# Chapter 4

# Yb-doped glass microcavity laser operation in water

#### 4.1 Introduction

The previous chapter analyzed the performance of a Yb:SiO<sub>2</sub> microtoroid laser tested in normal air. The knowledge learned through those experiments, and the key 1  $\mu$ m emission wavelength made it possible to build a microtoroid laser that operates efficiently in water. Whispering-gallery mode (WGM) microcavities feature an evanescent field component that extends into the surrounding medium [78]. Besides enabling a convenient way to couple optical power, the evanescent field provides a natural means by which resonant modes can interact with their environment. This latter feature is the basis of sensing techniques using passive whispering gallery microcavities [79, 80]. For example, heavy water  $(D_2O)$ , has been detected through measurement of quality factor (Q), and interleukin-2 molecules have been detected by measuring a resonance shift [81, 18]. Theoretical studies have shown that an active microcavity can offer higher sensitivity [82]. As an example of one such device, Lu et al. recently demonstrated a biosensor utilizing the evanescent field component of a dye-doped distributed-feedback laser [83]. In this chapter, a  $Yb:SiO_2$  microcavity laser that operates in water is presented. The work builds upon the ytterbium-doped silica microtoroid laser with record-low threshold operation in air [19]. To the best of the author's knowledge, laser action of a microcavity has not yet been demonstrated in water, and provides an important new feature for active biosensing applications.

# 4.2 Fabrication of microtoroid for laser in water

The microcavity used in this work is a silica toroid suspended on a silicon pillar and substrate [84]. These devices feature exceptionally high passive Q factors, making them well suited for laser applications when properly doped. In previous work, the sol-gel method has been used to dope the microcavity with erbium or ytterbium [19, 20]. Ytterbium is a more suitable rare-earth dopant than erbium for laser operation in water because the absorption coefficient of water is  $0.16 \text{ cm}^{-1}$  at 1.04

 $\mu m$  (Yb<sup>3+</sup> emission) compared to 10 cm<sup>-1</sup> at 1.55  $\mu m$  (Er<sup>3+</sup> emission) [22].

The ytterbium-doped silica gain medium is fabricated on a silicon chip according to the solgel chemical synthesis method detailed in Chapter 3. To briefly summarize, tetraethoxysilane, water, isopropyl alcohol, hydrochloric acid, and ytterbium nitrate are mixed in specific quantities at 70°C for three hours to produce the gel. The ytterbium concentration for the laser is  $1.1 \times 10^{19}$  cm<sup>-3</sup>. Next, the ytterbium-doped solgel is deposited in three layers onto a silicon substrate by spin coating. After each layer is deposited, the thin film is annealed at  $1,000^{\circ}$ C in normal atmosphere for three hours. The refractive index of the  $Yb:SiO_2$  film is 1.46, according to a spectroscopic ellipsometer measurement (Sentech SE850). Standard photolithography and a buffered oxide wet etch are used to create an array of glass disks with 180  $\mu$ m diameter. These disks are optically isolated from the silicon substrate by a  $XeF_2$  dry etch, leaving  $Yb^{3+}$ :SiO2 disks supported by silicon pillars. Finally, a  $CO_2$  laser symmetrically reflows each silica microdisk into a smooth microtoroid with 120  $\mu$ m principal diameter. This diameter is optimized for low resonator loss and pump threshold. The reduced index contrast of the toroid resonator in water (0.13) compared to air (0.46) increases both the minimum diameter required to avoid significant radiation loss, and also the fraction of power in the evanescent field [85]. A full vectorial finite-element simulation of the fundamental mode shows that the fraction of intensity located outside of the microtoroid increases from 0.6 to 3.6% when the toroid is immersed in water.

### 4.3 Laser testing in water

The Yb:SiO<sub>2</sub> microtoroid laser was characterized first in air and subsequently in water. A single sub-micron diameter fiber taper (Fibercore SMF980-5.8-12.5), phase matched to the microtoroid's whispering-gallery spatial mode, simultaneously injects pump light and retrieves lasing output as shown in Figure 4.1 [13, 8].



Figure 4.1. Image of testing setup showing evanescent coupling of fiber taper to 120  $\mu$ m Yb<sup>3+</sup>:SiO<sub>2</sub> microcavity (side view) in air.

FEM modeling demonstrates that the toroid mode index increases slightly in water compared to air, because the modes evanescent component increases and experiences the higher refractive index of water. It is possible to achieve a full range of coupling conditions in water from under- to over-coupled. The coupling gap increases in water for a given coupling condition, and is caused by spatial mode broadening of the fiber and toroid in water. The taper coupling to the microtoroid is controlled by a three-axis piezo nano-positioning system. A tunable, single-frequency, narrow linewidth (;300 kHz) semiconductor diode laser provides pump excitation in the 970 nm absorption band of ytterbium. At the fiber output, Yb<sup>3+</sup>:SiO<sub>2</sub> laser emission at 1040 nm is separated from unabsorbed pump light by a fiber-based WDM filter (45 dB isolation).

For testing in water, a glass cover slip is placed several millimeters above the silicon chip containing the toroid, and the air gap is filled with pure water to produce a temporary aquarium. A diagram of the water testing setup is shown in Figure 4.2. The fiber taper passes through a large water bubble, delivering pump light and extracting laser emission from the microtoroid. The viscosity of water decreases fluctuations in the taper to toroid gap, and creates a more stable coupling condition than in air. The fiber taper transmission is slightly less in water, due to both absorption in the water, and scattering losses incurred when the taper wave passes through the water/air interface. A larger aquarium would reduce the taper losses by placing the water/air interface at locations where the taper mode is more confined in glass fiber.

The quality factor of the microcavity pump resonance was measured to ascertain both the doping level as well as the impact of water absorption on the total microcavity Q. In air, the Q was measured to be  $2.8 \times 10^5$  ( $\lambda = 970.6$  nm). This Q factor is dominated by absorption of the Yb<sup>3+</sup> ions. Q factors



Figure 4.2. Diagram of the setup for testing in water (side view). The taper passes into the temporary aquarium, formed by injecting water in between a top glass slide on the silicon chip containing microtoroids. The toroid is completely surrounded by water.

for silica sol-gel microtoroids have been recorded as high as  $2.5 \times 10^7$  million. To compare, thermal silica microtoroids routinely have Q factors greater than  $1 \times 10^8$  [10]. Assuming the measured Q factor  $(2.8 \times 10^5)$  is dominated by pump absorption, the doping level is estimated to be  $1.1 \times 10^{19}$  cm<sup>-3</sup>, in good agreement with the value expected from fabrication  $(1.2 \times 10^{19} \text{ cm}^{-3})$ .

In water, the Q in the pump band is measured to be  $1.3 \times 10^5$  ( $\lambda = 970.8$  nm). Figure 4.3 shows the linewidth measurement of the pump resonance in water in the highly under-coupled regime, which gives the quality factor ( $Q = \lambda/\Delta\lambda$ ). According to published values of the absorption coefficient of water at 970 nm ( $\alpha = 45 \text{ m}^{-1}$ ), the WGM loss in water should cause a reduction in measured Q to only  $2.3 \times 10^5$  [22]. Therefore, there exists an additional source of absorption, such as external contaminants carried to the microtoroid by water. Experiments in water normally lasted up to 8 hours, and at the end of this time a decrease in Q factor was observed. The effect of water on solgel silica may be different than on thermally grown silica. Future research will investigate any difference, which could be due to porosity difference between thermal and solgel silica.



Figure 4.3. Plot of microtoroid pump-mode resonance in water with Lorenzian fit for the undercoupled condition ( $Q = 1.3 \times 10^5$  at  $\lambda = 970.8$  nm). Data is taken by measuring the transmitted power along the fiber taper while the pump laser wavelength is scanned in time.

The Yb:SiO<sub>2</sub> microtoroid laser output power was measured as a function of the absorbed pump power, for operation in air and water (see Figure 4.4). The microtoroid cavity supports both clockwise and counterclockwise modes, but only the power of the clockwise mode is recorded. Laser output in water was observed in more than ten microtoroids, illustrating the reproducibility of laser operation in water. The laser turn-on pump threshold is 3  $\mu$ W in air and 15  $\mu$ W in water, with corresponding slope efficiencies of 1.6% (air) and 0.5% (water). In water, there is a clear threshold for laser operation as well as a linear dependence of output power on absorbed pump power (see Figure 4.5). The maximum output power in water is 2  $\mu$ W.



Figure 4.4. Comparison of measured laser output power as a function of absorbed pump power for 120  $\mu$ m diameter Yb<sup>3+</sup>:SiO<sub>2</sub> microtoroid in air and water



Figure 4.5. Plot of laser output in water at low pump powers showing the laser threshold

The predictions of the laser threshold model detailed in **Chapter 3** are compared with experiment [21]. The affect of the water was included in the model by decreasing the intrinsic quality factor to the actual value that was measured. In water (air), the experimental threshold, 15  $\mu$ W (3  $\mu$ W), is in reasonable agreement with the prediction of the model, 4  $\mu$ W (1  $\mu$ W). Table 4.1 summarizes the laser performance in air and water.

Environment	Q	Pth (exp)	Pth (sim)	Efficiency	Evanescent component
		$\mu \mathrm{W}$	$\mu \mathrm{W}$	%	%
air	$2.8{ imes}10^5$	3	1	1.6	0.6
water	$1.3{ imes}10^5$	15	4	0.5	3.6

Table 4.1. Comparison of microtoroid laser results in air and water

#### 4.4 Summary

In conclusion, the author has demonstrated a solid-state microcavity laser whose cavity mode extends into water. The ytterbium-doped silica gain medium was fabricated by solgel synthesis. The laser generates light with an output power of 2  $\mu$ W and threshold of 14  $\mu$ W. The Schawlow-Townes equation quantifies the fundamental laser linewidth ( $\delta\nu$ ) for a single mode laser ignoring any technical noise [86].

$$\delta\nu = \frac{\pi h\nu^3}{PQ^2} \tag{4.1}$$

Given the experimental values for laser power ( $P = 2 \ \mu$ W) and the water absorption limited quality factor ( $Q = 6.5 \times 10^5$ ) at 1.04  $\mu$ m, we estimate that the fundamental linewidth of the ytterbium silica laser presented in this work is 60 kHz, which is significantly narrower than the linewidth of an ultra-high-Q passive microcavity (of order 1 MHz). As a result, this laser can have higher sensitivity to biological molecules in aqueous solution than a passive resonator. The laser threshold, output power, efficiency, and emission frequency can be modified by a molecule located close to the microtoroid, providing several possible sensing mechanisms.

In addition to biological sensing, this novel active laser sensor could find application as a chemical sensor, or be utilized for research on thin-films and other material properties.