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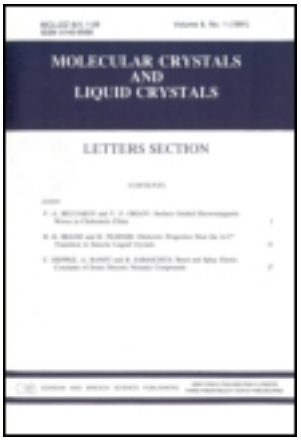

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Heat Resistant Polymer Matrix Containing Acrylo-Polyhedral Silsesquioxane for Erbium-Doped Waveguide Amplifier Applications

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Heat Resistant Polymer Matrix Containing Acrylo-Polyhedral Silsesquioxane for Erbium-Doped Waveguide Amplifier Applications

WOOK HYUN KIM,[1] SHI-JOON SUNG,[1] MYUNG-SEOK CHOI,[2] JONG TAE KIM,[1] AND YOON SOO HAN[3,*]

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*We report on the fabrication of fluorinated polymer film as host material for erbium ions (Er^{3+}) with a goal of achieving sufficient thermal stability, optical clarity and a chemical resistance to withstand typical fabrication processing. Precursor solutions were prepared using 2,2,3,3,4,4,5,5-octafluoropentyl acrylate as a fluoromonomer, tetrahydrofurfuryl acrylate as a solubility enhancer, Ebecryl 220 as a cross-linking agent, acrylo-polyhedral oligomeric silsesquioxane as a heat-resistance improver and Darocur 4265 as a radical photoinitiator with various weight ratios. F**luoro**polymer films prepared from the precursor solution had excellent transmission properties (low transmission losses less than 2% over the visible and near-infrared regions) and high thermal decomposition temperatures (greater than 350 °C). Er^{3+}-doped precursor solution was also prepared by adding of erbium(III) trifluoromethane sulfonate as an erbium source. The crosslinked, patterned and Er^{3+}-doped fluoropolymer films were successfully fabricated using the Er^{3+}-doped precursor solution by both micromolding in capillaries and soft-imprint lithography on glass substrates for Er^{3+}-doped waveguide amplifier applications.*

Keywords Acrylo-polyhedral oligomeric silsesquioxane; erbium; fluoropolymer; soft lithography

Introduction

In recent years, erbium(Er^{3+})-doped polymer waveguide amplifiers are attracting much attention because of the infrared emission properties of the Er^{3+} ions at approximately 1,550 nm resulting from its intra-4*f* transition, a standard wavelength used in telecommunication systems, and the potential of integration with several functionalities that are critical in integrated optical devices [1]. They can be used to compensate for signal losses, which include waveguide, coupling or intrinsic intensity losses [2–5]. To achieve greater

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gain per unit length in Er^{3+}-doped waveguide amplifiers (EDWAs), the concentration of Er^{3+} ion in EDWAs should be approximately 10 to 20 times higher than that in Er^{3+}-doped fiber amplifiers (EDFAs) [6]. However, at higher Er^{3+} ion concentration, energy transfer processes such as upconversion or energy migration results in decreasing the luminescence quantum efficiency of the $^4I_{13/2}$ excited-state by aggregation of Er^{3+} ions [7–10]. Consequently, molecular level dispersion of Er^{3+} ions is important for decreasing the concentration quenching by ion-ion interactions. Silica based inorganic matrices have been fabricated by high-temperature processes such as the flame hydrolysis deposition and chemical vapor deposition, that may cause clustering of Er^{3+} ions. On the contrary, by using polymers as a host matrix for Er^{3+} ions, a very high doping level of Er^{3+} ions can be achieved without causing a significant ion-cluster effect, thus polymer matrices are useful host materials for Er^{3+} ions [11,12]. The use of polymeric hosts for the fabrication of optical waveguide amplifiers offers many advantages, including good solubility, low processing temperature, low fabrication costs, simplified processing steps and compatibility with various processing techniques for patterning. In addition, various optical parameters—such as the refractive index, birefringence and thermal stability—can be easily controlled through blending and copolymerization, leading to superior device performance [13,14]. However, when conventional polymers are used for waveguide fabrication, they tend to exhibit high optical propagation losses, particularly at 1,550 nm, which corresponds to the telecommunication wavelength, because of the vibrational states of the O—H and **C—H** bonds. Such loss processes can be minimized by replacing the hydrogen atoms with heavier atoms through deuteration or fluorination of the polymer host material, causing the frequencies of the relevant vibrational overtones to shift [15,16].

Low thermal stability of polymeric hosts is another limitation on their application to Er^{3+}-doped waveguide amplifiers. Polyhedral oligomeric silsesquioxane (POSS) is an organic–inorganic hybrid material. This class of silicon compounds has the empirical formula $RSiO_{1.5}$, where R denotes an organic substituent. The incorporation of POSS units into polymer matrix by chemical bonding improves the polymer properties such as thermal stability, oxidation resistance, surface hardening and mechanical properties [17].

This study focuses on the fabrication of fluorinated polymer film as Er^{3+} host material with a goal of achieving sufficient thermal stability, optical clarity and a chemical resistance to withstand typical fabrication processing. For this reason, precursor solutions containing an acrylo-POSS were prepared in various compositions, and their thermal and optical properties were investigated. Cross-linked, patterned and Er^{3+}-doped fluoropolymer films were also fabricated by both soft-imprint lithography and micromolding in capillaries (MIMIC), and their pattering properties were compared.

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Experimental

Materials and Analytical Instruments

Figure 1 shows the structures of chemicals used for the Er^{3+}-doped fluoropolymer films. 2,2,3,3,4,4,5,5-Octafluoropentyl acrylate (OFPA) as a host polymer source, tetrahydrofurfuryl acrylate (THFA) as a solubility enhancer and erbium(III) trifluoromethane sulfonate (ErTFMS) as an erbium source were purchased from Aldrich Chem. Co. (USA). Ebecryl 220 (hexafunctional aromatic urethane acrylate oligomer) was obtained from Cytec Surface Specialties Inc. (Belgium) as a cross-linking agent. Acrylo-polyhedral oligomeric silsesquioxane (POSS) was purchased from Hybrid Plastics™ (USA) as a heat-resistance improver. Darocur 4265 was obtained from CIBA Specialty Chemicals (Switzerland) as radical photoinitiator. All reagents were used without further purification.

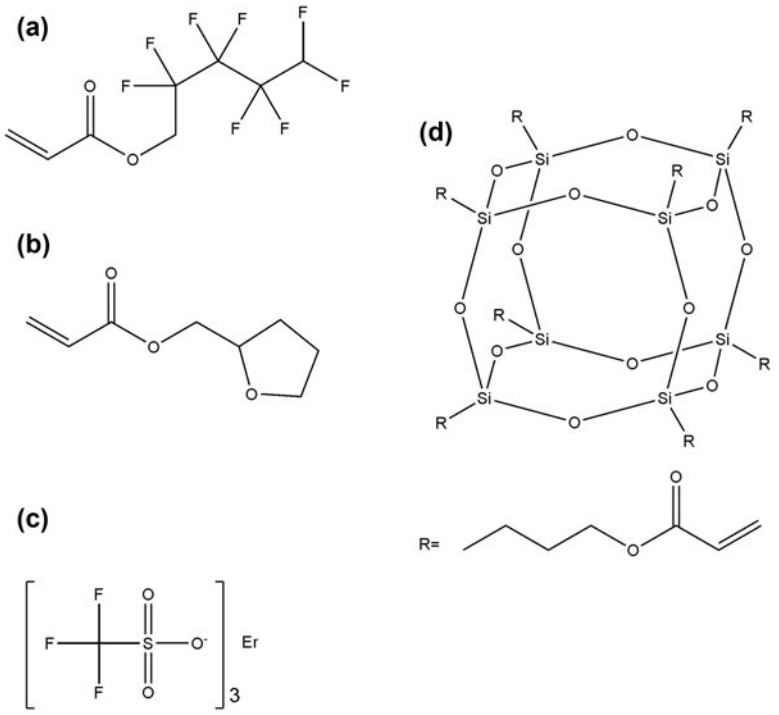

Figure 1. Chemical structures of (a) OFPA, (b) THFA (c) ErTFMS and (d) acrylo-POSS.

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UV-visible-NIR absorption and transmission spectra of the samples were obtained from a UV-Vis-NIR spectrometer (PerkinElmer Lambda 750, USA) at 1-nm intervals. Absorption and transmission data were evaluated using PerkinElmer's UV WinLab software. The thermal stability was measured using a Thermo Gravimetric Analysis (Perkin Elmer TGA-7, USA) apparatus in the region of 30 to 600 °C and operating at a scan speed of 10 °C/min in a N_2 atmosphere. Microstructures of the patterned films were observed with a scanning electron microscope (SEM; Hitachi S-4800, Japan) at an operation voltage of 3 keV. To evaluate the dispersion of Er^{3+} ions in the fluoropolymer films, an Electron Probe Micro Analyzer (EPMA; Shimadzu EPMA-1600, Japan) was used with a spot size of 1 μm and an acceleration voltage of 15 kV. The quantitative mapping of the elemental composition was performed over an area of approximately 70 μm^2 by monitoring the intensity of the characteristic X-rays.

Preparation of Precursor Solutions and Er^{3+}-doped Precursor Solutions

The precursor solutions were prepared with OFPA, THFA, Ebecryl 220 and acrylo-POSS in various compositions, as shown in Table 1. The ratio of OFPA and acrylo-POSS were changed from 75 to 45 wt% and from 0 to 30 wt%, respectively, to optimize the composition. The Er^{3+}-doped precursor solutions were also prepared by the addition of ErTFMS into the optimized precursor solution, in which the amount of ErTFMS was fixed with 5 wt% based on the overall polymer matrix precursors (i.e., OFPA, THFA, acrylo-POSS and oligomer). In summary, ErTFMS was added to THFA under strong mechanical stirring, and after dissolving ErTFMS in THFA, OFPA, acrylo-POSS, Ebecryl 220 and Darocur 4265 were added to the solution, which was stirred for 24 hours to obtain a homogeneous Er^{3+}-doped

Table 1. Compositions of precursor solutions

No.	OFPA (g)	THFA (g)	Acrylo-POSS (g)	Ebecryl 220 (g)	Darocur 4265[a] (g)
S0	0.75	0.2	0.0	0.05	0.01
S1	0.65	0.2	0.1	0.05	0.01
S2	0.55	0.2	0.2	0.05	0.01
S3	0.45	0.2	0.3	0.05	0.01

[a]1 wt% based on (OFPA + THFA + acrylo-POSS + Ebecryl 220) content.

precursor solution. The fluoropolymer and Er^{3+}-doped fluoropolymer films were prepared from the precursor and Er^{3+}-doped precursor solutions, respectively, by irradiation with UV light.

Soft-Imprint Lithography and MIMIC to Fabricate Er^{3+}-doped Fluoropolymer Patterns

Soft lithography is a convenient technique for generating patterned microstructures of organic polymers on the surfaces of solid substrates [18]. In this study, two different soft lithographic techniques, soft-imprint lithography and MIMIC, were applied to form micro-pattern of the Er^{3+}-doped fluoropolymer, as compared in Fig. 2. First, we fabricated the polydimethylsiloxane (PDMS) mold using commercial silicon elastomer (Sylgard 184™,

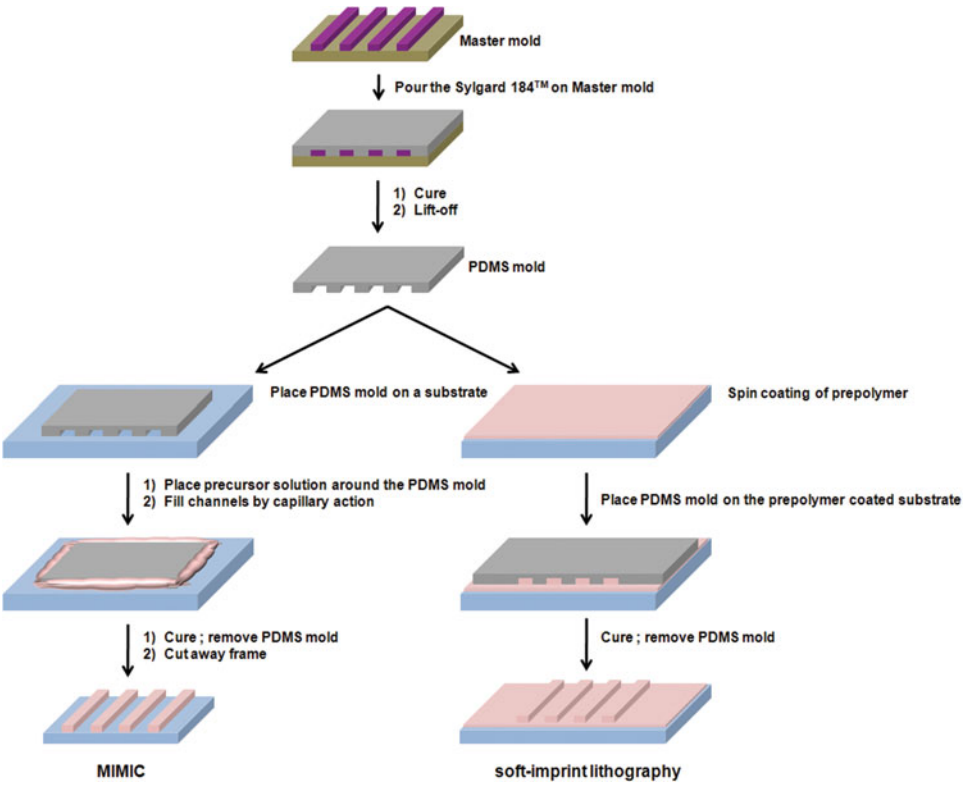

Figure 2. Soft lithography sequence using PDMS mold.

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Dow Corning, USA). PDMS pre-polymer, mixed at 1:10 ratio of the curing agent to elastomer base, was poured onto a master mold that was prepared by photolithographic method, and cured in an oven at 80 °C for 4 h. The PDMS mold was then peeled from the master mold and cut prior to use. For Er^{3+}-doped fluoropolymer pattern using soft-imprint lithography, a small drop of Er^{3+}-doped precursor solution was placed on a substrate. The PDMS mold was then brought into contact with the drop, and then pressure was applied during UV exposure (10 mW/cm^2 at 360 nm) for 200 s. The PDMS mold was then peeled from the substrate leaving the cured Er^{3+}-doped fluoropolymer pattern. For patterning via MIMIC, the PDMS mold was placed on a substrate to make conformal contact with that surface. As a result, a network of channels was formed between the PDMS mold and the substrate. The Er^{3+}-doped precursor solution was then placed at the open ends of the network of channels. The Er^{3+}-doped precursor solution spontaneously filled the channels by capillary action. The Er^{3+}-doped fluoropolymer pattern was then fabricated by UV exposure (10 mW/cm^2 at 360 nm) for 200 s, and the PDMS mold was removed. A network of Er^{3+}-doped fluoropolymer remained on the substrate surface.

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Results and Discussion

Optimized Composition of the Er^{3+}-doped Precursor Solutions

In the Er^{3+}-doped waveguide amplifier, the low transition loss of guided light in the Er^{3+} host materials is an important waveguide parameter. In polymers, optical absorption is caused by both molecular electronic excited states and by fundamental and overtone vibrations of molecular bonds. To achieve sufficient amplification, the signal loss from this absorption must be minimized [19]. We thus adopted the fluoropolymer system to minimize unwanted absorption by the polymer matrix at the emission wavelength (1,550 nm) of Er^{3+} ions. However, ErTFMS was not dissolved directly into the fluoromonomer (OFPA). As a solubility enhancer, we therefore adopted the acryl monomer THFA, instead of using organic solvents, because it shows isotropic phase at room temperature. When over 20 wt% of THFA was contained in the precursor solution, the homogenous Er^{3+}-doped precursor solution was obtained. Therefore, the ratio of THFA was fixed at 20 wt%. For pattern formation and high thermal stability, multifunctional acrylo-POSS were added as heat-resistance improver. The ratio of OFPA and acrylo-POSS were changed from 75 to 45 wt% and from 0 to 30 wt%, respectively, to determine the optimum composition of the precursor solution, as shown in Table 1 (S0∼S3). The optical transparency and thermal stability of the polymer matrix are important parameters for practical optical device applications. Optical devices are used inside and outside of buildings, so the thermal decomposition temperature of the host materials must be high enough to resist deformation [20]. To evaluate the effect of the crosslinking density on the optical transparency and thermal stability of the fluoropolymer films, we measured the transmittance and thermal decomposition temperature of the fluoropolymer films fabricated with the compositions shown in Table 1. Figure 3 compares the transmission spectra of the fluoropolymer films having a thickness of 10 μm, without ErTFMS, with various compositions (S0∼S3). The decrease of transmittance at 1,550 nm, which is the wavelength used in optical communication systems, was less than 2% in all the compositions (S1∼S3) compared to that of glass substrate, indicating that the optical propagation loss of light was slight in thin films applied to optical waveguiding devices. The loss in transmittance, at the wavelength of the pumping light source (488, 980, and 1,480 nm) used in the optical amplifiers, was also less than 3%. This result indicates that the loss of pump power by the host materials can be also minimized.

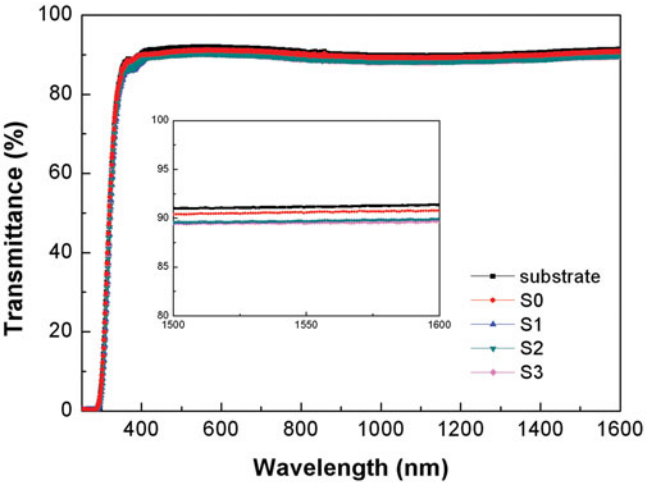

Figure 3. Transmission spectra of fluoropolymer films.

Figure 4 shows the TGA thermograms of fluoropolymer films with various compositions (S0∼S3). The fluoropolymer films had good thermal stabilities up to 350 °C (10 wt.% loss). With increasing the content of acrylo-POSS, the thermal decomposition temperature gradually increased from 329.5 to 361.5 °C due to higher crosslinking density and faster oxidation of POSS compared to fluorinated side-chain. The oxidation reaction of main chain from POSS ($SiO_{1.5}$) to silica (SiO_2) is faster than the decomposition of side-chain, because the fluorinated side group is more stable than alkyl chain. Therefore, the weight loss from side-chains was compensated by the weight gain from the oxidation of POSS [21]. In the view point of optical transparency and thermal stability, the fluoropolymer film prepared using the composition **S3** showed the excellent optical and thermal property, sufficient for application to optical devices. However, at high concentrations of acrylo-POSS,

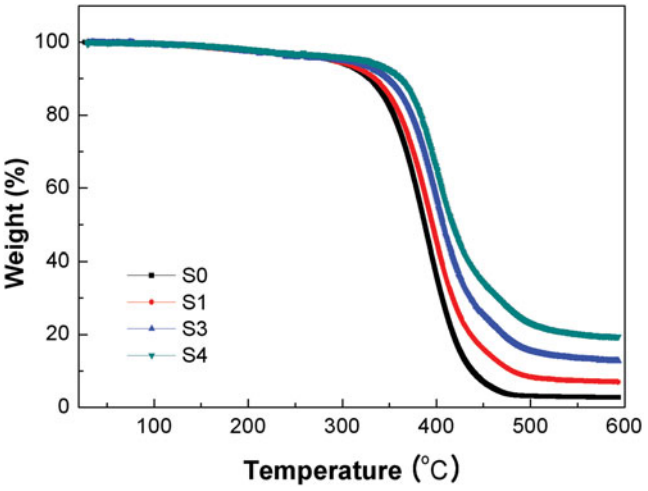

Figure 4. TGA thermogram of fluoropolymer films.

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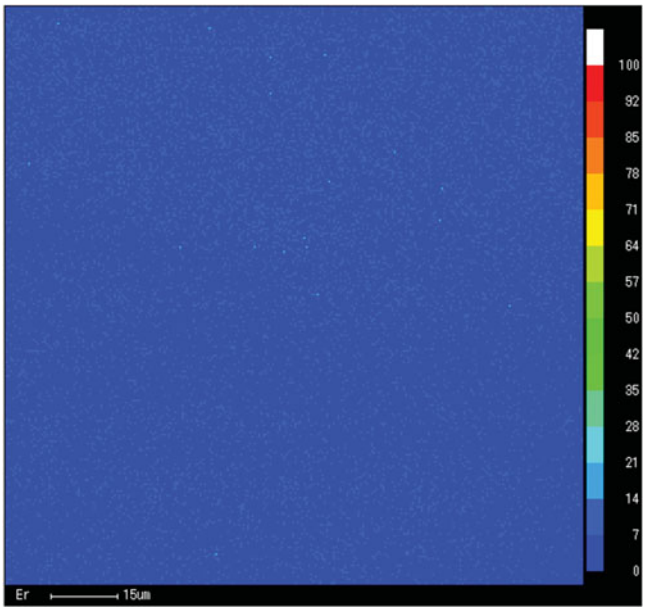

Figure 5. EPMA image by quantitative mapping of the Er^{3+}-doped fluoropolymer film.

more than 20 wt% based on the polymer matrix, the adhesion between fluoropolymer film and substrate was rapidly decreased due to their high crosslink density. The fluoropolymer film prepared from the S2 composition also had excellent optical and thermal properties, which made it sufficient for use in optical devices. From the above results, S2 was selected as the optimum composition for the Er^{3+}-doped precursor solutions.

In addition to the thermal stability and optical transparency of the Er^{3+} host materials, the uniform dispersion of Er^{3+} ions in the host film is also important to apply it to optical amplifiers. Rare earth ions tend to form clusters, and such aggregates quench the luminescence through the non-radiative decay channels from unwanted transitions by ion-ion interactions, etc [22]. The Er^{3+}-doped precursor solution was prepared by the addition of ErTFMS (0.053 g) to the previously optimized precursor solution (S2). To evaluate the dispersing property of Er^{3+} ions, an EPMA analysis was performed. Figure 5 shows the images obtained by quantitative mapping of the Er^{3+}-doped fluoropolymer film fabricated from the Er^{3+}-doped precursor solution. In the Er^{3+}-doped fluoropolymer film, good dispersion of the Er^{3+} ions was observed up to 5 wt%.

Fabrication of the Er^{3+}-doped Fluoropolymer Pattern by Soft-Imprint Lithography and MIMIC

It is important to fabricate the Er^{3+}-doped fluoropolymer patterns on a substrate for optical waveguide amplifiers. Soft lithography is currently used to form patterned microstructures of polymeric materials due to its simplicity of the method and the low cost of fabrication, and fidelity in transferring the patterns from the mold to the polymeric structures. In addition, the prevention of oxygen inhibition is the key for acrylate-based free-radical polymerization. Soft lithography is a good method for eliminating the oxygen inhibition by covering the PDMS mold.

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The Er^{3+}-doped polymer patterns were fabricated with the process condition described in experimental section, and their pattern sharpness was observed. However, the Er^{3+}-doped fluoropolymer patterns were partially separated from the substrate during the removal of the PDMS mold due to the low adhesion of the Er^{3+}-doped fluoropolymer patterns to the substrate. The fluoropolymers have many advantages, such as high thermal stability, chemical inertness, low surface tension and low dielectric constants, but their poor adhesion to other materials is a critical defect that affects some engineering aspects [23]. A surface treatment using ZAP 1020 (Chemoptics Inc., Korea), a trialkoxysilane with an acrylate functional group in 1-methoxy-2-propanol as an adhesion promoter, was introduced to enhance the adhesion between the substrate and the Er^{3+}-doped fluoropolymer pattern. Through heat treatment after spin coating, the trialkoxysilane forms self-assembled monolayers on the glass substrate surface. The adhesion of fluoropolymer to the substrate could be increased by coupling the acrylate functional group of the SAMs and other acrylate

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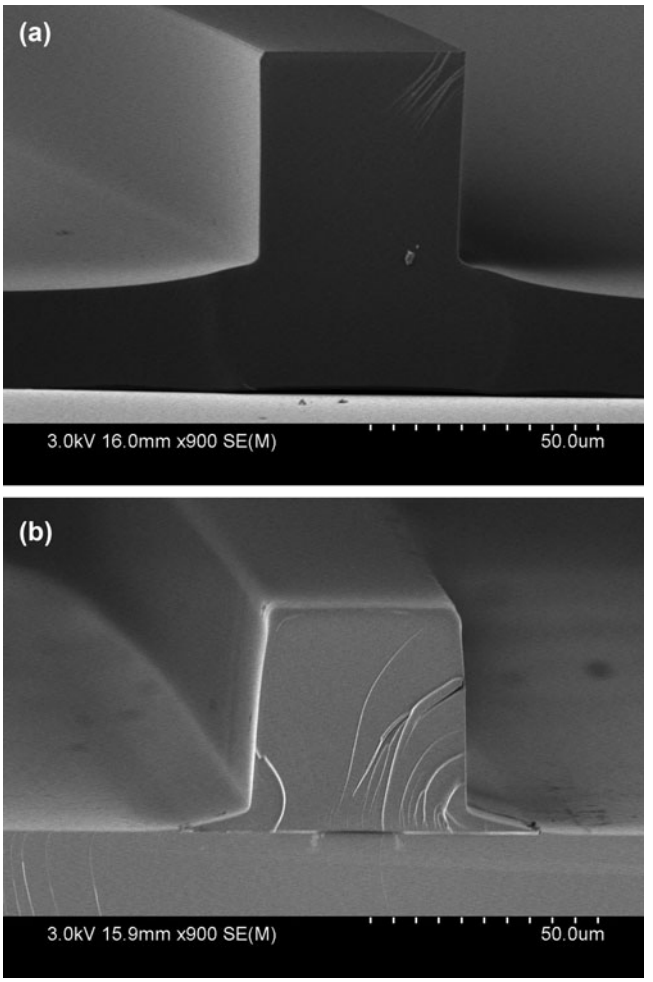

Figure 6. SEM images of a 50-μm-wide Er^{3+}-doped fluoropolymer pattern by (a) soft-imprint lithography and (b) MIMIC.

functional monomers of Er^{3+}-doped precursor solutions. The incorporation of an adhesion promoter eliminated the partial detachment of the fluoropolymer patterns by enhancing the adhesion between the substrate and the fluoropolymer patterns. Figure 6 shows an SEM image of the Er^{3+}-doped fluoropolymer pattern fabricated by soft-imprint lithography and MIMIC. As shown in Fig. 6(a), in the case of the polymer waveguide patterned with the soft-imprint lithography, a residual layer, i.e., a slab layer, is formed on the imprinted areas. Too thick layer of the slab causes a propagation loss due to power leakage to the slab guide, and is usually removed by an etching process [24]. On the contrary, the slab layer was not observed from the fluorpolymer patterns fabricated by MIMIC process as shown in Fig. 6(b). This implied that, by applying MIMIC process, the propagation loss could be minimized by removing the slab guide.

Conclusions

In summary, fluoropolymer films have been designed with high thermal stability, good film-forming properties and high optical transparency at 1,550 nm. By adjusting the precursor solution and the soft lithography conditions, we successfully fabricated Er^{3+}-doped fluoropolymer patterns without ErTFMS aggregation up to 5 wt%. This well-defined cross-linkable polymer has shown promise for optical device applications.

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