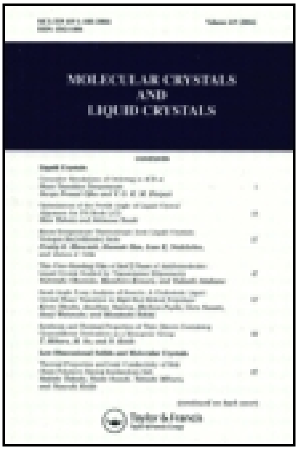

Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl19

Evolution of Optical Transmission Loss in Crosslinked Epoxy

H. H. Song [a], J.-H. Park [a] & K A Noh [a]

[a] Department of Macromolecular Science, Hannam University, Taejon, Korea

Version of record first published: 04 Oct 2006

Mol. Cryst. Liq. Cryst., 1998, Vol. 316, pp. 23–26
Reprints available directly from the publisher
Photocopying permitted by license only

Evolution of Optical Transmission Loss in Crosslinked Epoxy

H. H. SONG, J.-H. PARK, and K.-A. NOH
Department of Macromolecular Science, Hannam University, Taejon, Korea

Optical transmission loss changes upon curing the mixture of epoxy/ethylene diamine were studied. A UV-Vis spectrum measured during the cure process was utilized to monitor both cure process and transmission loss change simultaneously. It was found that initial homogeneity of the epoxy/EDA mixture was the key parameter to determine the optical transmission of the cured epoxy. Slow heating up to the curing temperature allowed the mixture homogenous, thus resulted in a structure of very low transmission loss. Subsequent curing rate was also another factor affecting the uniformity of the cured structure.

Keywords optical transmission loss; epoxy curing; UV-Vis spectrum

INTRODUCTION

Utility of polymer glass for transmitting optical signals[1]-[4] comes from many advantages including superior processibility, stability, high flexibility, and low preparation cost. Recently the optical polymers have been also utilized as an optical polymer matrix hosting second order or third order nonlinear optical (NLO) chromorphores. In second order NLO applications[5], the chromorphores dissolved in the polymer matrix are required to be aligned to produce NLO signal. Minimizing the relaxation of the oriented chromorphores, thus to maximize the NLO signal has been the key issue and the crosslinkable polymer[6],[7] has proven to be very effective. However, substantial increase of transmission loss takes place accompanying the crosslinking process, which has limited the use of crosslinkable polymers as optical matrices.

In the present study we attempted to elucidate the evolution of transmission loss during the crosslinking of polymer matrix. Emphasis was placed especially on the crosslinking conditions and their effects on the optical transmission loss.

EXPERIMENTAL METHODS

Diglycidly ether bisphenol A (DGEBPA) (M.W. = 350 $gmol^{-1}$) and ethylene diamine (EDA) were chosen for this study. Curing was controlled by changing the heating rates at 1, 3, 5, 7, and 10 °C/min. A UV-Vis spectrometer (Perkin Elmer Lamda series 14) was used to monitor the curing process. For the transmission loss due to scattering, the spectrum was analyzed at the wavelengths of 600-700 nm where the vibrational absorption is absent. The curing process, on the other hand, was monitored at the wavelength of 700 - 1100 nm where the characteristic absorption peaks of DGEBPA and EDA are present. Optical loss Φ (dB/cm) of the specimens at each wavelength was then determined from $\Phi = -\frac{10}{L}\log \frac{I}{I_o}$, where L is the thickness (cm) of the sample and I/I_o is transmission ratio at the individual wavelengths.

RESULTS AND DISCUSSION

In Figure 1, UV-Vis spectra of pure epoxy, epoxy/EDA before and after cure are compared. The spectrum of the pure epoxy shows flat absorption value up to the wavelength of 680 nm and several absorption peaks thereafter. The flat portion of the absorption spectrum at 500-700 nm consists of the absorption tail of the electronic transition and the Rayleigh scattering from the structural inhomogeneities.[8] The absorption peak at 1047 nm disappearing after the reaction is apparently associated with the amine in the EDA compound.

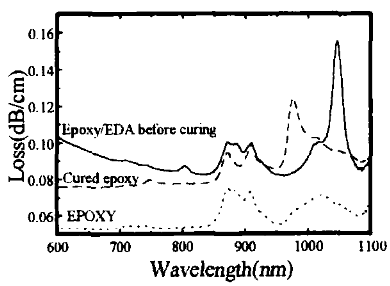

FIGURE 1. UV-Vis spectra of epoxy and epoxy/EDA before and after curing.

The absorption peak at 978 nm, on the other hand, grows with the curing process and can be attributed to hydroxyl group. Therefore, these two absorption peaks can be utilized to monitor the cure process. In Figure 2, changes of the absorption peak area at 1047 nm during the cure are plotted The decrease of the peak height noted in the figure represents the curing reaction. The temperature of rapid changes in the peak height also agrees with DSC reaction endotherm. Curves in Figure 3 represent the transmission losses at λ = 600 nm measured during the cure of mixture at different heating rates. In the curves of 1 and 3 °C/min., a large amount of initial drop in the transmission loss can be noted while other curves of higher heating rates maintain the initial transmission loss value until the curing begins. The slow heating rate apparently allowed the mixture homogeneous before the temperature reaches curing point. The curves are also manifesting that the optical loss increases as the curing proceeds and the increment depends on the heating rate. The epoxies cured at the higher heating rates of 5, 7, and 10 °C/min show a larger increment in the transmission loss during the curing process, resulting in a very high transmission loss. Two totally distinct behaviors, depending on the heating rates, observed in the Figure 3-A suggest that the initial homogeneity of the mixture is the key factor to control the structural uniformity, thus the optical transmission of the cured epoxy. Curves shown in Figure 3-B also represent the transmission loss of epoxy/EDA mixture obtained during the cure process. However, in this case all mixtures were slowly raised up to 50 °C at a heating rate of 1 °C/min. and subsequently cured at different heating rates as indicated in the figure. Initial drop to the identical transmission loss values can be seen in all curves. The

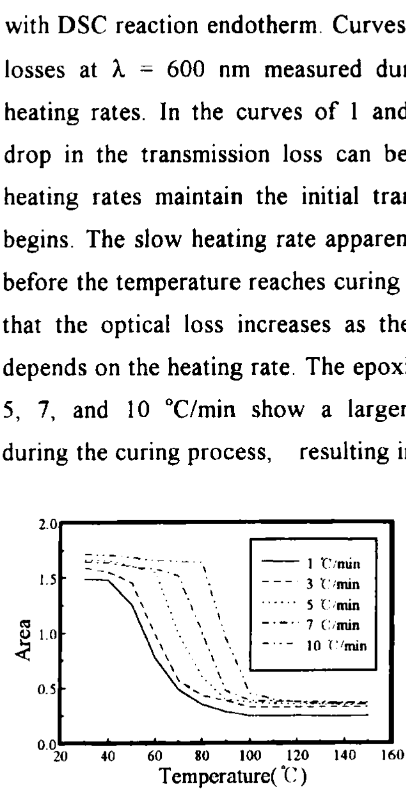

FIGURE 2. Absorption peak (1047nm) area changes during the cure process.

transmission loss increment upon curing and their heating rate dependence are also seen as was noted in the Figure 3-A. However, amounts of increment after curing is now far less than those observed in the curves of Figure 3-A. The epoxies cured in this way resulted in a structure of far better optical transmission.

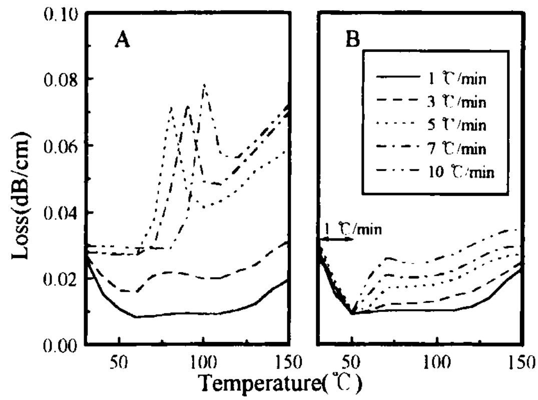

FIGURE 3. Transmission loss changes during the curing process at λ = 600nm.

Acknowledgments

This research was financially supported by the Korea Science and Engineering Foundation (951-1101-030-2).

References

[1.] R. Schriever, H. Franke, H. G. Festl and E. Kratzig, Polymer, **26**, 1423 (1985).
[2.] T. Kaino, Appl. Phys. Lett., **48**, 757 (1986).
[3.] R. R. Krchnavek, G.R. Lalk and D. H. Hartman, J. Appl. Phys., **66**, 5156 (1989).
[4.] R. Reuter, H. Franke and C. Ferer, Appl. Opt., **27**, 4565 (1988).
[5.] K. D. Singer, J. E. Sohn and S. J. Lalama, Appl. Phys. Lett., **49**, 248 (1986).
[6.] K. S. Lee, K. J. Moon, and H. K. Shim, Korea. Polymer J., **4**, 191 (1996).
[7.] J. A. F. Boogers, P. Th. A. Klaase, J. J. de Vlieger, D. P. W. Alkema, and A. H. A. Tinnemans, Macromolecules, **27**, 197 (1994).
[8.] T. Kaino in "Polymers for lightwave and integrated optics:technology and application", edited by L. A. Hornak, Mercel Dekker, Inc. (1992).