



# Low-loss modified SU-8 waveguides by direct laser writing at 405 nm

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**Abstract:** In this work we present a fabrication process to obtain a low-loss waveguide in the photo-curable resin SU-8 using direct laser writing at 405 nm wavelength. Polymer-based devices offer low-cost prototype fabrication, fabrication flexibility, reliability, low power consumption and potential for mass production. These characteristics, coupled with its high optical performance and low propagation losses, make it an attractive material for applications related to optical biosensing. Initially, a method to reduce SU-8 viscosity is described to allow film thicknesses of a few hundred nanometers, thus guaranteeing single mode propagation at visible range. This is achieved while also introducing an H-nu 470 photoinitiator, providing the displacement of the absorption peak of the material from 365 nm to 470 nm, thus allowing H-line polymerization and the direct laser writing at wavelengths 405 nm and above. Key material and structure characteristics such as absorbance, transmittance, roughness and chemical composition on the surface are analyzed for both pure and modified SU-8. We observe lower RMS surface roughness in the latter one. In spite of the chemical modification of the material, optical parameters like absorption and refractive index in the wavelength of interest are not affected. Single- and multimode optical waveguides are demonstrated. The sidewall roughness is measured at 5.6 nm, and the propagation loss for the single mode waveguide is 4.4 dB/cm at 633 nm wavelength, providing a high quality and low-cost fabrication platform for optical nano-devices.

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**OCIS codes:** (160.5470) Polymers; (130.5460) Polymer waveguides; (220.4241) Nanostructure fabrication.

## References and links

1. A. Melikyan, L. Alloati, A. Muslija, D. Hillerkuss, P. C. Schindler, J. Li, R. Palmer, D. Korn, S. Muehlbrandt, D. Van Thourhout, B. Chen, R. Dinu, M. Sommer, C. Koos, M. Kohl, W. Freude and J. Leuthold, "High-speed plasmonic phase modulators," *Nature Photon.* **8**, 229–233 (2014).
2. T. Toury, S. Brasselet and J. Zyss, "Electro-optical microscopy: mapping nonlinear polymer films with micrometric resolution," *Opt. Lett.* **31**(10), 1468–1470 (2006).
3. C. Chang, V. H. Tran, J. Wang, Y. K. Fuh and L. Lin, "Direct-Write Piezoelectric Polymeric Nanogenerator with High Energy Conversion Efficiency," *Nano Lett.* **10**(2), 726–731 (2010).
4. J. J. Ju, J. Kim, J. Y. Do, M. S. Kim, S. K. Park, S. Park and M. H. Lee, "Second-harmonic generation in periodically poled nonlinear polymer waveguides," *Opt. Lett.* **29**(1), 89–91 (2004).
5. J. W. Parks and H. Schmidt, "Flexible optofluidic waveguide platform with multi-dimensional reconfigurability," *Sci. Rep.* **6**, 33008 (2016).
6. J. Noh, S. Jeong and J. Y. Lee, "Ultrafast formation of air-processable and high-quality polymer films on an aqueous substrate," *Nature Commun.* **7**, 2041 (2016).
7. M. C. Estevez, M. Alvarez and L. M. Lechuga, "Integrated optical devices for lab-on-a-chip biosensing applications," *Laser Photon. Rev.* **6**(4), 463–487 (2012).
8. X. Chen, F. Yang, C. Zhang, J. Zhou and L. J. Guo, "Large-Area High Aspect Ratio Plasmonic Interference Lithography Utilizing a Single High-k Mode," *ACS Nano* **10**(4), 4039–4045 (2016).
9. C. H. Chan, T. Y. Wu, M. H. Yen, C. E. Lin, K. T. Cheng and C. C. Chen, "Low power consumption and high-contrast light scattering based on polymer-dispersed liquid crystals doped with silver-coated polystyrene microspheres," *Opt. Express* **24**(26), 29963–29971 (2016).
10. M. Hiltunen, J. Hiltunen, P. Stenberg, S. Aikio, L. Kurki and P. Karioja, "Polymeric slot waveguide interferometer for sensor applications," *Sensors* **22**, 7229–7237 (2014).

11. M. Fleger and A. Neyer, "PDMS microfluidic chip with integrated waveguides for optical detection," *Microelectron. Eng.* **83**, 1291–1293 (2006).
12. D. Duval, A. B. Gonzalez-Guerrero, S. Dante, J. Osmond, R. Monge, L. J. Fernandez and L. M. Lechuga, "Nanophotonic lab-on-a-chip platforms including novel bimodal interferometers, microfluidics and grating couplers," *Lab on Chip* **12**, 1987–1994 (2012).
13. H. Lorenz, M. Despont, N. Fahrni, N. LaBianca, P. Renaud and P. Vettiger, "SU-8: a low-cost negative resist for MEMS," *J. of Microm. and Microeng.* **7**(3), 121 (1997).
14. J. Zhang, K. L. Tan and H. Q. Gong, "Characterization of the polymerization of SU-8 photoresist and its applications in micro-electro-mechanical systems (MEMS)," *Polymer Testing* **20**(6), 693–701 (2001).
15. B. Li, X. Wang, H. Y. Jung, Y. L. Kim, J. T. Robinson, M. Zalalutdinov, S. Hong, J. Hao, P. M. Ajayan, K. T. Wan and Y. J. Jung, "Printing Highly Controlled Suspended Carbon Nanotube Network on Micro-patterned Superhydrophobic Flexible Surface," *Sci. Rep.* **5**, 15908 (2015).
16. K. Uchiyama, K. Okubo, M. Yokokawa, E. T. Carlen, K. Asakawa and H. Suzuki, "Micron scale directional coupler as a transducer for biochemical sensing," *Opt. Express* **23**(13), 1094–4087 (2015).
17. Y. S. No, R. Gao, M. N. Mankin, R. W. Day, H. G. Park and C. M. Lieber, "Encoding Active Device Elements at Nanowire Tips," *Nano Lett.* **16**(7), 4713–4719 (2016).
18. Y. Su, C. Liu, S. Brittan, J. Tang, A. Fu, N. Kornienko, Q. Kong and P. Yang, "Single-nanowire photoelectrochemistry," *Nature Nanotech.* **11**(7), 609–612 (2016).
19. D. R. Ponce, K. Lozano, T. Eubanks, A. Harb, D. Ferrer and Y. Lin, "Thermophysical Analysis of SU8-Modified Microstructures Created by Visible Light Lithography," *Journal of Polymer Science Part B: Polymer Physics* **45**, 1390–1398 (2007).
20. B. Y. B. Yang, L. Y. L. Yang, R. H. R. Hu, Z. S. Z. Sheng, D. D. D. Dai, Q. L. Q. Liu and S. H. S. He, "Fabrication and Characterization of Small Optical Ridge Waveguides Based on SU-8 Polymer," *J. Lightw. Tech.* **27**, 4091–4096 (2009).
21. K. K. Tung, W. H. Wong and E. Y. B. Pun, "Polymeric optical waveguides using direct ultraviolet photolithography process," *J. Appl. Phys. A* **80**, 621–626 (2005).
22. J. Kim and J. H. Shin, "Stable, Free-space Optical Trapping and Manipulation of Sub-micron Particles in an Integrated Microfluidic Chip," *Sci. Rep.* **6**, 33842 (2016).
23. D. Y. Kang, C. Kim, G. Park and J. H. Moon, "Liquid immersion thermal crosslinking of 3D polymer nanopatterns for direct carbonisation with high structural integrity," *Sci. Rep.* **5**, 18185 (2015).
24. Q. Wang, D. Zhang, H. Xu, X. Yang, A. Q. Shen and Y. Yang, "Microfluidic one-step fabrication of radiopaque alginate microgels with in situ synthesized barium sulfate nanoparticles," *Lab on a Chip* **12**, 4781 (2012).
25. M. B. de la Calle, Y. Madrid and C. Camara, "Speciation of antimony by atomic absorption spectrometry. Applicability to selective determination of Sb(III) and Sb(V) in liquid samples and of bioavailable antimony in sediments and soil samples," *Microchimica Acta* **109**, 149–155 (1992).
26. M. Thiel, J. Fischer, G. Von Freymann and M. Wegener, "Direct laser writing of three-dimensional submicron structures using a continuous-wave laser at 532 nm," *App. Phys. Lett.* **97**, 221102 (2010).
27. X. Ma and J. Wei, "Nanoscale lithography with visible light: optical nonlinear saturable absorption effect induced nanobump pattern structures," *Nanoscale* **3**, 1489–1492 (2011).
28. C. H. Lee, K. Jiang and G. J. Davies, "Sidewall roughness characterization and comparison between silicon and SU-8 microcomponents," *Mater. Character.* **58**(7), 603–609 (2007).
29. R. Murali, D. K. Brown, K. P. Martin and J. D. Meindl, "Process optimization and proximity effect correction for gray scale e-beam lithography," *J. Vac. Sci. Technol. B* **24**(6), 2936–2939 (2006).
30. J. Kim, D. C. Joy and S. Y. Lee, "Controlling Resist Thickness and Etch Depth for Fabrication of 3D Structures in Electron-Beam Grayscale Lithography," *Microelectron. Eng.* **84**, 2859–2864 (2007).
31. L. Mosher, C. M. Waits, B. Morgan and R. Ghodssi, "Double-Exposure Grayscale Photolithography," *J. Microelectromechan. Syst.* **18**(2), 308–315 (2009).
32. L. H. Gabrielli, D. Liu, S. G. Johnson, and M. Lipson, "On-chip transformation optics for multimode waveguide bends," *Nature Commun.* **3**(3), 1217 (2012).
33. J. C. Ramirez, L. M. Lechuga, L. H. Gabrielli and H. E. Hernandez-Figueroa, "Study of a low-cost trimodal polymer waveguide for interferometric optical biosensors," *Opt. Express* **23**(9), 1094–04087 (2015).
34. K. E. Zinoviev, A. B. González-Guerrero, C. Domínguez and L. M. Lechuga, "Integrated Bimodal Waveguide Interferometric Biosensor for Label-Free Analysis," *J. Lightw. Tech.* **29**(13), 1926–1930 (2011).
35. P. K. Dey and P. Ganguly, "A technical report on fabrication of SU-8 optical waveguides," *J. Opt.* **43**(1), 79–83 (2014).

## 1. Introduction

Polymer-based integrated photonics offers a number of advantages over silicon or III-V materials in a number of applications. The electrical, thermal and mechanical properties of polymers can be tailored to great extents to meet particular requirements in a case-by-case basis, as the past decades of material science evolution has demonstrated. For integrated optics, polymers have

been extensively employed, for instance, to improve electro-optical [1, 2] and optical nonlinear effects [3, 4]. In general, their notable disadvantage is a lower tolerance to the high temperatures that are often employed in conventional micro- and nanofabrication processes. On the other hand, if high temperatures can be avoided, polymers usually represent a lower-cost fabrication platform, which is always desirable, but specifically so for disposable devices, as is the case for optical biosensors, as an example.

By replacing the standard silicon or III-V technology with polymers, additional advantages can be conferred to biosensors, such as fabrication flexibility, reliability, low power consumption and potential for mass production accompanied by a reduction of production costs [5–9].

Polymeric photonic biosensors are at the core of the concept of photonic lab-on-a-chip [10], aimed towards solving existing limitations in achieving economically viable devices for point-of-care diagnosis [11, 12].

There are a number of choices for optically transparent polymers for fabrication of photonic structures. One of the most common and well-developed of those polymers is SU-8, an epoxy usually employed due to its mechanical stability, high aspect ratio, low cost, and well established fabrication process. Micro-electro-mechanical devices have been developed in SU-8 for decades [13–15], and more recently, optical devices followed [16–18]. I-line (365 nm) is the recommended exposure wavelength, although great results have also been demonstrated with electron beam and X-rays.

Recently, studies have shown that by mixing the photoinitiator H-nu 470 to the conventional SU-8 epoxy, it is possible to improve the chemical properties and thermal stability of fabricated structures with this photoresist, increasing its degradation temperature up to 400 °C [19]. This addition aims to change the maximum absorption peak of the photoresist from 365 nm to 470 nm wavelength, allowing a material conventionally used in near UV photolithography to be polymerized with a laser in the visible spectrum. The advantage of allowing exposure at longer wavelengths is that less expensive hardware can be used to allow the full benefits from technologies such as the photonic lab-on-a-chip.

The photoinitiator presence does not change the optical properties after the bake process, thus representing an excellent alternative for the fabrication of integrated devices for applications in the visible, near infrared and infrared domains, i.e., not only in biosensor applications, but also in telecommunications [5, 20–23].

In this paper we present the fabrication and characterization of low-loss optical waveguides using the modified SU-8 with H-line lithography (405 nm wavelength). We show that the use of the photoinitiator allows direct laser writing (DLW) of sub-micron structures with low sidewall roughness, low fabrication tolerance, and transparency comparable to those of the original SU-8. Characterization of thin film was performed by X-ray photoelectron spectroscopy (XPS) and atomic force microscopy (AFM), and single- and multimode waveguides propagation losses were measured at 633 nm wavelength.

## 2. Viscosity and surface roughness characterization

Optical waveguides in the visible range require sub-micron dimensions for single mode operation, thus we first reduced the viscosity of the SU-8 to obtain a 600 nm layer upon spin coating a SiO<sub>2</sub> substrate [20, 24]. We diluted SU-8 2100 (75% solid) in cyclopentanone to obtain solutions with 14% and 20% solid concentrations. The mixtures were stirred for 24 hours to ensure uniform coverage of the substrate later on. Spin calibration curves for both dilutions are presented in Fig. 1(a), which shows that we are able to reach the desired thickness range for optical waveguide fabrication, in our case around 600 nm.

The 14% solid SU-8 received the photoinitiator H-nu 470, the cationic cure accelerator AN-910E, and the OPPI photoacid generator to form concentrations of 0.1%, 0.1%, and 2.5% in weight respectively, and was then stirred for 48 hours. The exact amounts used of each component

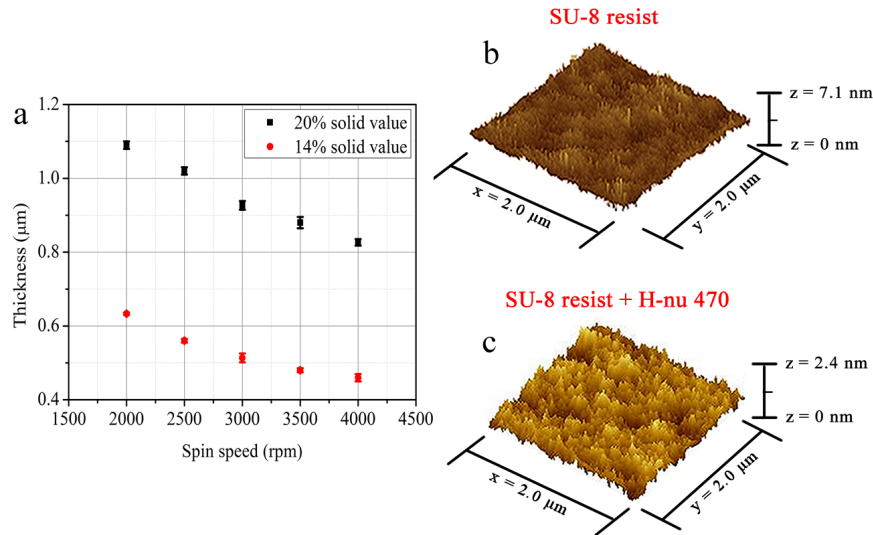


Fig. 1. Bulk modified SU-8 characterization. (a) Spin coating calibration curves for 2 different solid concentrations after dilution of the epoxy in cyclopentanone. (b) Surface roughness of the original SU-8 layer (14% solid) obtained by AFM. (c) Surface roughness of the SU-8 layer with the addition of the photoinitiator.

in the mixture are detailed in Table 1. After spin coating, the original (diluted) and modified SU-8 layers showed great uniformity under visual inspection. Surface roughness characterization by AFM showed RMS values of  $0.44 \text{ nm} \pm 0.05 \text{ nm}$  for the original resist and  $0.20 \text{ nm} \pm 0.05 \text{ nm}$  for the modified version. The scans were performed over a  $4 \mu\text{m}^2$  area, as presented in Fig. 1(b) and 1(c). The chemical contents of each sample were measured by XPS and are displayed in Fig. 2(a) and 2(b). The spectroscopy results indicate only a small concentration of antimony (2.45%) in the modified mixture that is not present in the original SU-8, which is known to increase the mechanical hardness and improve the thermal properties of the material [25].

Table 1. Components of the modified SU-8 mixture for H-line lithography.

SU-8 (75% solid)	9.33 mL
Cyclopentanone	40.66 mL
H-nu 470	54 mg
AN-910E	54 mg
OPPI	137 mg

The other 3 prominent energy peaks are similar in both curves, with a slight increase in oxygen and fluorine counts (accompanied by a decrease in carbon counts) in the modified mixture when compared to the original one.

### 3. H-line laser lithography

The SU-8 structures were exposed using DLW, which allows rapid prototyping and development cycle of nanometer-scale structures [26, 27] without compromising the possibilities of mass-production at lower costs and scalability to larger substrates.

The preparation of the samples, illustrated in Fig. 3, followed the usual SU-8 processing: after cleaning in piranha bath, they were dehydrated on a  $200^\circ\text{C}$  hot plate for 20 minutes, spin coated

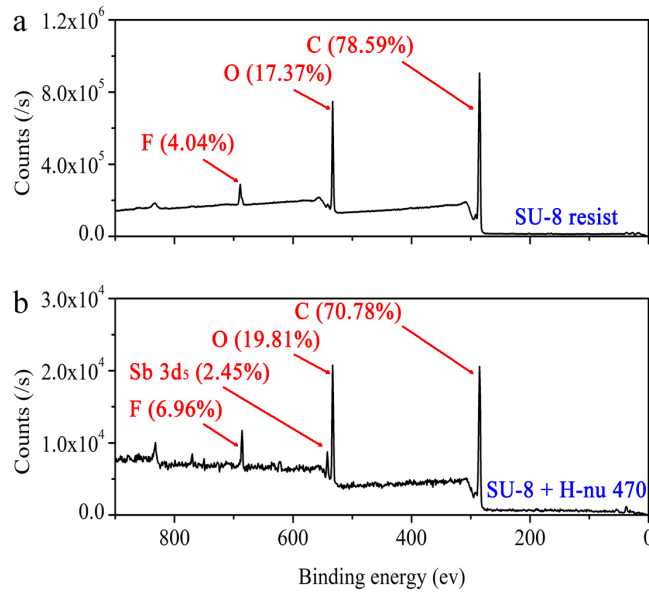


Fig. 2. SU-8 surface composition. (a) Original SU-8 layer obtained by XPS. (b) SU-8 layer with the addition of the photoinitiator.

with the modified photoresist, soft-baked at 65 °C for 1 minute, and then ramped up to 95 °C for a 3-minute pre-bake. We used a Heidelberg DWL 66FS system with a 2.0 mm head lens and 720 mJ/cm<sup>2</sup> maximal dose on substrate at 405 nm for exposure. After exposition process the photoresist was subjected to a post-exposure bake starting at 65 °C for 1 minute, ramped up to 95 °C and held for another minute. The fabricated structures were then developed in SU-8 Developer by submersion and stirring for 60 s and, finally, hard-baked for 5 minutes on a 150 °C hot plate.

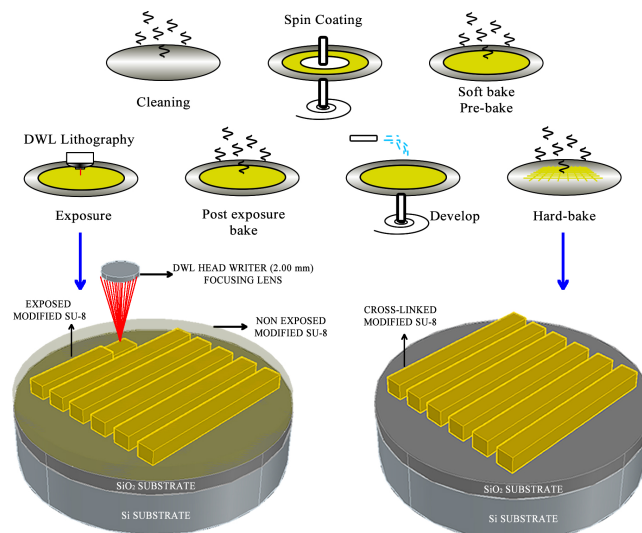


Fig. 3. Scheme of the fabrication process. Fabrication steps for the polymerization of the modified SU-8 with 405 nm DLW.

A dose test was performed with 3 different filters (5%, 15%, and 30%) and linewidths (600 nm, 10  $\mu\text{m}$ , and 100  $\mu\text{m}$ ) to characterize the polymerization of the modified SU-8. The results presented in Figs. 4(a)–4(c) show that, as expected, the required dose for polymerization of the full thickness of the photoresist layer is quite high. Nevertheless, with both the 5% and 15% transmission filter, complete exposure is attainable for all linewidths with laser energy above 70%. By inspecting Figs. 4(a)–4(c), we can also see that the exposure is not impacted by the linewidth of the structure, specially with the 30% filter, which indicates that the process is robust enough to allow the fabrication of structures with dimensions of different orders of magnitude in a single step. This benefit can be seen in the scanning electron microscope images of fully exposed structures in Fig. 5. These structures were obtained with the 30% filter and the geometrical tolerance measured for the 600 nm linewidth was 8%.

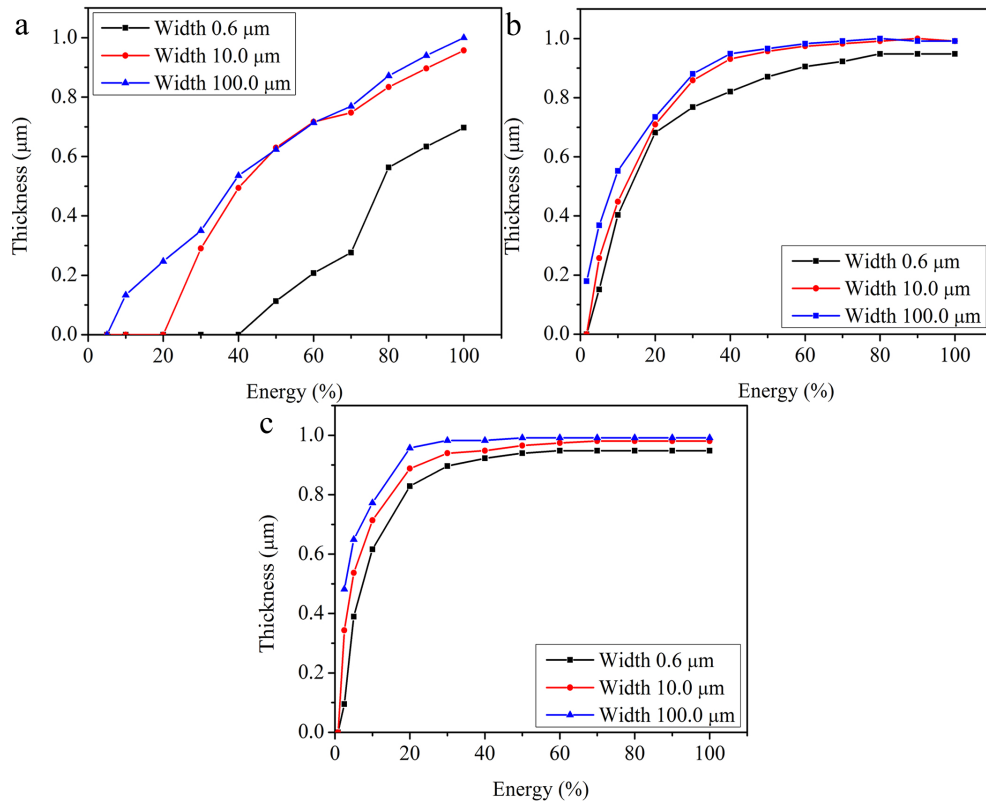


Fig. 4. Characterization of polymerization dose. (a) DLW dose calibration for 600 nm, 10  $\mu\text{m}$  and 100  $\mu\text{m}$  linewidth structures with a 5% transmission filter. (b) Same dose calibration as (a) but with a 15% transmission filter, i.e., overall 10% higher dose. (c) Same as (a) but with a 30% transmission filter (25% higher dose).

The sidewalls of the structures were also characterized using AFM scanning, which resulted in an RMS roughness of  $5.60 \text{ nm} \pm 0.02 \text{ nm}$ . Despite the addition of the photoinitiator, the sidewall quality of the device is not affected, which represents an improvement compared to reported results [28], because the implementation of fabrication with direct writing reduces the roughness on the device sidewalls when compared to conventional photolithography.

One last interesting feature of this process is the possibility of controlling the thickness on the final structure by modulating the laser energy almost linearly, as Fig. 4(a) show for the 5% filter. The use of varying core thickness for optical confinement has been show to provide an

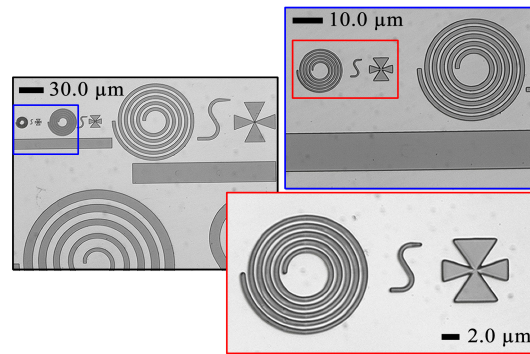


Fig. 5. Scanning electron microscope images of the fabricated structures at different scales..

excellent platform for the fabrication of gray scale photonic devices [29–32], i.e., devices that present multiple levels of core thickness—or even a continuum of thickness variation across the device—fabricated in a single exposure step. This technique is usually employed in the fabrication of large diffraction lenses, but with the length scales achieved in our experiments, it could be used in the future to enable multimode polymeric devices for optical interconnects and biosensors [33,34].

#### 4. Optical characterization

We characterized the absorbance and transmittance of the SU-8 samples with and without the addition of the photoinitiator over a wide range of wavelengths.

The results are displayed in Fig. 6(a) and 6(b). The addition of the photoinitiator results in a small increase in absorbance (loss in transmission), still providing a more than adequate platform for optical applications beyond 400 nm. Below that wavelength, the modified photoresist presents an accentuated absorbance due to the influence of H-nu 470, indicating that the material still absorbs at lower wavelengths even after hard-baking. The refractive indices measured at 633 nm were 1.59 for both samples, which allows the fabrication of well-confined waveguides on SiO<sub>2</sub>.

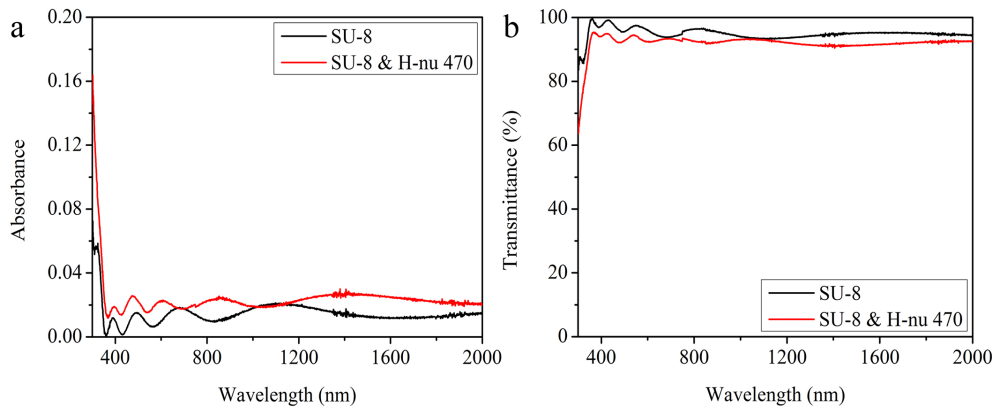


Fig. 6. Optical characterization of the SU-8 with and without addition of the photoinitiator. (a) Absorbance and (b) transmittance curves show that the addition does not significantly impact the optical properties of the material for wavelengths beyond 400 nm.

Waveguides were then fabricated over a 2.0 μm thick SiO<sub>2</sub> layer using DLW and left air-clad. Single-mode square waveguides with 600 nm sides were patterned with lengths of 5 mm, 10 mm,

and 20 mm for transmission loss measurements Fig. 7(a) shows an scanning electron microscope image of a few devices and Fig. 7(b) presents their optical characterization setup. We used two objective with numerical apertures 0.65 and 0.40 for coupling in and out of the waveguides, respectively. The source was a 633 nm wavelength laser with electric field polarized parallel to the substrate (preferential direction for the quasi-TE mode in conventional rectangular waveguides). The collected data was linearly fitted and resulted in propagation losses of only  $4.38 \pm 0.55$  dB/cm, as shown in Fig. 7(c), an excellent value for applications where fabrications costs are more critical than ultra-low loss devices.

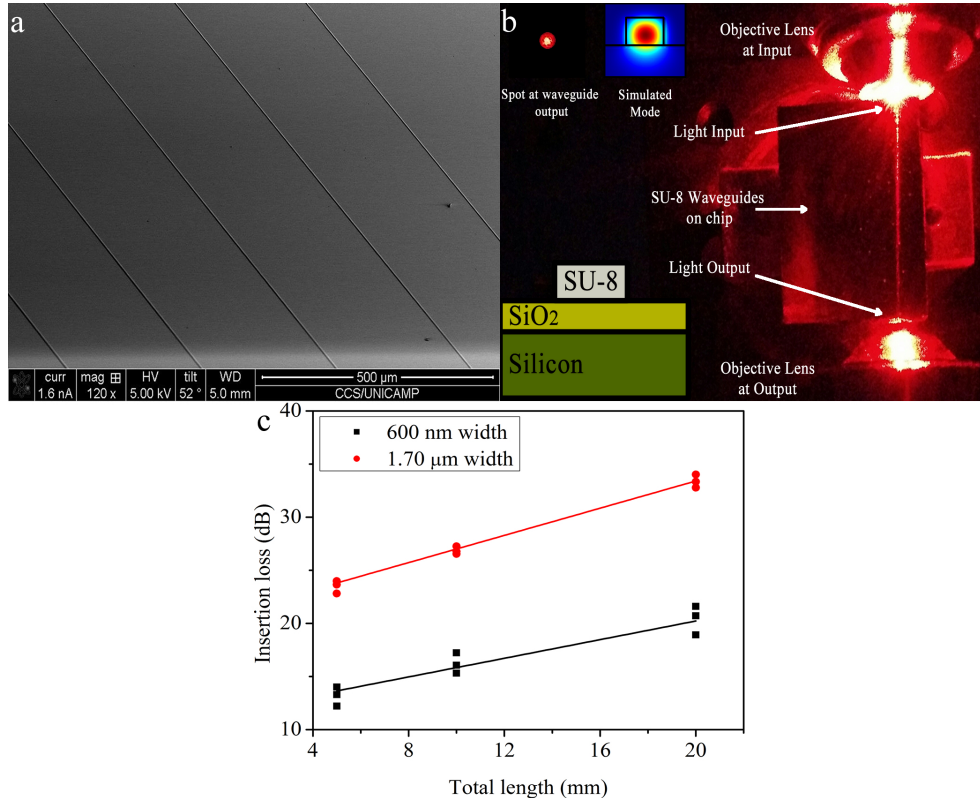


Fig. 7. Characterization of optical waveguides fabricated on SU-8 with the H-nu 470 photoinitiator. (a) Scanning electron microscope images of a few air-clad waveguides on a SiO<sub>2</sub> layer. (b) Insertion loss measurement setup with objectives for coupling light in and out of the waveguides at 633 nm wavelength. (c) Propagation loss calculation based on a linear fit of the measured loss for waveguides of different lengths.

Similarly, multimode waveguides with core width of 1.70 μm were also fabricated on the same 600 nm thick SU-8 layer and with the same length variations. Their optical measurements are also presented in Fig. 7(c), where the linear fit to the data resulted in a propagation loss of  $6.40 \pm 0.3$  dB/cm. Single- and multi-mode waveguide insertion losses were measured, achieving lower values than previously reported [35], demonstrating an excellent performance by fabricated devices with this novel technique.

## 5. Conclusions

This work describes the preparation and characterization of SU-8 as an optical material suitable for H-line photolithography. We show that the addition of the H-nu 470 photoinitiator that allows

absorption at 405 nm does not significantly impact other characteristics of the photoresist. The fabrication process is the same as for the original SU-8 processing and the resulting layer has excellent surface roughness and similar optical absorbance at longer wavelengths.

Patterning via DLW allowed us to reproducibly obtain sub-micron structures including single-mode waveguides for the 633 nm visible wavelength. Propagation losses on those were measured at 4.38 dB/cm, which enables the use of this material in optical applications where low-cost is critical, such as disposable lab-on-a-chip for point-of-care diagnosis.

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