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Photocure Behavior of Highly Transparent and Thermally Stable Photosensitive Polymer

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A new photosensitive prepolymer was designed and synthesized by esterification of an epoxy and methacrylic acid. Photopolymerization behavior of the prepolymer was quantitatively investigated by FT-IR spectroscopy. We also studied thermal stability by observing changes in the transmittance of the polymer film upon heating at 250°C for a given period of time.

Keywords Photosensitive polymer; Photocure behavior; Quantitative FT-IR spectroscopy; Thermal stability

INTRODUCTION

Recently, photosensitive polymers have been extensively studied since they have been used in many application fields including microlithography [1,2]. In this study, we report synthesis of a new photocurable polymer, which is expected to possess high transparency and good thermal stability. We investigated photocure kinetics of the

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polymer using FT-IR spectroscopy and thermal stability by observation of changes in the transmittance of the polymer film upon heating

EXPERIMENTAL

Photosensitive prepolymer was prepared by reaction with methacrylic acid (MAA) and epoxy resin (YX-4000H) using TPP as catalyst as shown in Figure 1. Chemical structure of the prepared prepolymer was confirmed by NMR and FT-IR spectroscopies. The photosensitive prepolymer was completely dissolved in a solvent with a crosslinking agent and a photoinitiator. The solution was spin-coated on glass substrate, resulting in good quality of the film with thickness of about 1 μm . The film was then exposed to UV (365nm) for various exposure periods. Photocure behavior of the prepolymer film was studied by monitoring change of absorbance at 810 cm^{-1} corresponding to double bond of MAA. We also studied thermal stability by observing changes in the transmittance of the polymer film upon heating at 250 $^{\circ}\text{C}$ for 1 hour.

RESULTS AND DISCUSSION

Figure 2 shows IR spectra of (a) YX-4000H and (b) YX-4000H/MAA. As shown in Figure 2(b), new characteristic absorption peaks of the prepolymer were observed at 3200–3500 cm^{-1} (hydroxyl group), 1730 cm^{-1} (carbonyl group), and 810 cm^{-1} (acrylate double bond), confirming the desired chemical structure of diacrylate prepolymer.

We confirmed photopolymerization of the prepolymer by FT-IR spectroscopy as shown in Figure 4. Absorbance at 810 cm^{-1} relating to

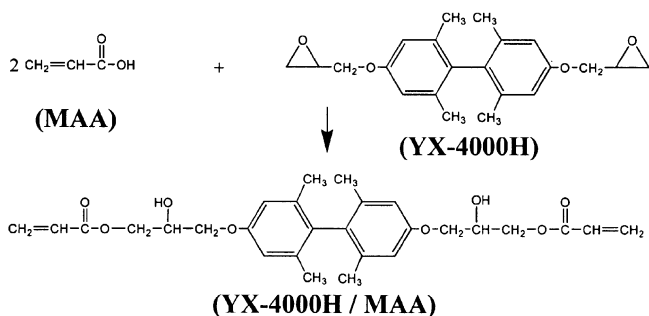


FIGURE 1. Scheme of synthesis.

$\text{CH}=\text{CH}_2$ twisting vibration of the prepolymer decreased extremely rapidly during the early UV irradiation period and then slowly decreased with exposure time. This implies the photocure reaction takes place very extensively in the early stage to a high conversion. The surface of the photocured polymer film was very smooth as shown in Figure 3. We also investigated the effect of photocrosslinking agent (DPHA) on the rate and the conversion of photocure reaction of the prepolymer. As shown in Figure 4(b), addition of DPHA resulted in slower rate and smaller conversion of the photocure reaction. The polymer was extremely stable, exhibiting almost no change of transmittance even after heating at 250°C for 1 hour.

CONCLUSIONS

A photosensitive diacrylate prepolymer was synthesized by reaction with MAA and epoxy resin. We confirmed the chemical structure of the prepolymer by FT-IR spectroscopy. We found the photocure of the prepolymer took place extremely rapidly to a very high conversion with UV radiation. We also observed the prepolymer manifested excellent thermal stability.

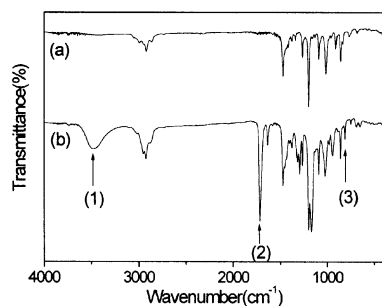


FIGURE 2. FT-IR Spectra of (a) YX-4000H and (b) YX-4000H/MAA.

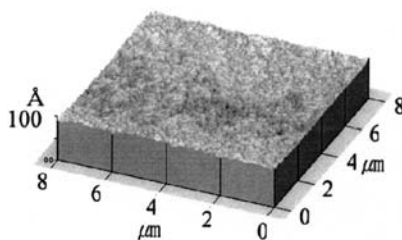


FIGURE 3. AFM photograph of photocured film of YX-4000H/MAA.

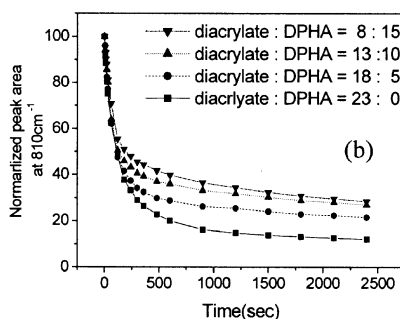
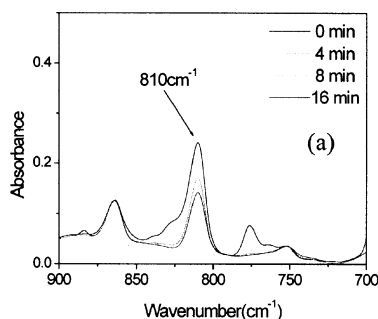


FIGURE 4. (a) FT-IR spectra of YX-4000H/MAA and (b) normalized peak area at 810 cm^{-1} as a function of irradiation time.

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