition. The vitrification composition was 72.0 wt % of polysulfone and 28.0 % of solvent at 15°C and 79.8 wt % of polysulfone and 20.2 % of solvent at 60°C, respectively. We also found that the slope of the vitrification line changed with temperature and it was steep in the case of higher temperature. From the figure of phase diagram and vitrification line, we found small amount of water (10-20 wt %) can induce the vitrification of polymer solution in PSf/NMP/water system at 15°C and more amount of water is needed to induce the vitrification at higher temperature.

#### 4. REFERENCES

1. J. G. Wijmans, J. Kant, M. H. V. Mulder, and C. A. Smolders, *Polymer*, 26, 1539 (1985)
2. W. R. Burghardt, L. Yilmaz, and A. J. McHugh, *Polymer*, 28, 2085 (1987)
3. G. E. Gaides and A. J. McHugh, *Polymer*, 30, 2118 (1989)
4. J. Y. Kim, H. K. Lee, K. J. Baik, and S. C. Kim, *J. Appl. Polym. Sci.*, 65, 2643 (1997)
5. A. J. . Reuvers, F. W. Alterna, and C. A. Smolders, *J. Polym. Sci. Polym. Phys. Ed.*, 24, 793 (1986)
6. F. Fedos, *J. Polym. Sci. Polym. Lett. Ed.*, 17, 719 (1979)