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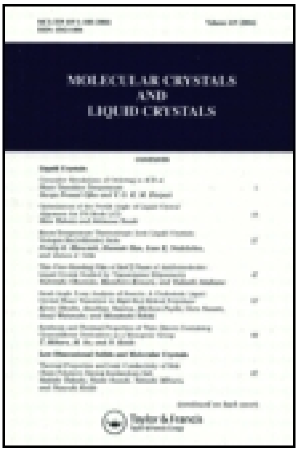

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Fabrication and Characterization of Erbium-Doped Fluoropolymer Patterns via UV-Nanoimprint Lithography for Use in Planar Optical Amplifiers

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Fabrication and Characterization of Erbium-Doped Fluoropolymer Patterns via UV-Nanoimprint Lithography for Use in Planar Optical Amplifiers

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The Er-doped precursor solutions were first prepared using 2,2,3,3,4,4,5,5-octafluoropentyl acrylate as a fluoromonomer, 2,2,3,3,4,4,5,5-octafluoro-1,6-hexanediol diacrylate as a cross-linking agent, poly(2,2,3,3,4,4,4-heptafluorobutyl acrylate) as a binding polymer, Darocure 4265 as radical photoinitiator and erbium(III) trifluoromethane sulfonate as an erbium source with various weight ratios. The crosslinked, patterned and erbium-doped fluoropolymer films were fabricated by UV-nanoimprint lithography on glass substrates for application as planar optical amplifiers. The fluoropolymer films acting as a host material for the Er ions showed excellent transmission properties—more than 80% over the visible and near-infrared regions— a high thermal decomposition temperature greater than 350°C and the ability to support a high Er concentration—up to 10 wt% based on the polymer matrix. These results were good enough for the films to be used in planar optical amplifier applications.

Keywords erbium; fluoropolymer; UV-nanoimprint lithography; planar optical amplifiers

Introduction

In the past few years, trivalent rare-earth (RE) ions have received much attention because of their potential applications in light-emitting diodes[1–3], lasers devices[4, 5] and optical amplifiers[6–10]. Among the RE ions, erbium (Er) is the most interesting dopant because of its emission at a wavelength around 1,550 nm, a standard wavelength used in telecommunication systems, resulting from its intra-4*f* transition.[11, 12] Currently, erbium-doped fiber amplifier (EDFA) technologies are commonly used in optical communication systems to compensate for signal losses, for example, waveguide, coupling or intrinsic intensity losses in optical splitters.[13–15] In addition, erbium-doped waveguide amplifiers (EDWAs) are now being developed. While EDWAs are used for access and home network applications, EDFAs are usually used for long-distance optical communication and networking systems because

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of their non-compatibility with integrated optical devices. The concentration of Er in EDWAs is approximately 10 to 20 times higher than that in EDFAs so that a high optical gain can be realized in a smaller and shorter device than is possible with EDFAs.[16] However, a high Er concentration may be detrimental because of ion–ion interaction phenomena such as up-conversion or cross-relaxation processes, which might reduce pump efficiency and gain performances.[17, 18] Consequently, dispersion of Er ions is important for decreasing the concentration quenching and enhancing pump efficiency.

In order to satisfy the above requirements, polymers have attracted significant attention as a host material for Er because of their many advantages, such as low cost, high packaging density, and simple processing steps for a wide range of substrates. In addition, polymers can easily control the various optical parameters—such as the refractive index, birefringence and thermal stability—through blending and copolymerization, which leads to superior device performance.[19, 20] However, when conventional polymers are used for waveguide fabrication, they have tended 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 replacement of 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.[21, 22]

In this study, we demonstrated the fabrication of cross-linked, patterned and Er-doped fluoropolymer films with UV-nanoimprint lithography (UV-NIL). The optical, thermal and dispersing properties of these films were also investigated. We focused on the fabrication of Er-doped fluoropolymer patterns for application as the core material in planar optical amplifiers with the goal of achieving excellent optical clarity, thermal stability, good dispersion of the Er ions and chemical resistance to be able to withstand typical fabrication processing and operation conditions.

Experimental

Materials

Figure 1 shows the structures of the chemicals used for the Er-doped fluoropolymer films. The fluoromonomer 2,2,3,3,4,4,5,5-octafluoropentyl acrylate (OFPA) and the cross-linking agent 2,2,3,3,4,4,5,5-octafluoro-1,6-hexanediol diacrylate (OFHDDA) were purchased from ABCR GmbH & Co. KG (Germany). The binding polymer poly(2,2,3,3,4,4,4-heptafluorobutyl acrylate) (PHFBA), the erbium source erbium(III) trifluoromethane sulfonate (ErTFMS) and the solvent tetrahydrofuran (THF) were purchased from Aldrich

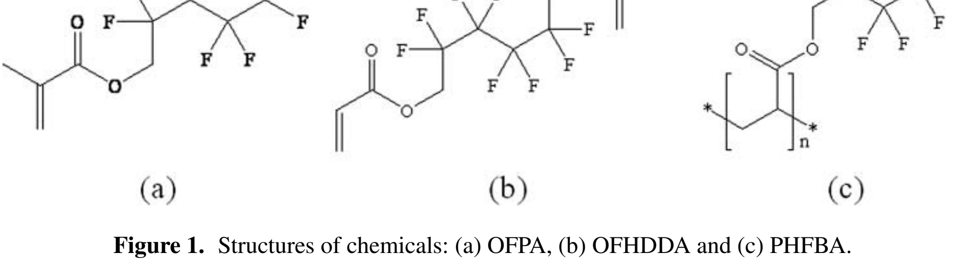

Figure 1. Structures of chemicals: (a) OFPA, (b) OFHDDA and (c) PHFBA.

Chem. Co. (USA). The radical photoinitiator Darocure 4265 was purchased from Ciba Specialty Chemicals Inc**.,** (Basel, Switzerland). All of the reagents were used without further purification.

Analytical Instruments

UV-visible-NIR absorption and transmission spectra of the samples were obtained from a UV-Vis-NIR spectrometer (PerkinElmer Lambda 750, USA). Absorption and transmission data were evaluated using PerkinElmer's UV WinLab software, and the data interval was 1.00 nm. The thermal stability was measured using a thermogravimetric analysis (Perkin Elmer TGA-7, USA) apparatus in the region from 30 to 500°C at a scan speed of 10°C/min in a N_2 atmosphere. In order to evaluate the dispersing property of Er ions in the samples, an electron probe microanalyzer (EPMA) was used. The EPMA (Shimadzu EPMA-1600, Japan) was operated at a spot size of 1 μm and an acceleration voltage of 15 kV. Quantitative mapping of the elemental composition was performed over an area of approximately 75 μm^2 by monitoring the intensity of characteristic X-rays. The microstructure of the Er-doped fluoropolymer pattern was observed using a field emission scanning electron microscope (FE-SEM, Hitachi S-4800, Japan)

Preparation of the Precursor Solutions and Er-Doped Precursor Solutions

The precursor solutions were prepared with OFPA, OFHDDA, PHFBA and Darocure 4265 in various compositions, as shown in Table 1 (a), and the weight ratio of OFPA:OFHDDA was changed from 4:1 to 1:4. The Er-doped precursor solutions were prepared by the addition of THF solutions of ErTFMS into the precursor solutions with stirring at room temperature, and the amount of ErTFMS was changed from 5 to 15 wt% based on the polymer matrix of the precursor solutions. In order to investigate the dispersing properties of the

Table 1. The composition of precursor solutions (a) and Er-doped precursor solutions (b).

Precursor solution	OFPA (g)	OFHDDA (g)	PHFBA (g)	Darocure 4265[a] (g)		
			(a)			
PS1	3.60	0.90	0.50	0.045		
PS2	3.00	1.50	0.50	0.045		
PS3	2.25	2.25	0.50	0.045		
PS4	1.50	3.00	0.50	0.045		
PS5	0.90	3.60	0.50	0.045		
Er-doped precursor solution	**OFPA (g)**	**OFHDDA (g)**	**PHFBA (g)**	**ErTFMS[b] (g)**	**THF (g)**	**Darocure 4265[a] (g)**
			(b)			
EPS1	3.00	1.50	0.50	0.25	1.00	0.045
EPS2	3.00	1.50	0.50	0.50	1.00	0.045
EPS3	3.00	1.50	0.50	0.75	1.00	0.045

[a] 1 wt% based on (OFPA + OFHDDA) content.

[b] 5 ∼ 15 wt% based on (OFPA + OFHDDA + PHFBA) content.

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Er ions in the samples, Er-doped precursor solutions were prepared using the composition of Table 1 (b). The fluoropolymer and Er-doped fluoropolymer films were prepared from their respective precursor solutions by irradiation with UV light.

UV-NIL for Fabrication of the Er-Doped Fluoropolymer Films

The general UV-NIL process for the patterning of Er-doped fluoropolymer was as follows. First, we fabricated the polydimethylsiloxane (PDMS) mold using commercial silicon elastomer (Sylgard 184^{TM}, Dow Corning, USA). Sylgard 184^{TM} was poured onto a master mold and then cured at 80°C for 4 hours by an organometallic crosslinking reaction. Second, the Er-doped precursor solution was deposited on a glass substrate, and then the PDMS mold was placed above the deposited layer followed by soft-baking at 70°C for 1 minute on a hot plate. Lastly, the Er-doped fluoropolymer pattern was fabricated by UV exposure (10 mW/cm^2 at 360 nm) for 200 seconds and then hard-baked at 120°C for 1 minute.

Results and Discussion

Optimized Composition of the Er-Doped Precursor Solutions

In planar optical amplifiers, low optical absorption of the guided light (1,550 nm) in the host materials is an important parameter. In polymers, optical absorption is usually caused by both molecular electronic excited states and by fundamental and overtone vibrations of molecular bonds. In order to achieve sufficient amplification, the signal loss due to this absorption has to be reduced.[23] Thus, we adopted the perfluoropolymer system to minimize unwanted absorption by the polymer matrix at the emission wavelength (1,550 nm) of Er. The next important parameter is the thermal stability of the polymer matrix for practical application to optical devices. Optical devices are used outside or inside buildings, so the thermal decomposition temperature of the host materials should be high enough to resist deformation.[24] To evaluate the optical transparency and thermal stability, we measured the transmittance and thermal decomposition temperature of the fluoropolymer films fabricated with the compositions of Table 1 (a). Figure 2 shows the transmission spectra of the fluoropolymer films having a thickness of 5 μm, without Er, prepared with various compositions (PS1 $\sim$ PS5). The transmittances in the visible and near infrared region decreased with increasing OFHDDA content. In particular, for the cases of PS4 and PS5, the transmittances were greatly decreased. In the films prepared from PS1 and PS2, the transmittance at the input light signal (1,550 nm) and pumping wavelengths (980 and 1480 nm) were over 85%. Table 2 shows the thermal decomposition temperature of the fluoropolymer films. The fluoropolymer films revealed good thermal stabilities up to about 390°C, where over 95% of their mass remained. While the transmittances decreased by increasing the OFHDDA content, the thermal decomposition temperature gradually increased from 356.39 to 391.23°C because of its high crosslinking density. We concluded that the thermal stability of the fluoropolymer films fabricated using the compositions, PS1 to PS5, was excellent and sufficient for optical device applications demanding high thermal resistance.

From the viewpoint of the transmittance and the thermal stability, the fluoropolymer films prepared from the PS2 composition showed excellent optical and thermal properties, enough to be used in optical devices. From the above results, PS2 was selected as the optimum composition for the Er-doped precursor solutions.

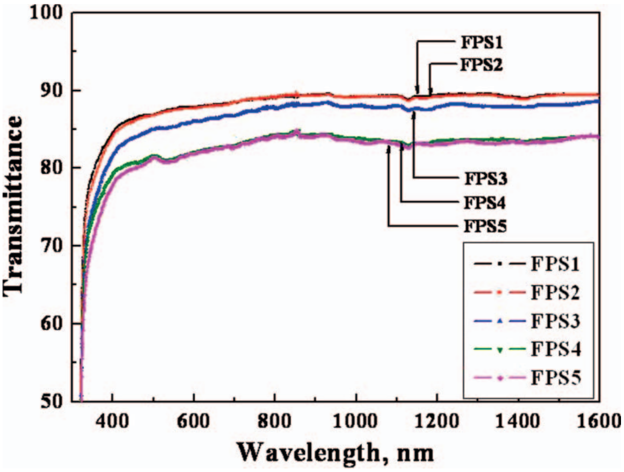

Figure 2. Transmission spectra of fluoropolymer films.

In addition to the thermal stability and the optical transparency, the dispersing property of Er ions in the host polymers is an important factor to be considered for application to planar optical amplifiers. Specifically, RE ions tend to form clusters between them, and such aggregates reduce the emission efficiency through non-radiative decay channels or they induce a luminescence from unwanted transitions by ion–ion interactions.[25] The Er-doped precursor solutions were prepared by addition of ErTFMS to the previously optimized precursor solution (PS2). To evaluate the dispersing property of Er ions, an EPMA analysis was performed. Figure 3 shows the images obtained by quantitative mapping of the Er-doped fluoropolymer films fabricated from the compositions given in Table 1 (b). In the case of the films prepared from the EPS1 and EPS2 solutions, good dispersion of the Er ions was observed, as shown in Figures 3 (a) and (b), respectively. However, as shown in Figure 3 (c), the polymer film from FEP3 showed an aggregation of the ErTFMS because of the excess amount of ErTFMS. Considering the concentration quenching of Er ions by aggregation, the optimum amount of ErTFMS was less than 10 wt% of the polymer matrix.

In the planar optical amplifier, Er is incorporated in the core of an optical waveguide. Using an external laser, such as a 980- or 1,480-nm diode laser, the Er is excited into one of its higher energy levels, and it then relaxes rapidly into the first excited state. An emission (1,550 nm) from the first excited state to the ground state is induced.[26] Figure 4 shows the

Table 2. Thermal decomposition temperature (T_d) of fluoropolymer films

Fluoropolymer films	T_d (°C)
FPS1[a]	356.39
FPS2[a]	363.50
FPS3[a]	366.34
FPS4[a]	379.61
FPS5[a]	391.23

[a] F means film, and PSx (x = 1∼5) means the composition of the film in Table 1 (a).

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Figure 3. EPMA images from a quantitative mapping of the Er-doped fluoropolymer films prepared from (a) EPS1, (b) EPS2 and (c) EPS3.

absorption spectra of the Er-doped precursor solution and the Er-doped fluoropolymer film as a function of wavelength from 300 to 1600 nm at room temperature. The important point to note for the Er-doped fluoropolymer film is that the peaks related to the absorption of Er seen around 490, 520, 650, 800, 980 and 1480 nm and arising from the $^4F_{7/2}$, $^2H_{11/12}$, $^4F_{9/2}$, $^4I_{9/2}$, $^4I_{11/2}$ and $^4I_{15/2}$ transitions, respectively, were observed, and absorption at 980 nm—an ideal pump wavelength—from the $^4I_{11/2}$ transition was clearly observed.

Fabrication of the Er-Doped Fluoropolymer Pattern by UV-NIL

For application to planar optical amplifiers, it is important to fabricate an Er-doped fluoropolymer film pattern on a substrate. UV-NIL, used in this study, is a promising technique and is regarded as a lithographic method good enough to be used on a large surface with relatively simple processing. In particular, the prevention of oxygen inhibition is a key issue with acrylate-based free-radical polymerization, and UV-NIL could eliminate the oxygen

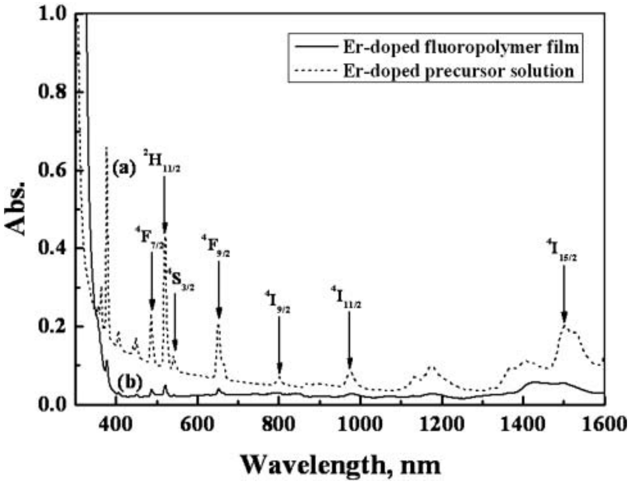

Figure 4. UV-Vis-NIR spectra of (a) the Er-doped fluoropolymer precursor solution and (b) the Er-doped fluoropolymer film.

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inhibition by covering the PDMS mold. We investigated the lithographic properties of Er-doped precursor solutions by UV-NIL, and the resulting process conditions were as follows. First, the PDMS mold was fabricated by the conventional method using commercial silicon elastomer on a master mold.[27] Second, the solvent (THF) was evaporated from the Er-doped precursor solution, EPS2, in a vacuum oven to obtain a viscosity suitable for spin-coating. Third, the Er-doped precursor solution was deposited onto a glass substrate by spin-coating to allow pattern replication, and then a PDMS mold was placed above the coated layer. Lastly, the Er-doped fluoropolymer pattern was fabricated through the UV curing process.

However, during the removal of the PDMS mold, the Er-doped fluoropolymer patterns were partially separated from the substrate along with the PDMS mold because of the low adhesion of the Er-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, whereas their poor adhesion to other materials is a critical defect for some engineering aspects.[28] A surface modification using ZAP 1020 (Chemoptics Inc., Korea), composed of trialkoxysilane with an acrylate functional group and 1-methoxy-2-propanol as an adhesion promoter, was introduced to enhance the adhesion between the substrate and the Er-doped fluoropolymer pattern. Through heat treatment after spin coating, the trialkoxysilane forms self-assembled monolayers (SAMs) on the surface of the glass substrate.[29] The adhesion of fluoropolymer to the substrate could be increased by polymerization between the acrylic C-C double bonds of SAMs and other acrylic C-C double bonds of Er-doped precursor solutions (Figure 5). By incorporating an adhesion promoter, we were able to eliminate the partial detachment of the fluoropolymer patterns due to an enhancement in adhesion between the substrate and Er-doped precursor solution. In order to evaluate surface wettability, we measured contact angles before and after modification with ZAP 1020. The contact angle between the precursor solution and the substrate was decreased from 29.79 to 7.11° by treatment of ZAP 1020, as shown in Figure 6.

Meanwhile, other defects such as film shrinkage and pinholes were observed after the imprinting. It was found that the unwanted residual solvent in the polymer film induced the film shrinkage and the pinholes after the imprinting, which can cause serious problems in pattern uniformity and sharpness.[30] To solve this problem, the solvent in the Er-doped precursor solution was completely eliminated by a soft-bake before irradiation with UV light. This soft-bake was typically carried out at 70°C for 1 minute.

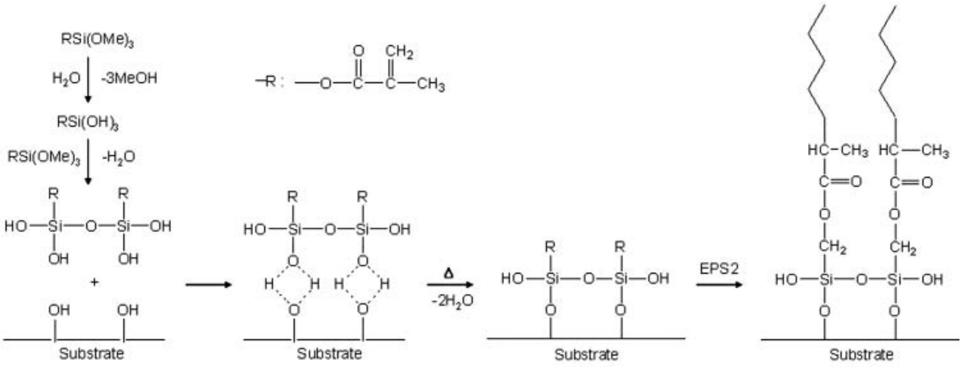

Figure 5. Reaction mechanism of the adhesion promoter.

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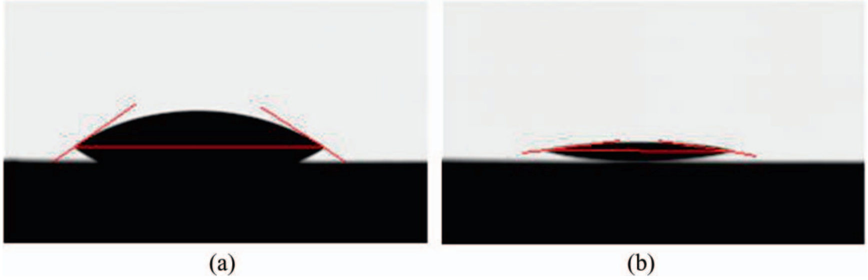

Figure 6. The contact angles of the precursor solution on the substrate before (a) and after (b) surface modification.

Finally, by the introduction of an adhesion promoter and soft-baking, we were able to form uniform and pinhole-free Er-doped fluoropolymer patterns by UV-NIL without any detachment.

Figure 7 shows the schematic diagram of the optimized conditions of UV-NIL, and the Er-doped fluoropolymer pattern fabricated from the composition of EPS2 is displayed in Figure 8. Pattern sharpness and uniformity sufficient for optical device applications were obtained.

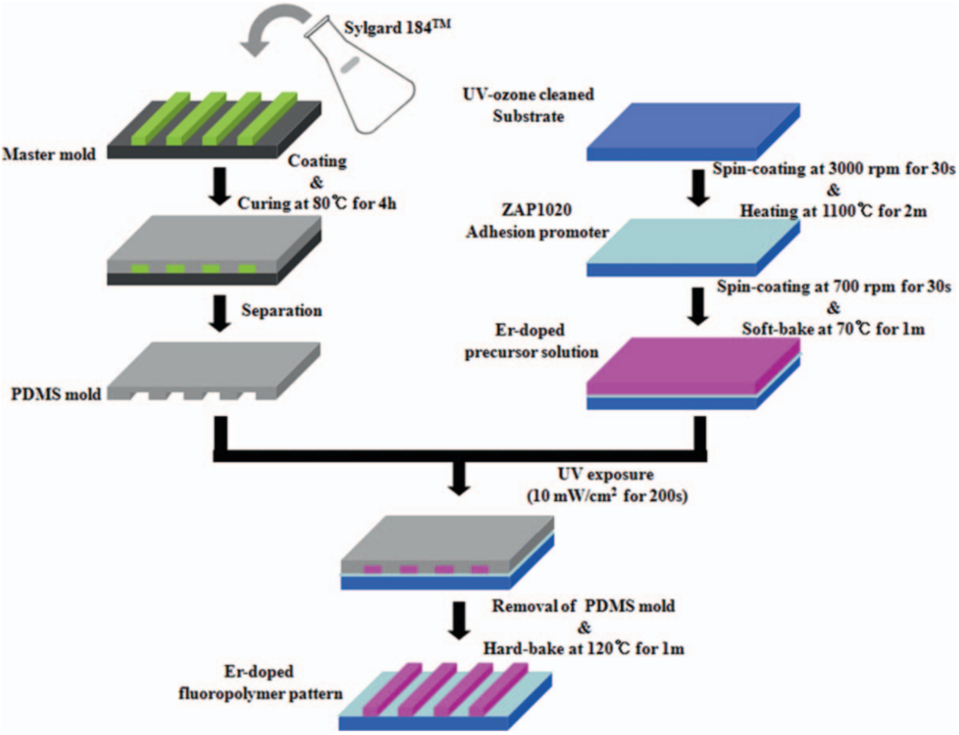

Figure 7. Schematic diagram and process conditions for the UV-NIL optimized in this study.

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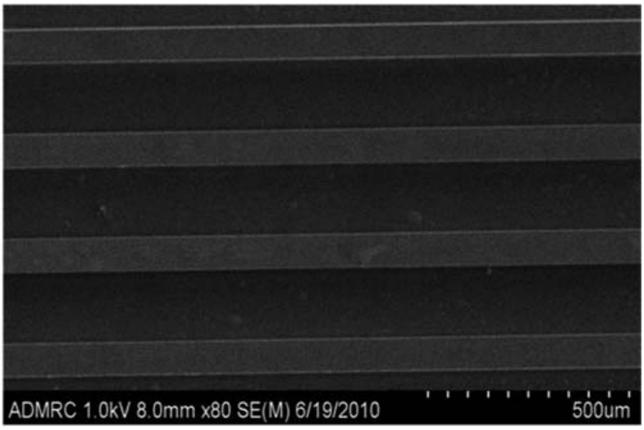

Figure 8. SEM image of an 80-μm-wide Er-doped fluoropolymer pattern fabricated by UV-NIL.

Conclusions

In summary, fluoropolymer films have been designed to have a high Er content without aggregation, good film-forming properties, high optical transparency at 1,550 nm and high thermal stability. By adjusting the Er-doped precursor solution and the UV-NIL conditions, we successfully fabricated an Er-doped fluoropolymer pattern without aggregation of the Er ions for concentrations up to 10 wt%. A well-defined, cross-linkable polymer has shown promise for optical device applications.

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