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Improved Temporal Stability of Electro-optic Property in Sol-gel Matrix bearing a Silylated Dye

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We simply synthesized the triethoxysilane bearing the second-order nonlinear optical(NLO) active chromophore or chalcone derivatives. In order to improve the poor temporal stability of second-order NLO effect in sol-gel matrix, we introduced the photo-crosslink between the side chain.themselves. Photo-crosslink formation was confirmed with UV-VIS absorption and infrared vibrational spectroscopy. Under a polarized light, we investigated the decaying behavior of molecular order parameter after corona poling.

Keywords: sol-gel, chalcone, electro-optic, temporal stability

INTRODUCTION

Sol-gel materials for photonic applications have been highlighted over a decade. Poor temporal stability of electro-optic property was the important issue to overcome. In an attempt to improve its temporal stability, we designed the photo-crosslinkable system using NLO active triethoxysilane and silylated chalcone. In this work, we investigated the photo-crosslink between the side chains by virtue of absorption and infrared spectroscopy. The temporal stability after poling and photo-curing was simply checked observing the decay of molecular order parameter by polarized absorption spectroscopic technique.

EXPERIMENTAL

Synthesis of silylated dye(SGDR1) was described using disperse red 1 in the literature [1]. Silylated chalcone(SGCHC) was also synthesized using 4-(2-hydroxyethoxy)chalcone and isocyanatopropyl triethoxysilane. Synthetic procedure will be described in elsewhere.

RESULTS AND DISCUSSION

The silylated precursor bearing NLO active chromophore and chalcone derivative[2] was prepared by simple urethane forming reaction between the alcohol and isocyanate in trialkoxysilane. The synthesized silylated compounds were illustrated in Figure 1.

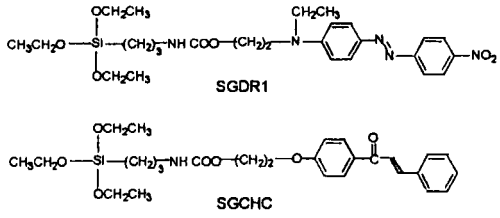

FIGURE 1 Silylated compounds for photo-crosslinkable sol-gel matrix.

UV-VIS spectral analysis under irradiation at 365nm

We used two films of SGDR1/SGCHC and SGCHC only on quartz for UV-VIS spectral analysis. In the case of SGCHC only, the intensity of absorption maximum at 332 nm from double bond in chalcone gradually disappeared within 30 minutes on exposure to UV light(365 nm) (Figure 2 inset). This is strongly attributed to the 2+2 cycloaddition reaction forming the cyclobutane ring similar to the photochemical reaction between cinnamoyl derivatives. The film of SGDR1/SGCHC, however, showed relatively slow rate of disappearance of double bond in chalcone unit, which is due to the dilution effect (Figure 2). Resulting from above absorption characteristics, we confirmed that the photo-crosslink can occur in the matrix.

IR spectral analysis under irradiation at 365nm

In addition to the UV-VIS absorption analysis, we investigated the change of the IR spectrum during exposure to UV light. As is shown in Figure 3, the characteristic band of double bond in chalcone appeared at 1604 and 1510 cm^{-1} before irradiation. With the exposure time, the intensities of two bands decreased drastically in SGCHC, which is another information to support the formation of photo-crosslink.

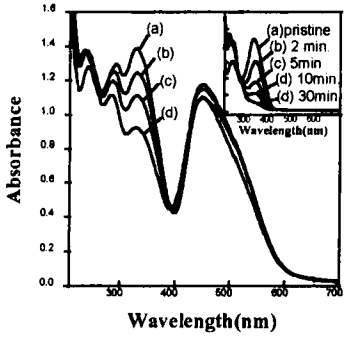

FIGURE 2 Absorption spectra of SGDR1/SGCHC(Inset: SGCHC) (a) pristine;(b)30min.;(c) 60min.; (d) 120min. (exposure time)

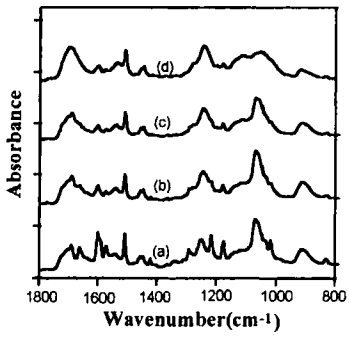

FIGURE 3 Infrared spectra of SGCHC film.(a) pristine (a) 2 min.; (b) 5 min;(c) 10 min.;(d) 30 min. (exposure time)

Decaying behavior of molecular order parameter

When observing the electro-optic properties of polymeric materials, we normally poled the samples to induce the noncentrosymmetry at certain high temperature. During poling 200-210°C of SGDR1 only, some portion of the chromophore was decomposed. Crosslink can be induced at relatively low temperature during poling. We treated the films of SGDR1/SGCHC by corona poling technique at 170°C. The polarized absorption spectroscopy could give us the molecular order parameter, A_2 with light polarized parallel to the plane of incidence(TM) [3]. We could employ the relationship

between absorption as a function of the angle of incidence before and after poling. Since the molecular order is strongly related to the electro-optic property, we traced the decaying behavior of A_2 during annealing at 100 °C. In the case of SGDR1 only, the order parameter was fell to 20% of initial one even after 30 minute annealing, which was expected. Poling and photo-cured film of SGDR1/SGCHC exhibited much better stability compared to SGDR1 only. Upon exposure to UV light for 30 minutes, 80% of initial value was sustained even after 15 hour annealing (Figure 4).

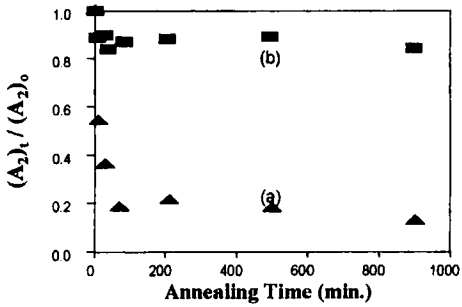

FIGURE 4 Decay of A_2 during annealing at 100 °C. (a) SGDR1 only; (b) SGDR1/SGCHC

Acknowledgments

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