Fabrication of contact lens containing high-performance wire grid polarizers

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Abstract
Contact lens containing high-performance wire grid polarizers (WGPs) were fabricated in this study. The base polymeric membrane was synthesized by copolymerization of 2-hydroxyethyl methacrylate, 3-[tris(trimethylsiloxy)silyl]propyl methacrylate, 1-vinyl-2-pyrrolidinone, methyl methacrylate, and ethylene glycol dimethacrylate. The nanopattern was formed by nanoimprint lithography using a UV curable rubber-toughened epoxy resin on the base membrane. Two consecutive angled aluminum evaporations were performed on the nanopattern, followed by reactive ion etching to form the WGP. The WGP on the membrane was then covered by another layer of the base polymeric material to form the final contact lens. This membrane demonstrated high performance as a polarizer, confirmed by measuring transmittance of transverse magnetic polarized light and transverse electric polarized light. This membrane also demonstrated good performance as contact lens in terms of modulus, water content, and oxygen permeability.

Keywords wire grid polarizer, contact lens, nanoimprint lithography, angled evaporation

Introduction

Polarizers are essential components of many electronic devices and are used in a variety of applications such as in LCDs and 3D viewing glasses. Conventional polarizers are manufactured using iodine doped poly(vinyl alcohol) film. However, there is serious loss of light transmittance in these materials due to absorption of light by this type of polarizer film. It is also difficult to use most common polarizers in very small electronic devices. Therefore, wire grid polarizers (WGPs) have received a large amount of research attention as a next generation category of polarizers.[1-13] WGPs are formed by arranging nanometer-scale metal wires on transparent substrates. Transverse magnetic (TM) polarized light is transmitted through WGPs, while transverse electric (TE) polarized light is reflected. Because WGPs can be formed on very small size scales, it is possible to integrate them into new microelectronic devices. Due to the design push towards microelectronic devices, WGPs could be effective components in new devices in fields such as microscopy, beam splitters, and other imaging systems, as well as leading to brighter LCD screens due to recycling light. However, these practical applications require further investigation of WGP fabrication before becoming a reality.[1-13]

In order to fabricate a WGP, a metal grating must be well arranged on the substrate. Nanoimprint methods are among the easiest methods to form the grating structure. Nanoimprint lithography has received a great deal of attention as a next generation lithographic technique that could replace current lithographic methods used in the electronics industry. Using nanoimprint lithography, several hundred nanopattern copies can be formed using the same template. Compared with traditional photolithography, nanoimprint lithography is far superior in terms of production speed and economic feasibility. Development of these new lithographic techniques is an ongoing process in the electronics industry.[14-20]

WGPs have been manufactured using nanoimprinting, as reported previously in journals and patents.[21-30] In order to demonstrate their high performance as polarizers in the visible light spectrum, the period of the WGP array must be less than 100 nm. Even though WGPs with periods on the order of 10^1 nm have already been reported, it is still very difficult to reliably form nanopatterns with 100 nm periods without defects. To that end, we have found a novel method to make WGPs with 110 nm periods by using a 220 nm period nanopattern in consecutive angled aluminum evaporation steps and reactive ion etching.[31] Because this WGP has a viewing angle problem, we have also reported a 90 nm period WGP fabricated in a similar manner with a 180 nm period nanopattern.[32]

Contact lenses are traditionally made using poly(dimethyl methacrylate) (PMMA) as a base material, but they can be improved greatly by using poly(2-hydroxyethyl methacrylate) (PHEMA).[33-35] While PHEMA has many favorable physical properties for use in contact lenses, it has a low oxygen permeability which is not suitable due to the need for oxygen flow from air to the cornea. Because blood vessels are not connected to the human cornea, oxygen

must be supplied from the atmosphere. Therefore, the oxygen permeability of contact lenses must be above 24 DK during the day. In addition, in order to use the contact lens while sleeping, the oxygen permeability of the contact lens must be above 87 DK.[35] A great deal of research has been conducted in the last 20 years to increase oxygen permeability of contact lenses. Many hydrophilic monomers were copolymerized with PHEMA to fabricate contact lenses. Recently, a silicone-containing hydrogel moiety has been shown to increase oxygen permeability.[36-40]

In our previous study, embedding a WGP into a flexible material was attempted for the first time.[41] Polydimethylsiloxane (PDMS) was used as a flexible material. Because PDMS get the good biocompatibility and oxygen permeability, it could be said that the analogical contact lens containing WGP was fabricated. Because the WGP used in the study got the 180 nm period, the serious problem in the viewing angel was occurred.[32]

In this study we fabricated the WGP which got the 90 nm period nanopattern and had not the viewing angle problem. And the contact lens containing WGP was fabricated using materials which were used to manufacture the real contact lens. For the first time the real contact lens containing high performance WGP was introduced in our study.

Experimental

Materials

2-hydroxyethyl methacrylate (HEMA), 1-vinyl-2-pyrrolidinone (NVP), methyl methacrylate (MMA), ethylene glycol dimethacrylate (EGDMA), 3-[tris(trimethylsiloxy)silyl]propyl methacrylate (TSPMA), 2-hydroxy-2-methylpropiophenone (HMPP), triarylsulfonium hexafluoroantimonate salt (50 wt% in propylene carbonate) (TSHA), and propylene glycol monomethyl ether acetate (PGMEA) were purchased from Aldrich Chemical (St. Louis, MO, USA). HMPP was used as a photoinitiator for the polymerization of the base membrane materials. TSHA was used as a photoacid generator for the nanoimprinting process. Bisphenol F-type epoxy resin (YDF-170) and NBR-based epoxy resin (R-1309) were purchased from Kukdo Chemical (Korea). The adhesion promoter Silquest A-187 silane ((3-glycidyloxypropyl)trimethoxysilane as the main ingredient) was purchased from Crompton Co. (Lisle, IL, USA). The mold release agent 1H,1H,2H,2H-perfluorodecyltrichlorosilane (FDTS) was purchased from Gelest Inc. (Morrisville, PA, USA).

Instruments

The nanoimprinting was conducted using a Nanonex NX2000 imprinting tool (Monmouth Junction, NJ, USA) with vacuum capability and an ELC-430 UV curing system by Electro-

Lite Corporation (Bethel, CT, USA). The deposition of aluminum was conducted using electron-beam evaporation. Reactive ion etching (RIE) was conducted using an LAM 9400 tool (LAM Research Corporation, Fremont, CA, USA). Transmission of TM and TE was measured the equipment which we made ourselves with Nikon Eclipse TE300 microscope (Tokyo, Japan), HR4000CG spectrometer (Ocean Optics Inc., Dunedin, FL, USA), and TE and TM were measured more than 5 times and the value was obtained by averaging the results. The viewing angle was measured by tilting the sample. Scanning electron microscopy (SEM) was conducted using a Hitachi SU8000 scanning electron microscope (Tokyo, Japan). An Instron5944 universal testing machine (Norwood, MA, USA) was used to estimate the tensile strength, elongation, and modulus. The length, width, and thickness of specimen were 60 mm, 10 mm, and 100 μm, respectively. A minimum of six specimens were tested, and the average results were reported. An Atago Nar 1T Abbe refractometer was used to measure the reflective index at 22 °C. LED (wavelength of D-Line) was used as a light source in the refractometer. The oxygen permeability was measured using a 201T Permeometer (Jurong East, Singapore) at 35±0.5 °C. Before measuring the oxygen permeability, the membrane was soaked in a solution of 0.142 M NaCl for at least 24 h. A gravimetric method was used to measure the water content, which was estimated by measuring the weight of the swollen state in water (W_s) and the weight of dried state (W_d).

Water content (%) = $(W_s - W_d)/ W_s \times 100$

Polymerization of the component materials for contact lens

HEMA, NVP, MMA, TSPMA, EGDMA were copolymerized with HMPP as a photoinitiator on a Teflon mold. The stock solution was prepared by mixing HEMA (17.00 g, 0.131 mol), MMA (0.200 g, 2.00 mmol), NVP (0.900 g, 8.10 mmol), EGDMA (0.100 g, 0.504 mmol) and TSPMS (1.80 g, 4.26 mmol). HMPP (0.100 g, 0.609 mmol) was then added to the stock solution. The polymerization was conducted under UV light for 20 min at room temperature. The synthesized polymer was cast as a film, which was used for estimating the mechanical properties. In order to form the base membrane for the contact lens, the amount of the resin was adjusted to obtain a final thickness of 50 μm.

Nanoimprint process

In order to achieve easy release from the original silicon oxide master mold after finishing the imprinting process**,** the mold was vapor-coated with FDTS. And Silquest A-187 silane was used as an adhesion promoter to increase the adhesive strength between the base membrane and the nanopattern. The nanoimprint resist formulation was made by dissolving the bisphenol F-type resin and NBR-based epoxy resin in PGMEA with mixing ratio 85:15

(YDF-170:R-1309),[42] and was followed by the addition of the TSHA (3.0 wt% to the epoxy resin). Using spin coating, a very thin film was formed on the base membrane. The thickness of film was controlled by changing the concentration of the resin. By using larger amounts of PGMEA, a thinner nanoimprint could be obtained. The imprinting pressure was typically less than 50 psi due to the low viscosity of the epoxy resin. 10 min of reaction time was sufficient to cure the epoxy resin.

Aluminum deposition

The deposition mount was tilted to 40° to deposit aluminum on the sidewalls of the imprinted nanopattern, but it was not deposited on the base of the trench. The rate of aluminum deposition was 0.5 nm/s. In order to deposit aluminum on both sidewalls of the nanopatterns, the aluminum deposition process was repeated from the opposite direction,[31,32] The final aluminum thickness on both sidewalls was controlled to be 20 nm. This process is shown in Figure 1.

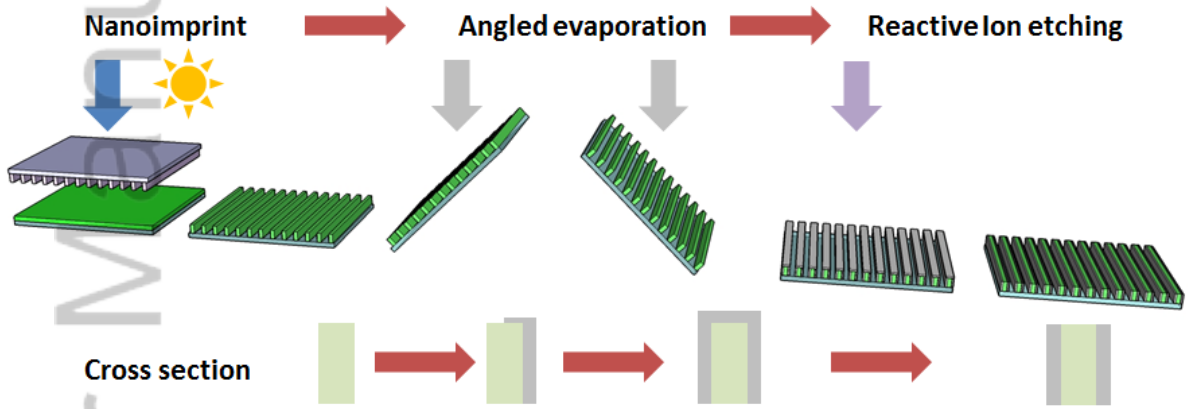


Figure 1. Schematic of double angled aluminum evaporation and reactive ion etching in the WGP formation process.

Reactive ion etching process

The aluminum layer deposited on top of the nanopattern needed to be removed in order to form the grid pattern. We used an anisotropic plasma etching process to selectively remove the top aluminum layer while leaving the aluminum on the sidewalls of the grating relatively intact. Aluminum etching was performed using a LAM 9400 tool (Fremont CA, USA). The

detailed process has been previously reported,[31,32] The overall fabrication process of the WGP is illustrated in Figure 1.

Results and Discussion

Fabrication of contact lenses containing WGP

In this study, we fabricated contact lens containing high performance WGPs for practical use, aiming to achieve not only high polarization performance but also high performance as a contact lens. Therefore, we used materials that demonstrate both excellent mechanical properties and favorable oxygen permeability. First, we selected the materials for fabricating the contact lens, and then made the membrane via polymerization. Many different formulations have been used to make contact lens. Among our previous research results, we selected a lens formulation which demonstrates excellent physical properties. Therefore we conducted the copolymerization of 2-hydroxyethyl methacrylate (HEMA), 1-vinyl-2-pyrrolidinone (NVP), methyl methacrylate (MMA), 3-[tris(trimethylsiloxy)silyl]propyl methacrylate (TSPMA), and ethylene glycol dimethacrylate (EGDMA) to form the contact lens, and used 2-hydroxy-2-methylpropiophenone (HMPP) as a photoinitiator**.** The formulation used in this study is shown in Table 1.

Table 1. The formulation of contact lens fabricated in this study.

	Amount (mmol)	Mol %	Weight (g)	Weight %
HEMA	131	89.8	17.0	85.0
NVP	8.10	5.55	0.900	4.50
MMA	2.00	1.37	0.200	1.00
TSPMA	4.26	2.92	1.80	9.00
EGDMA	0.504	0.346	0.100	0.500
HMPP	0.609		0.100	

HEMA has been one of the mostly widely used materials for making contact lens since they were first being introduced in 1962. PHEMA has good physical properties for use in contact lenses; because the pendent hydroxyl group in PHEMA can interact with water, contact lenses made with this material position themselves properly on the eye. MMA was used to reinforce the desired mechanical properties, and EGDMA served a similar purpose through crosslinking reactions. MMA and EGDMA can aid the manufacturing process in maintaining the shape of the lens. NVP is a hydrophilic monomer that can increase the water content of the contact lens. TSPMA was used to increase the oxygen permeability because it has a large number of silicon groups at the side chains. HMPP was used as a photoinitiator at 0.5 weight %.

All of these components were mixed thoroughly, and the photopolymerization was carried out using UV irradiation on the Teflon mold (1.50 cm × 1.50 cm). The thickness of the membrane was controlled to 50 ± 2 μm after polymerization was completed. Nanoimprinting was then carried out on this membrane using an epoxy resin at a mixing ratio of 85:15 (YDF-170:R-1309), which was selected due to its high adhesive strength. The original silicon oxide mold (1.10 cm × 1.10 cm) used for nanoimprinting featured a nanopattern with a 180 nm period, a 110 nm linewidth, and a 200 nm height. Before the nanoimprinting, Silquest A-187 silane was vapor-coated on the membrane to increase the adhesive strength between the membrane and the nanopattern. In the early stages of this study, we used both surface modification of the base membrane by oxygen plasma and Silquest A-187 treatment to increase the adhesive strength. However, because the results showed that Silquest A-187 treatment alone was enough to achieve the desired adhesive strength, only Silquest A-187 treatment was carried out to increase the adhesive strength between the base membrane and the nanopatterns. Surface treatment with 1H,1H,2H,2H-perfluorodecyltrichlorosilane (FDTS) was done on the silicon oxide mold in order to better control the demolding process.

A scanning electron microscopy (SEM) image of the formed nanopattern after nanoimprinting is shown in Figure 2(a). The nanopattern was formed with a 180 nm period, 70 nm linewidth, and 200 nm height. After the nanopattern was tilted 40°, aluminum was then deposited by evaporation. This process was done again from the exact opposite direction. The thickness of the deposited aluminum on the sidewall was controlled to be 20 nm, as our previous studies show that the WGP has the best performance as a polarizer in the case of 20 nm aluminum thickness.[31] An SEM image is shown in Figure 2(b) after aluminum deposition. The upper side of the aluminum was removed by reactive ion etching, leaving aluminum only at the side walls. Finally, the WGP was fabricated with a 90 nm period and 20 nm linewidth. An SEM image of the WGP is shown in Figure 2(c). A schematic diagram of the WGP fabrication is shown in Figure 1. The same formulation used to make the base membrane was used for covering the formed WGP. The thickness of the membrane covering the WGP was controlled to 50 ± 2 μm, and the final WGP-containing membrane had a total thickness of 100 ± 4 μm. A photo of the final lens is shown in Figure 3(a) and an SEM image of the cross section is shown in Figure 3(b). In Figure 3(b), the bottom layer is the base membrane and the upper layer is the covered membrane. The WGP is located between the two layers.

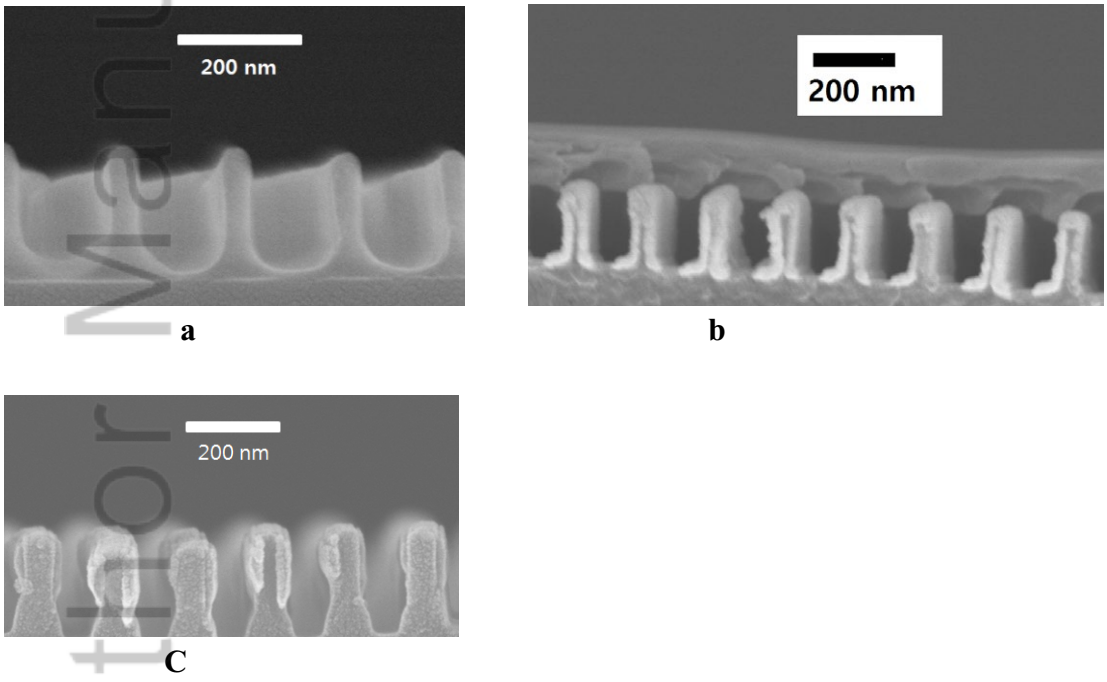
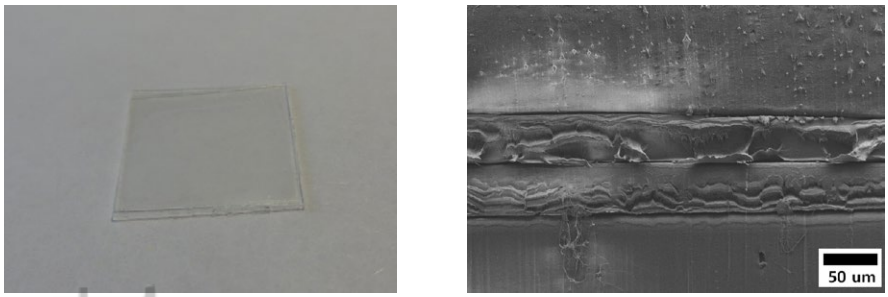


Figure 2. SEM images of nanopatterns (**a)** after nanoimprinting, (**b**) after angled aluminum evaporation, and (c) after reactive ion etching.



a b

Figure 3. (a) Photo of the lens containing the WGP, and (b) SEM image of the cross section of the lens.

Physical properties of the lens containing WGP

The physical properties of the fabricated membrane relevant to use as a contact lens (such as tensile strength, elongation, modulus, refractive index, water content, and oxygen permeability) were measured. The fabricated membrane behaved like a hydrogel, meaning that the physical properties are influenced by humidity; as a result, measurements were recorded at two different relative humidity (RH) levels of 16% and 98%. When the contact lens was inserted in eye, it was wet through with tear. Considering this condition the physical property of the sample was estimated at 98% RH. And in order to compare the data of general condition, it was also estimated at 16% RH. Prior to measurement, the specimens were held at these conditions for 6 hours in order to reach equilibrium. The results are presented in Table 2.

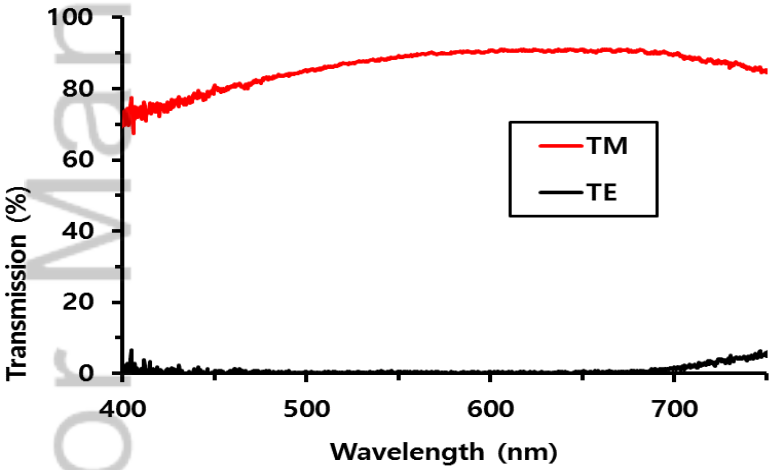
Table 2. The physical properties of the contact lens in this study

Relative humidity	Tensile strength (MPa)	Elongation (%)	Modulus (MPa)	Refractive index	Water content (%)	Oxygen permeability (DK)
16 % RH	1.82±0.09	54.7±3.1	3.33±0.18			
98 % RH	0.27±0.02	172.5±8.5	0.16±0.01	1.435±0.0005	37.8±1.7	27.4±1.3

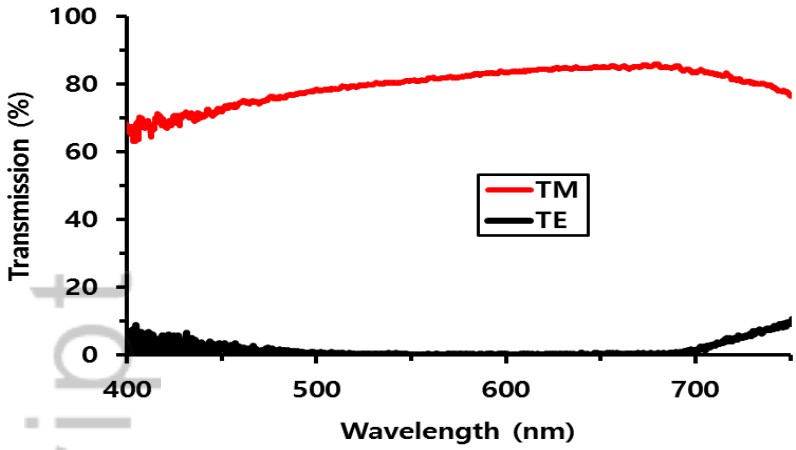
The results in Table 2 show that this material has suitable physical properties for use as a contact lens. In particular, the oxygen permeability of this membrane is 27.4 DK, which is superior to that of the general PHEMA-based material. This feature can help to decrease oxygen-related malfunctions of the lens.

The results showed that the film formed in this study got enough tensile strength comparing with the commersial soft contact lens, and its elongation and modulus were in the range of the general soft contact lens.[43-45] The water content was increased proportionally with the amount of NVP in the formulation, and the oxygen permeability was also increased proportionally with it.[46] Therefore, if the NVP amount is increased in the formulation, water content and oxygen permeability may be incresed. But because its increase bring to reducing of tensile strength and modulus, it cause hardship in the lens manufacture and the nanoimprinting process in this study. Therefore we think that the present formulation is the optimum condition.

TE and TM values of this membrane were measured in order to estimate its potential as a polarizer, and the results are shown in Figure 4.



a



b

Figure 4. TE and TM curves of contact lenses with nanoimprinted WGP, under RH of (**a**) 16% and (**b**) 98%.

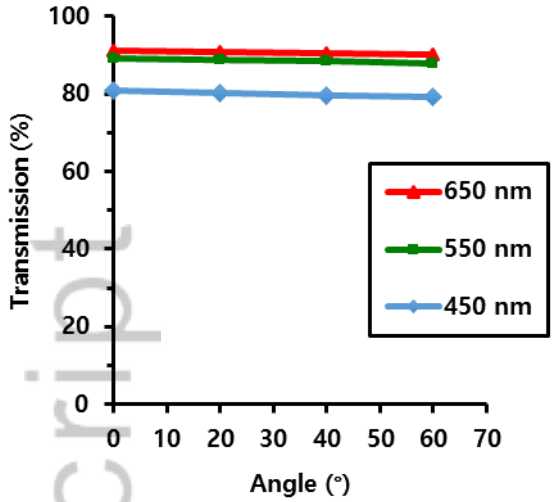
In the results shown in Figure 3, the TM values in the visible wavelength region were around 80% transmittance at both 16% RH and 98% RH, while the TE values were almost 0% in both cases. This result shows that this membrane can act as an effective polarizer. The TM/TE ratio is defined as the extinction ratio, and its value as a function of excitation wavelength is presented in Table 3.

Table 3 Measured optical transmission (%) of TM/TE polarized light through nanopatterned rubber-toughened epoxy/aluminum WGP.

Relative humidity	450 nm	550 nm	650 nm	750 nm
16 % RH	80.9/0.01 8090	89.2/0.10 892	91.1/0.10 911	85.1/4.78 17.8
98 % RH	72.9/1.89 38.6	81.2/0.13 625	85.1/0.22 387	77.0/9.26 8.32

In Table 3 resuts, in case of 16% RH the highest extinction ratio showed at 450 nm. Because the extintion ratio largely depend on the TE value, even though the maximun TM value showed at 650 nm, the highest extintion ratio was shown at 450 nm where the lowest TE value showed. In the case of 98% RH the highest extintion ratio showed at 550 nm. The humidity increase brings to decrease the TM value and increases the TE value. We think that these results are due to the light scattering by water.

Another important property of a polarizer is the viewing angle. The transmission of TM light through the polarizer was measured at different viewing angles at wavelengths of 650, 550, and 450 nm. The results are shown in Figure 5. And the measured optical transmissions (%) of TM/TE polarized light through the WGP at different angle are shown in Table 4.



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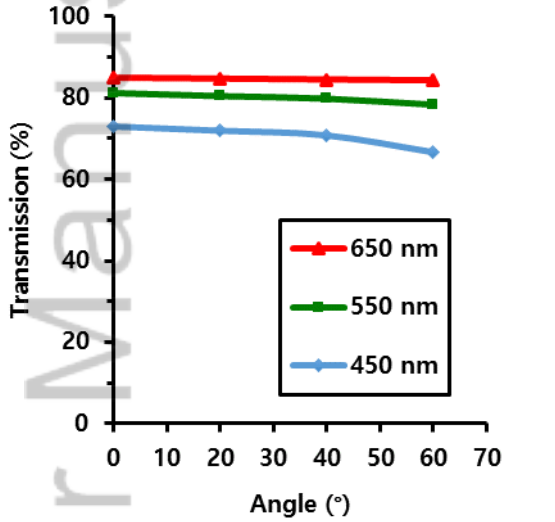

b

Figure 5. The transmission of TM light through the nanoimprinted WGP lens at different angles, at (**a**) 16% and (**b**) 98%.

Table 4. Measured optical transmission (%) of TM/TE polarized light through the WGP at different angle

	450 nm			550 nm			650 nm		
Angle (°)	20	40	60	20	40	60	20	40	60
16% RH	80.3/0.02 4015	79.7/0.01 7970	79.3/0.01 7930	88.8/0.11 807	88.5/0.08 1106	87.8/0.09 975	90.8/0.11 825	90.5/0.09 1006	90.2/0.10 902
98% RH	71.9/1.42 50.6	70.8/0.11 644	66.8/0.17 405	80.5/0.12 671	79.9/0.11 726	78.5/0.18 436	84.8/0.21 404	84.5/0.22 384	84.2/0.20 421

In Figure 5 and Table 4, at 16% RH, no decrease in transmission was observed at less than a 60 degree viewing angle. At 98% RH, a minor decrease in transmission occurred at 450 nm. Therefore, we can conclude that this WGP has a wide viewing angle.

Water content and oxygen permeability of the contact lens sample containing the WGP were estimated and those values were compared with the value in Table 2. The estimated water content was 36.3 ± 1.9 %. It means that only 4% was reduced. We concluded that embeding of the WGP did not have much effect to the water content. But the estimated oxygen permeability of the contact lens sample containing the WGP was reduced from 27.4±1.3 DK to 21.5±1.5 DK. This result showed that embeding of the WGP had an effect to the oxygen permeability. And we think that 21.5 DK is not so bad value for contact lens but it need to be improved.

Conclusion

Contact lens containing WGPs were fabricated in this study. Copolymerization of HEMA, MMA, NVP, TSPMA, and EGDMA were used to synthesize the base membrane for the lens. In making the WGP, a 180 nm period nanopattern was formed using rubber-toughened epoxy and nanoimprinting on the base membrane, and the WGP was formed by two angled aluminum evaporation steps followed by reactive ion etching. This WGP had a 90 nm period and a 20 nm linewidth. The same polymeric compound used to make the base membrane was then used to cover the WGP. The formed membrane has very good physical properties for use as a contact lens, as well as favorable optical properties for use as a polarizer.

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