Journal of the Optical Society of Korea, Vol. 11, No. 4, December 2007, pp. 183-191

Effect of Modification of SeO_2/Acrylamide Ratios on Diffraction Efficiencies in PVA/AA Photopolymer Films

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(Received September 7, 2007 : revised November 5, 2007)

The highest diffraction efficiency (DE) value after illumination and post-curing of photopolymer films were obtained at the SeO_2/Acrylamide (AA) Ratios of AA 3.0 g, SeO_2 1.0g and the DE's were stable values of over 90%. By the addition of SeO_2, the maximum DE at the initial stage of illumination was reached at 300 seconds, which suggests SeO_2 slows down the photopolymerization of AA, which enhances the maximum DE value by giving more migration time. DE variation curve for the optimum composition during extended-time illumination of 9,000 seconds resembles a sine curve due to the combination of the monomer diffusion and the photopolymerization, and the photopolymer film expanded by about 8% after photopolymerization due to monomer migration.

OCIS codes : 160.532

I. INTRODUCTION

Recently, research on holographic recording material by optical recording has been attracting strong interest and applications on digital recordings, watermarking, and hologram recording have been continuously conducted [1-3]. Holographic recording materials are silver halides, photographic emulsions, dichromated gelatin, photo resists, photopolymers, photochromics, photo thermoplastics, photorefractive materials. Among them, photopolymers which show high diffraction efficiency (DE) record images and data by the intensity modulations of recording optical light. Since photopolymers can be recorded by light exposure (self-developing), chemical treatments are not required for recording data or images and only drying processes are needed for manufacturing final products. Due to these advantages, photopolymers have be applied on various holographic optical elements (HOE) and films for security/surveillance holograms, also can be applied in near future as a media for holographic digital data storage (HDDS) and 3-D images, high speed optoelectric parts [4-6].

The fundamental theory of holographic recording with photopolymer can be explained: Photopolymer consists of binding polymer, monomer, photosensitizing dye, and initiator. Upon exposure of laser light with holographic data, photo polymerization can be initiated by absorbing energy and by sensitizing dye and decomposition of initiators. Holographic exposure produces spatial diffraction patterns due to photo-initiated polymerization. Concentration gradient of un-reacted monomers between exposed and unexposed areas induces diffusion of the monomers from unexposed zone to exposed zone. This diffusion produces a compositional gradient, establishing refractive index grating, which made it possible to record digital data or optical patterns. Reduction reaction of photo initiator proceeds when the film is illuminated by laser light of 532 nm wavelength. Photopolymerization reaction generally happens in 3 steps. The reaction mechanism is expected to follow 3 reaction steps as shown in equation (1). Polymer binder is not included in the reaction mechanism, however the reaction speed and reaction process are severely influenced by the properties of the binders, mainly local viscosities.

$$\begin{aligned}&\mathrm{DYE} \rightarrow h\nu \rightarrow \mathrm{DYE}^*\\&\mathrm{DYE}^* + \mathrm{Am} \rightarrow \mathrm{Am}^* + \mathrm{LDYE}\\&\mathrm{Am}^* + \mathrm{M} \rightarrow \mathrm{P}\end{aligned} \tag{1}$$

DYE: photosensitizer
Am: photoinitiator
DYE*: excited photosensitizer
Am*: excited photoinitiator

LDYE: whitened photo sensitizer (decolored)
M: monomer
P: polymerization

When the volume of photopolymer film decreases upon light exposure, the shrinkage of exposed film may cause the changes of the recording angle and the reading angle due to the deviation of Bragg angle. This shrinkage of film limits the recording density increase. To solve this film shrinkage problem, two kinds of shrinkage-free photopolymer material were introduced; photo-attaching or photo-detaching photopolymers in which monomers are attached on or detached from polymeric binders by light exposure, and ring opening photopolymers in which photopolymerization occurs through ring opening polymerization by cations generated from light sensitive photoacid generators (PAG). These two photopolymers showed low shrinkage upon exposure (~0.5%), however diffraction efficiencies are considerable low and the research on enhancing diffraction efficiencies has been continuously pursued [7-10].

Ring opening photopolymers are considered to be the best candidates for commercial photopolymer material. Ring opening photopolymers from Aprillis Co. and Dupont Co. are currently circulated as prototype photopolymers for optical recording apparatus testing [11].

In general, reflection patterns in light induced polymerizing photopolymers are explained as in Figure 1. At first, lighted beams with reflection pattern by positive and negative interferences illuminated the photopolymer film, and then the photopolymerization took place on the area of positive interference of light. Monomer diffusion takes place due to the concentration differences between photo-polymerized zones of low monomer concentration and unpolymerized zones of high concentration.

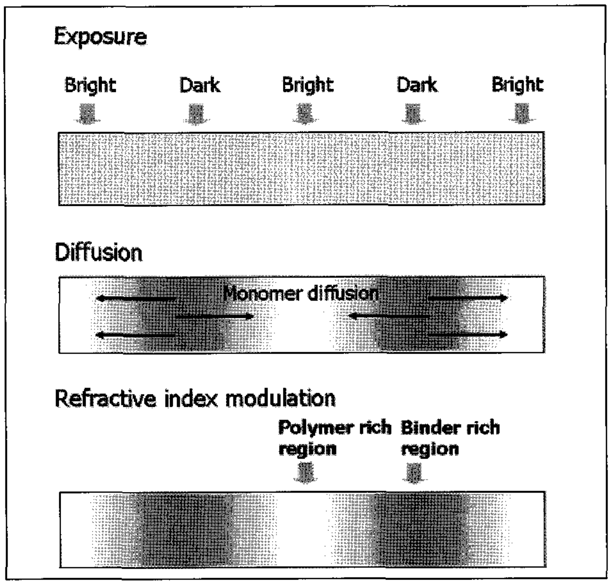


FIG. 1. Diffraction Grating Formation in holographic media after Exposure.

Binding polymers must contain monomers and other ingredients in stable condition without phase separation throughout light induced photopolymerization and must contain adequate viscosity for appropriate monomer diffusion with high transparency. As binding polymers for photopolymers, PMMA's, PVA's, siloxane polymers, and polycarbonates have been used. Among them, PVA's are water soluble and so the film manufacturing is more convenient, however water and heat resistances of the films are low and there are few water soluble initiators for photopolymer synthesis. The popular initiator for PVA photopolymer is TEA, which has slow initiating speed, so activator is required to maintain proper polymerization speed. Siloxane polymers have good thermal and photo stabilities and water resistance so attracted notice in photopolymer research. Solubility of siloxane polymers and monomers, initiators, and other ingredients are usually poor, so enhancement on the solubility should be performed to develop commercial siloxane photopolymers [12-19].

Inorganic light sensitive particle addition such as Kalcogenide materials have been attracting strong interest due to their unique photo-active characteristics and they are used as inorganic thin-film holographic storage materials. In this study, the effects of SeO_2 inorganic crystal addition are examined with green laser light (532 nm) sensitive PVA/AA-based photopolymer systems [16-21]. SeO_2 crystals act as an oxidizing agent which is self reduced by light. SeO_2 crystals are used in this research to promote photosensitivity of dye and so enhance the photopolymerization characteristics. (Figure 2) $FeCl_3$ also acts as photosensitizer which absorbs blue light (408 nm) as powerful as Eosin Y in green light (488~532 nm). However, $FeCl_3$ used in photopolymer system showed volatility of stored data or images by changing of DE which make it difficult at present to adopt as data storage medium unless applications in real time writing-reading medium. So, SeO_2 crystals are considered to be introduced to prepare photopolymer systems in this research. Enhancements in optical charac-

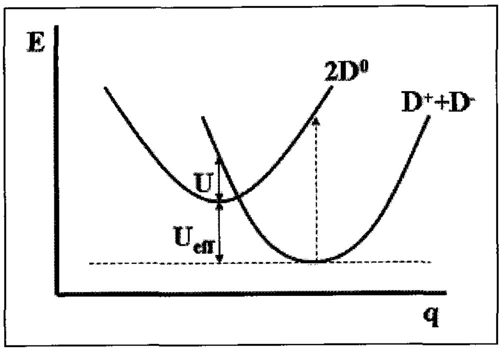


FIG. 2. Energy State by Metallic Bonding of Selenium.

teristics and storage stabilities of photopolymer systems are examined by adding SeO_2 crystals as nano scale mixtures. The catalytic behaviors of inorganic photosensitive materials would be examined to develop optimized photopolymer for holographic recording [20, 21].

II. MATERIALS AND METHODS

2.1 Materials

Components for the photopolymer system in this research are PVA for binder, acrylamide (AA) for photopolymerization monomer, TEA (triethanol amine) for initiator and Eosin Y for a photosensitizer which has high absorption on 488 nm ~ 532 nm wavelength laser light as was shown in Figure 3 (HP 8453 UV-Vis Spectrometer). Our previous research showed reliable response on holographic recording with these compositions [22-24]. SeO_2 crystals are selected as photo-oxidized inorganic crystals. The molecular weights (Mw) of PVA polymer binders were selected as 89,000 ~ 98,000, which are considered to be the adequate internal viscosities for AA monomers to diffuse freely through the binding matrix of photopolymer films. In this experiment, high pure grade chemicals were used without further purification, since small amounts of impurities wouldn't make serious differences in density modulation upon photopolymerization.

2.2 Synthesis of photopolymer film

The first step of the synthesis of photopolymer films is the process of complete mixing of every component. PVA has to be heated up to 80℃ to be melted and liquefied. Previously mixed solutions of AA, TEA, Eosin Y and SeO_2 are mixed with liquefied PVA to be clear solutions. In this experiment, PVA was heated first in water bath without mixing other components for longer than 30 minutes, and then it was cooled down to room temperature until it was used for mixing. AA, TEA, Eosin Y, and SeO_2 were completely solvated to be a transparent solution, and then mixed with cooled PVA for more than 2 hours with magnetic stirrer for high viscous PAV to be evenly mixed with other ingredients

The composition variation experiments of the addition of SeO_2 inorganic particle were made in two different methods. Firstly, the optical characteristics of the films were observed with the variation of inorganic SeO_2 component, while other ingredients were kept constant. Secondly, total amounts of AA and SeO_2 were kept constant at 4.0 g, while the compositions of AA and SeO_2 differentiated relatively when the photopolymer films were prepared. And the optical properties were detected afterwards. The compositions adopted in these experiments were shown in Table 1. Photopolymer films without mixing SeO_2 were prepared and optically observed as standard samples for comparisons with other films with SeO_2.

TABLE 1. Components of Photopolymer film

SeO_2 Content Variation Experiment (Experiment 1).

No.	Standard composition (water: 100 mℓ)	SeO_2	비고
Std.	PVA 10 g, AA 4 g, TEA 2 ml, Eosin Y 0.003 g		
1	PVA : 10.0 g AA : 4.0 g TEA : 2.0 ml Eosin Y : 0.003 g	0.5 g	-
2		1.0 g	-
3		1.5 g	-
4		2.0 g	-
5		2.5 g	separation
6		3.0 g	separation

AA + SeO_2 Total Content Variation Experiment (Experiment 2)

No.	Standard composition (water: 100 mℓ)	AA	SeO_2
Std.	PVA 10 g, AA 4 g, TEA 2 ml, Eosin Y 0.003 g		
1	PVA : 10.0 g TEA : 2.0 ml Eosin Y : 0.003 g	3.5 g	0.5 g
2		3.0 g	1.0 g
3		2.5 g	1.5 g
4		2.0 g	2.0 g
5		1.5 g	2.5 g
6		1.0 g	3.0 g

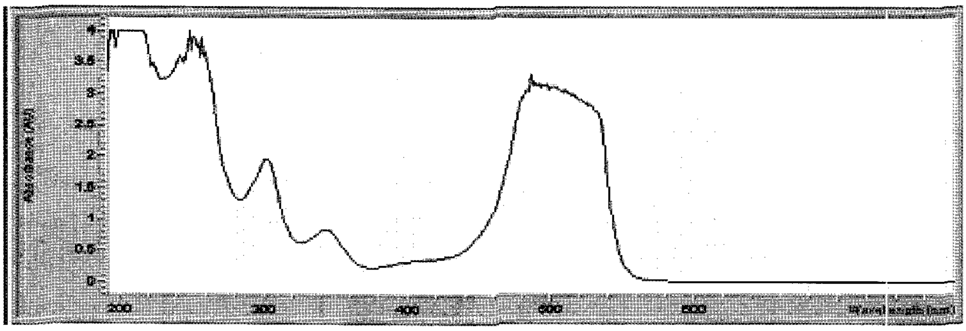


FIG. 3. Absorbance peak of Eosin Y (HP 8453 UV-Vis Spectrometer).

2.3 Optical characteristics measurement apparatus for photopolymer films

Most important optical characteristics for photopolymer films are DE (diffraction efficiency) and TE (transmittance efficiency). Angle selectivity, which is closely connected with storage density and recording sensitivity, may be the important optical character. High angular selectivity is required for photopolymer films for HDDS (holographic digital data storage), however, in low density holographic storage application, angular selectivity is not sensitive for recording property. DE was adopted as the reference property to decide the optical property of the photopolymer film in this experiment. There are two equations for the calculation of DE values. Equation (2-1) is derived from the ratio of the intensity of diffracted beam and the intensity of original incident beam of the laser light. In Equation (2-2) DE's are estimated from the ratio of the intensity of pure diffracted beam and the intensity of pure transmission beam in which scattered and diffracted beams before passing the photopolymer film, the reflected beam on the photopolymer surface, and the scattered beam inside the photopolymer film are not considered in this calculation. Equation (2-2) are usually adopted for DE calculation in most research with an assumption of having high TE value of the films, so Equation (2-2) was utilized for DE calculation in this research.

$$\mathrm{DE}\ (\%) = \frac{\text{Diffraction Beam Intensity}}{\text{Incident Intensity of the Applied laser light}} \times 100 \quad (2\text{-}1)$$

$$\mathrm{DE}\ (\%) = \frac{\text{Diffraction Beam Intensity}}{\text{Diffraction Beam Intensity} + \text{Transmission Beam Intensity}} \times 100 \quad (2\text{-}2)$$

The optical characteristics measurement apparatus used in this research was the DE measurement apparatus designed by PrizmTech Co. as was shown schematically in Figure 4. This apparatus was composed of the Ar-ion laser of maximum power of 100 mW and wavelength of 532 nm, spatial filter, lens, PBS (polarizing beam splitter), shutter, mirrors, power detector, and controller. In this research, the intensity of laser light was fixed at 50 mW, the opening and closing times of the shutter were fixed at 3s, 0.5s, and the 2θ were fixed at 40° to obtain maximum diffraction efficiencies. The intensity of laser light, the opening times of the shutter, and 2θ values may be modified by operating conditions for the best results.

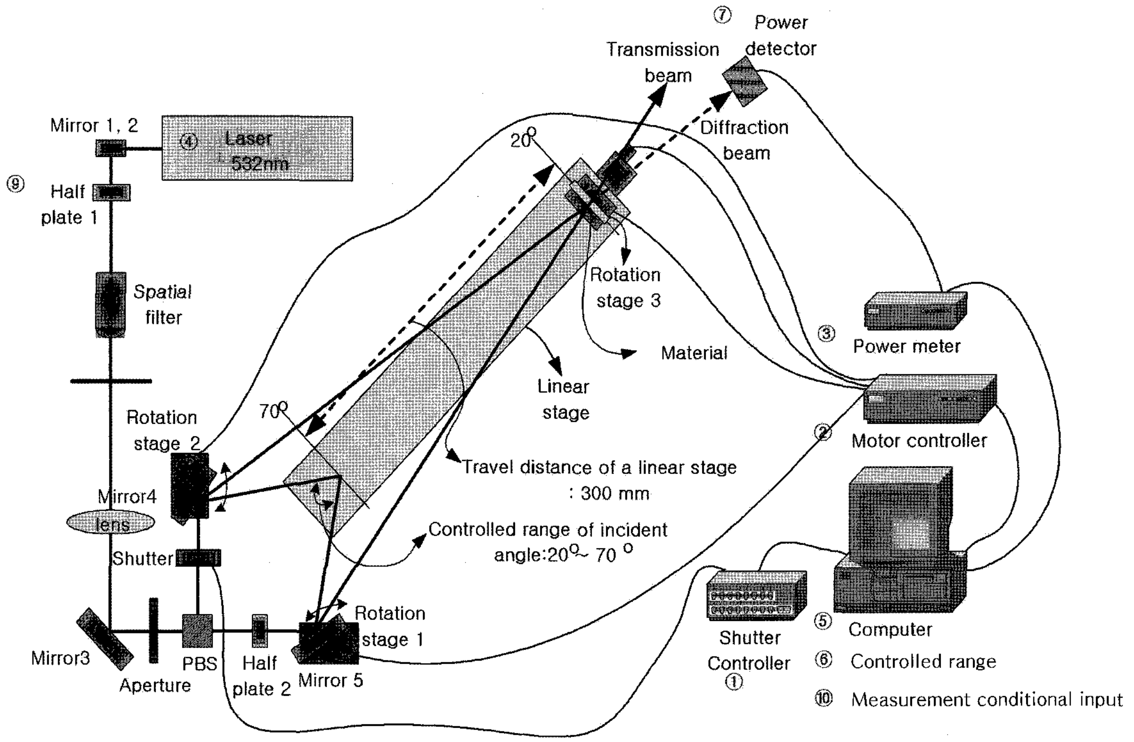


FIG. 4. Measuring systems for diffraction efficiency of photopolymer system
- Laser : 532 nm-Green Ar-ion Laser 100 mW
- Detector : Newport Optical Power Meter. Model 1830-c
- Sutter controller : SURUGA SEIKI F77-6
- Operating program : MPAS (by Prism Tech. Co., Ltd)

2.4 X-ray diffraction measurement

XRD is applied for determining distances between atoms and atomic layers by illuminating X-ray to the material and observing the constructive interference of the diffracted X-ray. Usually, XRD is used for crystalline materials, which have ordered arrays of atoms. In this research, XRD was adopted to examine whether added SeO_2 is in crystalline state or amorphous state. RINT2000 (Rigaku) generates X-rays, D3 system (Bede) is attached for data processing.

III. RESULTS AND DISCUSSION

3.1 Diffraction Efficiency (DE)

DE's were obtained by manipulating the measuring system (Figure 5) using MPAS operating program (S/W by Prism Tech. Co., Ltd). After exposing for 3 seconds, the shutter was closed, and the intensities of diffracted beams of reference beams were measured. The above sets of measurements were repeated up to 300 times to get DE value. The intensity of the beam was controlled at 50% of the maximum intensity of the laser (100 mW). DE's of samples in Experiment 1 (Table 1) were expressed in Figure 5.

In Figure 5, maximum DE's were observed at 100~150 seconds for the standard sample without SeO_2, DE's dropped up to 30% at measuring time of 900 sec (15 minutes). This is a typical behavior of DE of conventional photopolymers. By the addition of SeO_2, the maximum DE reached slower at 300 seconds, which suggests SeO_2 slow down the photopolymerization of monomers by absorbing and storing light energy. At the initial photopolymerization stage, density of exposed zone increase as the beam exposure continues. As the polymerization completed and the monomers turned into polymers, the density of the exposed zone stays constant, however the surroundings of the exposed zone keep polymerizing, so the density difference decreases. As the contents of SeO_2 increase, the DE's at 900 seconds increase because of the smaller decrease from the maximum DE values. This phenomenon can be explained as the effect of SeO_2, which first absorb light energy and then transfers to initiate polymerization. By storing light energy, the efficiency increase and the photopolymerization slow down to allow more monomer to transfer from dark area to exposed area. As SeO_2 increase, the final DE's (after 900 seconds) increase. Table 2 shows the final DE's for every samples.

In Sample No. 5 and 6, phase separations occurred during drying the photopolymer films because the amount of AA and SeO_2 which were in powder form exceeded the maximum solvation concentration in PVA. If the phase separations occurred, transparencies of the photopolymer films decrease, and the scattering of light and the diffraction of light detected together. The diffraction of Sample No. 5 and 6 could not be observed due to the scattering of the samples.

The components of the photopolymer samples of experiment 2 (table 1) were designed to determine optimum ratio of monomer and inorganic compound while avoiding phase separation between non-polar organic phase and polar organic and inorganic phases. With avoiding phase separation and scattering problems of the photopolymer films in DE measurements, the total amounts of powder components, AA and SeO_2, were fixed at the maximum value without the phase separation in Experiment 2. To get the optimum ratio between AA and SeO_2 several different ratios of AA/SeO_2 were selected and the DE's for each samples were observed and the experimental results were shown in Figure 6. For the sample 5 and sample 6 in experiment 1, DE's could not be measured because of the large scattering from the phase separation of added AA and SeO_2 with PVA binder. For the sample 5 and sample 6 in experiment 2, DE's were too small to be accurately observed due to the small amount of added photopoly-

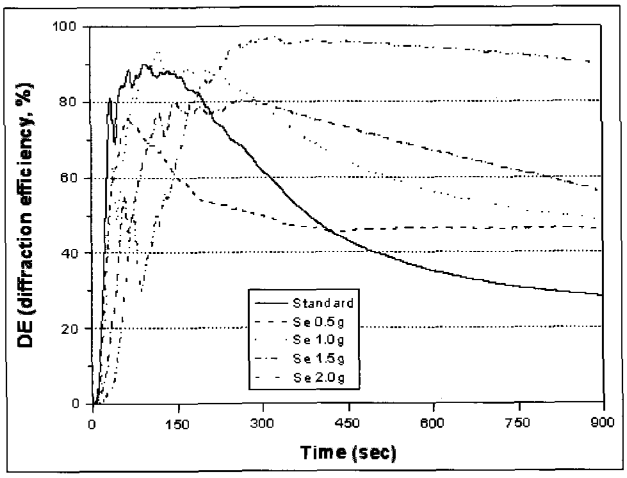


FIG. 5. Diffraction efficiency of samples in 1st experiment.

TABLE 2. Diffraction Efficiency of Samples after 15 minutes (Experiment 1).

No.	DE (diffraction efficiency, %)
Standard	28.5%
1	46.3%
2	48.8%
3	56.1%
4	88.8%
5	- (separation)
6	- (separation)

merizing monomer AA.

In experiment 2, DE's of sample 1, 2, and 3 showed stable growth and plateau after extended illumination past maximum DE values. However, sample 4 showed relatively late growth of DE compare to other samples. AA contents of sample 4 were smaller than other samples, because not enough AA were supplied for photopolymerization unless AA transfer from neighboring area by diffusion. So the low DE growth rates depended not only on the photopolymerization reaction rate but also on the diffusion rate of AA. The final DE values were summarized in Table 3 after 15 minutes of illuminations and post-curing of each sample, sample 2 of AA 3.0 g, SeO_2 1.0 g showed best DE characteristics and final stable values of over 90% continuously.

For the composition of sample 2 in experiment 2 which showed optimum composition in this experiment, long time illumination experiment to observe DE variation was conducted, in which illumination time was 10,000 seconds. DE of over 90% sustained throughout the illumination of 10,000 seconds, not like DE's of previous photopolymers without adding SeO_2 which were decreasing after 600 seconds. This stable tendency of DE values were shown for other samples, sample 1, and sample 3, of experiment 2. Figure 7 shows DE variation curve for sample 2 in experiment 2 during illumination of 9,000 seconds. The curve resembles a sine curve due to the combination of the monomer diffusion speed and the speed of photopolymerization.

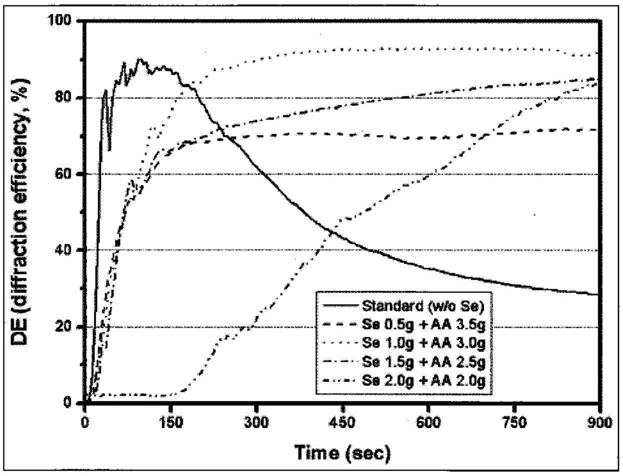


FIG. 6. Diffraction efficiency of samples in 2nd experiment.

TABLE 3. Diffraction Efficiency of Samples after 15 minutes (Experiment 2).

No.	DE (diffraction efficiency, %)
Standard	28.5%
1	71.7%
2	91.8%
3	85.0%
4	83.8%
5	- (no reaction)
6	- (no reaction)

3.2 XRD (X-ray diffraction)

In Figure 8 (a), X-ray diffraction peaks of pure SeO_2 inorganic crystals were shown in which sharp crystalline peaks of SeO_2 from 20° to 50° were clearly found. Figure 8 (b) shows the X-ray diffraction peaks of photopolymer samples without adding SeO_2. The diffraction did not show crystalline sharp peaks, instead there was a broad curve which was typical for amorphous polymers. In Figure 8 (c), the X-ray diffraction of photopolymer sample with SeO_2 was shown. The sharp crystalline peak which were shown in crystalline SeO_2 disappeared, however the peak at 20° increased greatly compare to (b). SeO_2 inorganic crystals were solvated in photopolymer since there was no crystalline SeO_2, and the SeO_2 mixed with organic photopolymer in smaller scale than X-ray crystalline size which were in nano-scale, and the polymer ordering showed improvement, which was shown through the increase of the peak at 20°.

3.3 Contraction rate of Photopolymer Film on Photopolymerization

Measuring thicknesses of photopolymer films were conducted to estimate the contraction rate of photopolymer films during photopolymerization at illumination. Digimicro (ZC-101, NIKON Co.) was adopted for thickness measurements, which had accuracy of 10^{-4} mm, and the effective thickness were observed in 10^{-2} mm order in this experiment.

Thicknesses of the photopolymer films before illumi-

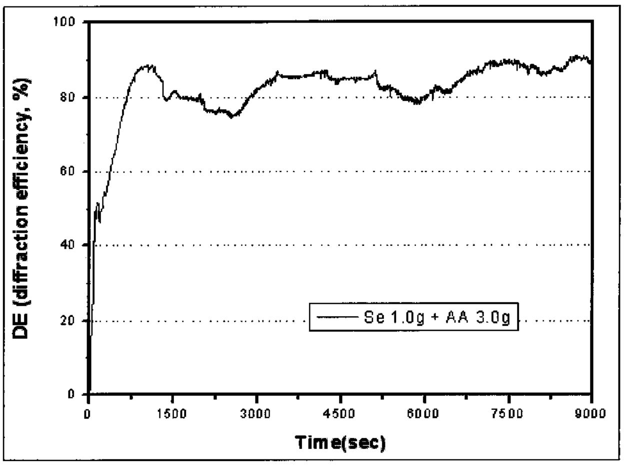
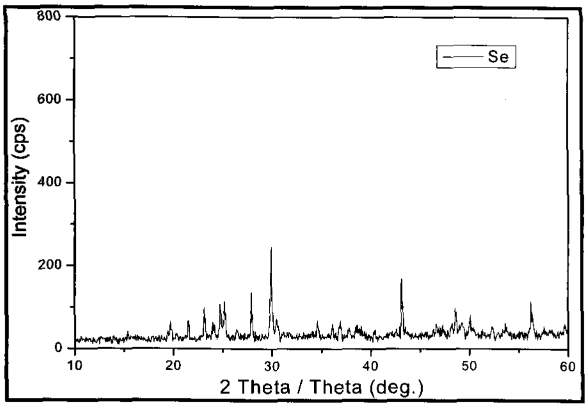
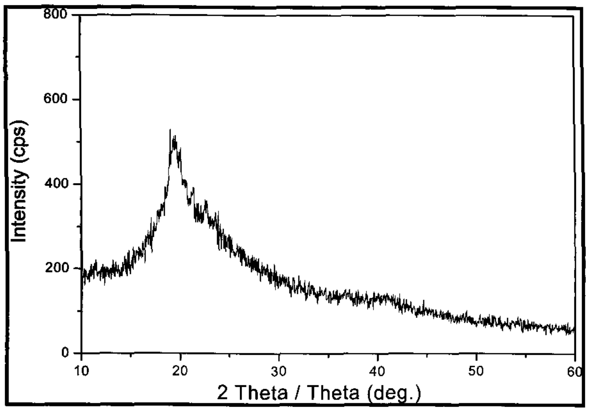


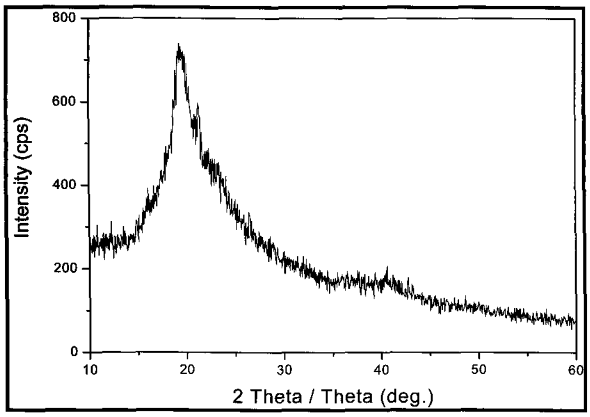
FIG. 7. The change of DE during 9,000 sec.



(a) SeO_2



(b) Film without SeO_2 crystals



(c) Film with SeO_2 crystals

FIG. 8. XRD peak of SeO_2 crystals and photopolymer film.

nation were summarized in Table 4. The films were made by dropping 2.0 ㎖-solution of photopolymer mixture on the slide glass and by drying for 72 hours at room temperature of relative humidity of 40%. The average thickness of the films was 100±10 ㎛, and the flatness of the films was so good as to minimize the scattering and the reflection at the film surface. Films were made with 100 ㎛ thickness for faster drying and better surface quality. For preparing 100 ㎛ films, 2 ㎖ photopolymer mixture was dropped on the glass. By volumetric calculation, the thickness of the film before drying should be 1.2 mm.

TABLE 4. Thickness of Photopolymer Film. (unit: ㎛)

No.	Thickness	No.	Thickness	No.	Thickness
1	97	8	101	15	91
2	95	9	95	16	95
3	94	10	97	17	97
4	98	11	99	18	96
5	95	12	95	19	98
6	98	13	94	20	95
7	96	14	98		
Average	96.2 ㎛		Deviation	± 3 ㎛	

TABLE 5. Thickness of Film by Photopolymerization. (unit: ㎛, %)

No.	Thickness	After	No.	Thickness	After
1	97	105	11	99	111
2	95	101	12	95	103
3	94	101	13	94	102
4	98	106	14	98	106
5	95	101	15	91	95
6	98	106	16	95	102
7	96	105	17	96	103
8	101	113	18	97	105
9	95	100	19	98	106
10	97	103	20	95	105
Average	103.9		Conversion rate		+ 8%

When photopolymerization proceeded by illumination, AA monomers were photopolymerized to produce poly-acrylamide. Synthesized poly-acrylamide chains were intercalated with PVA polymer binder because the monomers were permeated inside the PVA polymer binder. By this photopolymerization, monomers were used and so the concentration of the monomer decreases. During the photopolymerization reaction, monomer from the surrounding area migrated to the illuminated area where monomers are consumed for the photopolymerization and so the concentration of the monomer was low. By this migration and photopolymerization of the monomers, the photopolymer film expands during illumination as shown in Table 5. In other photopolymers using PMMA or epoxy as a binder, contraction of the photopolymer film on illumination observed due to photopolymerization of monomers soaked inside the binder, and this contraction sometimes became a cause for crack and surface problems like flatness and transparency. The photopolymer in this research which was using PVA as a binder expanded on illumination. High flexibility of PVA polymer enables monomer to migrate into the illuminated area from dark area of destructive interferences where photopolymerization did not take place. The flexible PVA binder expanded when AA monomers migrated to the illuminated zone. There was neither expansion nor contraction observed upon exposure of 10 minutes to sun light, which suggested that there were little changes of volume during photopolymerization of AA.

IV. CONCLUSION

From this research, overall optical characteristics of PVA/AA based photopolymer were improved by the addition of SeO_2, and the optimum amount of SeO_2 was determined. New findings can be summarized as follows;

1. By the addition of SeO_2, the maximum DE reached slower at 300 seconds, which suggests SeO_2 slow down the photopolymerization of monomers by absorbing and storing light energy. This slow-down of photopolymerization enhanced the maximum DE by allowing more migration of AA monomers.
2. As the contents of SeO_2 increase, the DE's at 900 seconds increase because of smaller decrease from the maximum DE values. This phenomenon can be explained asthe effect of SeO_2, which first absorbs light energy and then transfers to initiate photopolymerization. This energy transfer gave time for AA monomer to migrate into the illuminated zone.
3. The final DE value after 15 minutes of illuminations and post-curing of sample 2 (AA 3.0 g, SeO_2 1.0 g) showed best DE characteristics and final stable values of over 90% continuously.
4. DE variation curve for sample 2 in experiment 2 during extended-time illumination of 9,000 seconds resembles a sine curve due to the combination of the monomer diffusion and the photopolymerization.
5. In the XRD peaks of figure 8. The sharp crystalline peaks which were shown in crystalline SeO_2 disappeared, however the peak at 20° increased greatly compared to that of pure photopolymer. SeO_2 inorganic crystals seemed to be solvated in photopolymer since there was no crystalline SeO_2. The SeO_2 enhanced the crystallization of AA monomer during photopolymerization.
6. High flexibility of PVA polymer enables monomer to migrate into the illuminated area from dark area of destructive interferences where photopolymerization did not take place. The flexible PVA binder expanded of about 8% when the photopolymerization took place. This expansion also supported active migration of AA monomer form dark to illuminated area.

ACKNOWLEDGEMENTS

The present Research has been conducted by the Research Grant of Kwangwoon University in 2006.

The present Research has been conducted by Seoul R&BD Program (Grant No. 10583).

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