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Soft Lithographic Fabrication of High *Q* Polymer Microcavity Arrays

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ABSTRACT

As new synthetic, low-loss polymers are developed, polymer optical cavities are experiencing a revolution, in both fabrication design and functionality. Recently, a fabrication technique was developed that enabled planar arrays of polymeric resonators to achieve cavity *Q* factors greater than 1 million. In the present letter, this molding technique is expanded to fabricate resonators from polymers that have either thermal or UV curing mechanisms. The quality factors and broad band spectrum of these devices are determined at 680, 1300, and 1550 nm. These resonant cavities demonstrate quality factors which are competitive with photonic crystals and microdisk resonators.

With the development of several low-loss polymers, resonant cavities fabricated from polymers are moving out of the lab and into more mainstream applications such as biosensors.[1] These devices can have functionalities which more traditional silica and silicon devices cannot achieve such as mechanical flexibility, cleanroom-free fabrication, and low-cost materials. Additionally, gain media can be directly integrated into the polymer before curing.[2] There are currently two distinctly different methods of fabricating planar arrays of polymeric microcavities: molding from a master structure[3,4] or direct lithographic patterning of the polymer.[5]

To achieve resonant cavities with quality factors greater than 1 million, three fundamental criteria must be realized: ultrasmooth surfaces, low material loss polymer, and low-loss coupling.[6] When using a soft lithographic molding technique, the easiest method to achieve a smooth surface is by starting with a master structure with a smooth surface. Currently, both the microsphere and the microtoroid resonator are known to have ultrasmooth surfaces (as demonstrated by their cavity *Q* factors > 100 million), therefore, they are ideal master structures.[7,8] To achieve the second two criteria, we utilized a molding polymer with low optical loss and tapered optical fiber waveguides, which have low coupling loss.

To fabricate material-limited resonant microcavities, a master array of UHQ microtoroids was fabricated in a cleanroom.[7] The major toroidal diameter ranged from 30 to 150 μm, with minor diameters ranging from 2.5 to 6 μm. These dimensions were replicated in the polymer microtoroid resonators. The fabrication of the polymer microcavities from the master array occurs outside the cleanroom, and the molding process is outlined in Figure 1. The molding material chosen was polydimethylsiloxane or PDMS (RTV 184, Dow Corning 10:1). This material is typically used in molding microfluidic devices because of its ability to accurately replicate the master structure.[9] It has also been shown to accurately mold microlenses and microspheres, therefore, its capability to accurately reproduce very smooth surfaces is well documented.[10] To aid in the release of the PDMS mold from the complex three-dimensional silica microtoroid master, the master was first exposed to trimethylchlorosilane (TMCS) vapor.

In the present molding process, there are several features of the microtoroid that must be replicated to maintain the quality factor: the toroidal ring, the overhang, and the pillar. If the overhang is not replicated, the microtoroid will not be optically isolated from the substrate and the quality factor will suffer. Because this silicone molding process is able to reproduce all of these features, high-*Q* resonators can be fabricated. However, an additional important characteristic of PDMS is its transparency in the UV. This transparency allows it to be used as a mold for UV curable polymers.

To verify the flexibility of the molding process, resonant cavities were fabricated using either a thermally curing polymer (Crystal Cast 9024, Industrial Polymers) or an ultraviolet curing polymer (Efiron WR-509, Luvantix). Once the silicone mold was fabricated and filled with the polymer of choice, a glass cover slip was placed on top of the mold. Downward pressure applied to the cover slip displaces air bubbles and allows the polymer to fill the mold completely. Because of its increased viscosity, Crystal Cast requires an intermediate de-airing step at ~150 mTorr for 20 min before the glass cover slip is placed over the mold to further reduce bubbles. The cover slip also acts as a substrate to facilitate

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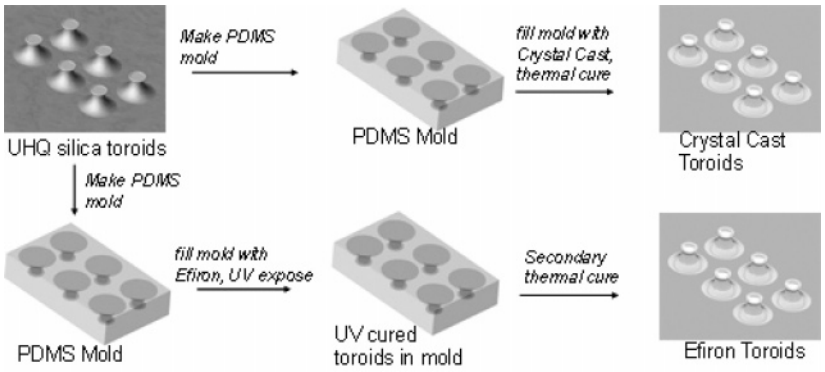

Figure 1. Outline of the soft lithographic process used to fabricate Crystal Cast or Efiron microtoroid resonators. In the fabrication of either resonant cavity, a PDMS negative mold of the silica ultrahigh-*Q* microtoroid resonator array was made. To form Crystal Cast microtoroids using soft lithography, this PDMS mold was then filled with Crystal Cast, de-aired, and thermally cured. Finally, the Crystal Cast microtoroids were released. To form Efiron microcavities, the PDMS mold was filled with Efiron, exposed to a UV source, released from the mold, and thermally post-cured.

removal of the replica from the mold after curing. It is important to note that the PDMS inverse molds can be used repeatedly. While an exhaustive study of the lifetime of the molds has not been performed, PDMS molds have been re-used as many as 50 times without degrading their replication ability, as judged by the resonator *Q* factor. Two factors that appear to play a role in reducing the lifetime are the UV lamp and the oxygen plasma. If the mold needs to be exposed to the UV lamp to cure the polymer resonator or to an oxygen plasma to release the resonator, the mold becomes brittle.

As stated previously, the two polymers cure by different mechanisms. Crystal Cast only requires a thermal cure at 80 °C for 1 h in a gravity oven (Figure 2a). In contrast, to completely cure Efiron, both ultraviolet and thermal curing steps are necessary. After exposing Efiron to an ultraviolet lamp with an intensity of 20 mW/cm^2 for 22 min in the silicone mold, the replicas were released from the mold and were post-cured at 80 °C for an additional 30 min in a gravity oven (Figure 2b). The ability to mold and cure Efiron is only possible because of the transparency of the silicone molds in the ultraviolet wavelength range. This confirms that the silicone molds can be used to fabricate polymer resonators that cure by two distinctly different mechanisms. Additionally, no surface treatment was necessary to release either polymer from the mold.

Characterization of the microresonators was performed at 680, 1300, and 1550 nm by measuring the resonator line width, free-spectral range, and analysis of the modal structure. For testing purposes, a single-frequency, tunable external-cavity laser was coupled to a single-mode optical fiber containing a short, tapered section. The tapered optical fiber waveguide coupled power into the "whispering gallery modes" of the Crystal Cast and Efiron microtoroids. Tapered fibers are made by heating a standard, optical fiber with an oxyhydric torch while stretching the fiber.[11] They function as high-efficiency probes of microresonators.[12] During testing, the polymer microtoroids were placed on a high-resolution translation stage (100 nm step resolution) and were monitored by two cameras (top and side view) simultaneously. With the tapered waveguide in close proximity to the polymer microtoroid, optical laser power was launched,

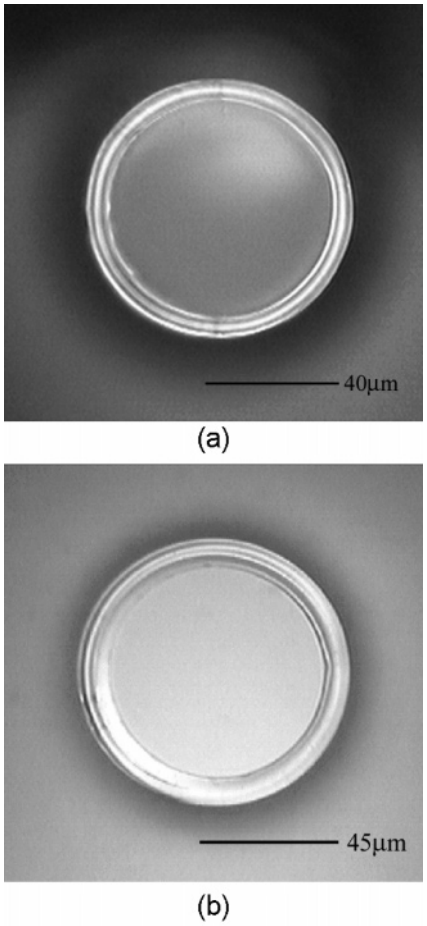

Figure 2. Optical micrographs of the (a) Crystal Cast and (b) Efiron toroidal microresonators.

and the transmission spectra were monitored. The modal structure was dominated by principal transmission minima believed to be the fundamental transverse mode of the polymer microtoroids.

The intrinsic *Q* factor of the resonator modes was determined by scanning the laser (line width of 300 kHz) and measuring both the transmission and the line width (full width at half-maximum) for several waveguide-resonator

Table 1. Microcavity Characterization

polymer	quality factor at 680 nm × 10^5	quality factor at 1300 nm × 10^5	quality factor at 1550 nm × 10^5	absorption at 680 nm (dB/cm)	absorption at 1300 nm (dB/cm)	absorption at 1550 nm (dB/cm)
Crystal Cast	1.61−2.27	2.23−4.18	1.02−1.43	2.63−3.71	0.64−1.22	1.63−2.28
Efiron	1.98−3.18	1.51−2.41	1.20−1.57	1.87−3.03	1.21−1.91	1.53−2.04

coupling conditions in the undercoupled regime.[11] To minimize the effect of thermal distortion on the mode structure, the optical input power was kept below 1 μW using an optical attenuator, and the laser scan frequency was optimized so as to ensure that neither scan direction (increasing frequency vs decreasing frequency) nor scan frequency had any observable impact on line width. Figure 3a shows a Crystal Cast microtoroid spectra at 1300 nm, and Figure 3b shows an Efiron spectra at 680 nm and corresponding Lorentzian fits. These two line widths represent the highest Q values (narrowest line widths achieved for each polymer). Table 1 shows the range of Q values achieved for Efiron and Crystal Cast at the three wavelengths tested.

In addition to determining the quality factor, the material absorption can also be calculated because the Q's were material-limited. This ability is a unique feature of these toroidal resonant cavities. Typically, there are multiple dominant loss mechanisms and separating them to isolate a single one (like material loss) would be unreliable. This is most apparent in the case of lithographically patterned polymethylmethacrylate (PMMA) resonators.[13] The theoretical material-limited Q is greater than 10^7 in the visible; however, the highest experimentally measured Q is 10^5. This difference is primarily due to surface roughness induced losses from the lithographic blemishes on the periphery of the ring resonator. However, in the present experiments, because the surface scattering loss and coupling loss are both negligible, the material loss is the dominant loss mechanism, and, therefore, the material loss or material absorption can be determined.[4]

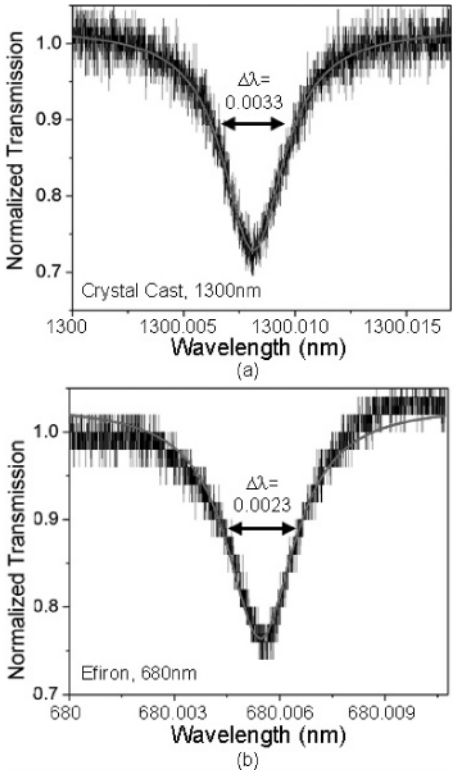

Figure 3. Spectra and Lorentzian fit (gray line) of (a) Crystal Cast microtoroid at 1300 nm with a loaded Q of 3.95 × 10^5 and (b) Efiron microtoroid at 680 nm with a loaded Q of 2.96 × 10^5. The loaded quality factors were determined from the indicated linewidths (the Lorentzian fits are shown).

Using the microtoroid to determine the material loss was first demonstrated with the PDMS resonators.[4] In this case, it was possible to directly compare the absorption measured using the microcavity[17] to the absorption spectra from the manufacturer, and there was excellent agreement across a range of wavelengths. The measured material absorption values for Crystal Cast and Efiron at all three wavelengths are also listed in Table 1. An alternative method of determining material loss is using a waveguide prism coupler. In this case, a thin film of the polymer is spun onto a substrate, and two prisms couple light into the thin film, forming a polymer waveguide approximately 1 cm long. By using the resonator instead of the waveguide prism coupler, the effective testing length increases from 1 cm to 1 m, for a cavity with a Q of 1 million; however, the footprint of the device is the size of the resonant cavity or ~100 μm in diameter.

Polymer resonators are finding applications in biological sensing[1] and in telecommunications.[13] In both fields, the quality factor of the microcavity is important as it determines both sensitivity[14] and lasing threshold or bandwidth.[15] To fabricate higher Q resonators, it will be crucial to find lower-loss polymers; however, in conjunction with low-loss polymers, the fabrication design is equally crucial, as has been seen in the fabrication evolution from the ultrahigh-Q microsphere to the ultrahigh-Q microtoroid. By using the ultrahigh-Q microtoroid resonator as the master structure, the smooth reflowed surface is transferred to the polymer device. Additional functionality, such as a gain media, can be integrated without affecting this process. Because of the inherent mechanical flexibility of many polymers, tuning can be achieved by simply stretching the cavity.[16] With the large variety of polymers available and new ones being developed, devices with quality factors above 100 million will soon be realizable on a loss-cost, disposable platform.

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(17) In prior work, it has been shown that the smoothness of the replicated resonator surface is sufficient to enable material-loss-dominated Q factors when the intrinsic Q (Q_0) is in excess of 1 million. Therefore, in order to solve for the material absorption, the intrinsic Q must be determined. The intrinsic modal line width (and Q_0) was computed using a simple coupling model to analyze the loaded transmission spectra. The material absorption was then calculated using the relation $\alpha = 2\pi n_{\text{eff}}/\lambda Q$ where α is the material absorption, n_{eff} is the effective refractive index of the material determined from the free-spectral-range, and λ is the wavelength of the resonance. The effective refractive index (n_{eff}) of the microresonator was determined from the free-spectral range. The principal transmission minima were identified by performing a broad-band transmission spectrum measurement; the exact location of each minimum was verified by scanning the laser (line width 300 kHz) and monitoring the resonance behavior. Using the relation $n_{\text{eff}} = \lambda^2/\pi d\lambda_{\text{FSR}}$ where λ is the center wavelength of the free spectra range, d is the diameter of the resonator, and λ_{FSR} is the free-spectral-range, the effective refractive index was calculated at a series of discreet, resonant wavelengths. The effective refractive index was observed to increase slightly as the wavelength decreases. For Crystal Cast and Efiron, the effective refractive index in the visible is very similar, $n_{\text{eff}} = 1.48$; however, in the near-IR, the effective refractive indices of the two polymers changed to 1.33 and 1.38 respectively.

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