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Short vertical tube furnace for the fabrication of doped glass microsphere lasers

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We report on the design of an electric tube furnace that can be used for the fabrication of doped glass microsphere lasers. The tube furnace has a short hot zone of length 133 mm and is based on a quartz tube design. Doped laser glass particles, specifically Er:Yb phosphate glass (IOG-2), of ~1 μm diameter are blown into the furnace using a 60 ml syringe and microspheres ranging in size from 10 to 400 μm are collected at the output of the tube furnace in a Petri dish. The furnace operates at a wall temperature of ~900 °C and is capable of making microspheres from glasses with glass transition temperatures of at least 375 °C. High quality ($Q \sim 10^5$) whispering gallery modes have been excited within the microspheres by optically pumping at 978 nm via a tapered optical fiber.
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I. INTRODUCTION

As industry strives toward the miniaturization of optics-based telecommunication systems for increased bandwidth and data transfer, a demand for a reduction in the size of optical and optoelectronic components has risen. This has fueled interest in developing micron-sized laser sources that can be integrated into photonic systems.[1] There are many different designs of submillimeter sized lasers including microdisks,[2] microtoroids,[3] and dielectric microspheres.[4] The first laser action in a solid-state sphere was demonstrated by Baer[5] using Nd: yttrium aluminum garnet spheres with a diameter of 5 mm and a lasing threshold of 100 mW. Subsequently, experimental efforts focused on reducing laser thresholds and improving the quality factors. Nowadays, lasing thresholds at microwatt levels have been reached,[6,7] while, simultaneously, the size of the spheres has decreased dramatically to a few tens of microns.

One standard technique for producing a single passive (i.e., nondoped) silica microsphere is by heating the tip of a tapered optical fiber in the focused beam of a CO_2 laser;[8,9] surface tension pulls the melted glass tip into a spherical shape, thereby creating a whispering gallery mode (WGM) resonator. In order to make a single microsphere doped with rare-earth ions (e.g., Yb^{3+} or Er^{3+}), a micron-sized fiber must first be drawn from a bulk sample of the doped glass and the fiber tip is subsequently melted to form the microsphere.[10] Alternatively, the core of an optical fiber can be exposed by chemical etching and subsequently melted using a CO_2 laser to form a microsphere.

The above procedures are tedious if one desires more than one microsphere and a more time efficient method was devised which relies on the use of a microwave plasma torch.[11] A bulk sample of glass is ground into micron-sized particles which are dropped through the plasma torch. Another technique for fabricating multiple spheres relies on the same principles, but the glass powder is melted in an electric vertical tube furnace.[12,13] In this work the authors used a 1.5 m long aluminum tube heated to ~1300 °C to make solid and hollow glass microspheres. The heating elements covered 1 m of the vertical tube. A vertical tube furnace with a rotating plate at its bottom was used by Peng *et al.*[14] and, after falling from the furnace, the melted glass particles land on the spinning plate and roll off to form spheres. Chalcogenide glass microspheres have also been made using a vertical tube furnace,[15] whereby crushed glass powder was initially sieved to obtain the required particle size. Larger particles were separated from smaller particles by sedimentation in a liquid. The small particles are buoyant in the hot air of the furnace and so were injected into the bottom of the furnace and collected at the top. Larger particles could be dropped through the top of the furnace and collected at the bottom. The upper limit of attainable sphere size was restricted by the terminal velocity of the particles and the length of the furnace.

A novel method for the simultaneous production of many small spheres was demonstrated by Grillet *et al.*[16] A tapered optical chalcogenide fiber, 30 mm in length and 1 μm in diameter, was pumped at 1550 nm with 25 mW of power. Optical absorption combined with the small mode volume of the chalcogenide fiber caused the fiber taper waist to melt and break apart; surface tension quickly pulls the small pieces into spheres with diameters between 3 and 15 μm. Another interesting approach evaporated telluride glass from the end of an optical fiber using a CO_2 laser. Glass particles in the vapor cool to form spheres with diameters less than 10 μm.[17]

In this paper, we report on a simple and inexpensive method of fabricating rare-earth doped, solid glass microsphere lasers using a vertical tube furnace with a relatively

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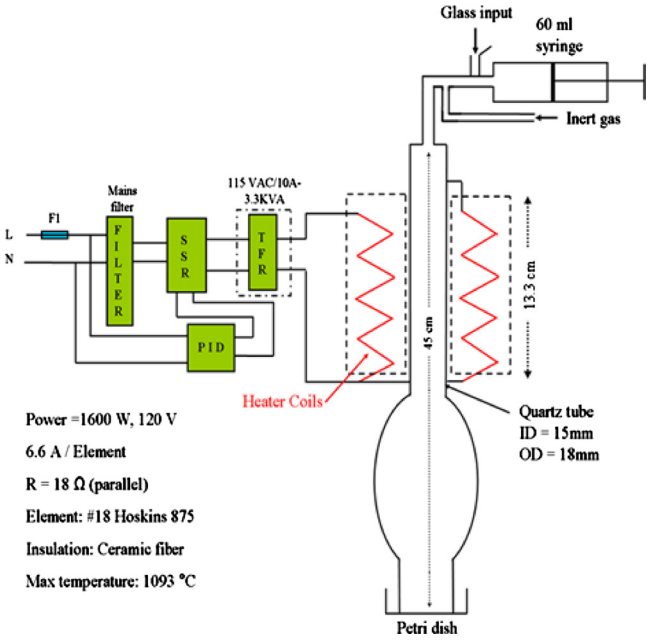


FIG. 1. (Color online) Experimental setup showing the PID control, the heater, the quartz tube, and the glass injection system. L: live, N: neutral, F1: fuse, PID: proportional, integral, and derivative, TFR: step down transformer, SSR: solid state relay.

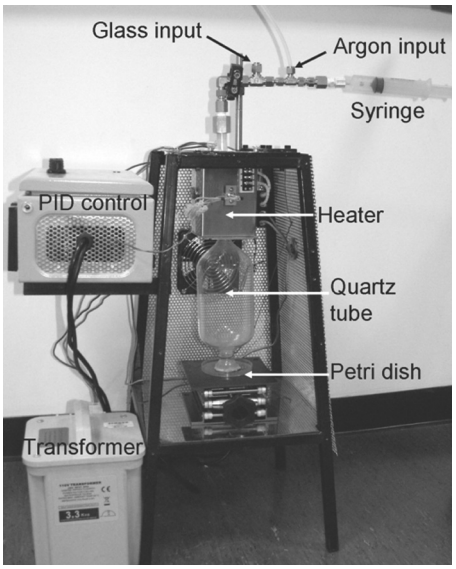


FIG. 2. A photograph of the vertical tube furnace.

short (~133 mm) hot zone, which relies on heating argon gas in a quartz tube. The setup is capable of simultaneously producing hundreds of high quality microsphere lasers, without any need to prefilter the particle size, by employing a novel glass injection system. After fabrication, the microspheres are optically pumped at 978 nm via a tapered optical fiber and high Q-factor WGM resonances have been observed in the microspheres.

II. EXPERIMENTAL DESIGN

In order to fabricate the microspheres at sufficiently high temperature, an industrial, single-heater-element, tube furnace, rated at 1600 W (120 V_{ac}), is used. The tube furnace is mounted vertically and a quartz tube 450 mm in length (with an inner diameter of 15 mm and an outer diameter of 18 mm) is placed inside the single element heater, as illustrated in Fig. 1. The space between the quartz tube and the furnace wall is packed with fiber glass for insulation purposes. An Omron proportional integral derivative (PID) controller is used to control the temperature setpoint of the furnace within ± 5 °C. The controller can provide temperature control action designed for specific process requirements by tuning the three constants in the PID controller algorithm. By allowing an under/overshoot of 5 °C, we can accurately repeat the process to the desired setpoint. A photograph of the furnace is shown in Fig. 2.

The furnace apparatus is mounted inside a frame to maintain its stability and verticality. The control box, which houses the PID controller, a main filter to eliminate transformer spiking on switch on, and a solid-state relay (SSR) to protect the control from load transients, is attached to the frame. An external, step down transformer from 220 to 120 V_{ac} is used to power up the heater coils. The furnace is 3.175 cm in diameter and is 13.3 cm long so the Watt density on the furnace walls is 6.1 W/cm^2. The heating element is 9 m long with 20.17 cm^2 of surface area per meter of wire, yielding a wire Watt density of 1.8 W/cm^2.

A right angle injection system has been designed to blow the doped glass powder safely into the heated area. A buffer of argon gas is injected into the quartz tube to prevent moisture from entering the spheres as they form. The argon is introduced at low flow rate of ~12 S l/min and this low flow is maintained constantly during the process to reduce the chimney effect, i.e., rising hot air currents which could push the spheres out of the system. The quartz tube has two temperature zones: a hot zone inside the tube furnace and a cold zone outside the tube furnace. A longer cold zone is required to quench the microspheres as they leave the hot zone to ensure high quality sphere formation.

III. DEMONSTRATION AND RESULTS

The glass used is a commercial laser glass fabricated by Schott. We use IOG-2, a phosphate glass doped with 3 wt % Er^{3+} and 4 wt % Yb^{3+}. The glass is ground to a powder using a mortar and pestle. The highest quality microspheres were obtained when the particle sizes are around 1 μm in the glass powder. When the set temperature of 900 °C within the furnace is reached, approximately 0.0005 g of crushed laser glass is placed in the upper tee junction of the system and the cap is closed. Using a syringe ~20 ml of argon gas is injected into the quartz tube in less than 1 s; this blows the glass material into the heated furnace tube area and prevents back flow of the particles. The cloud of hot glass particles collide and coalesce to form larger particles and surface tension pulls the particles into a spherical shape, thereby forming the microspheres. The spheres fall from the hot zone in the furnace into the cold zone, where they cool and solidify. They are then collected in a Petri dish attached to the end of the quartz tube. The Petri dish is in contact with the bottom of the quartz tube in order to seal the tube and reduce the

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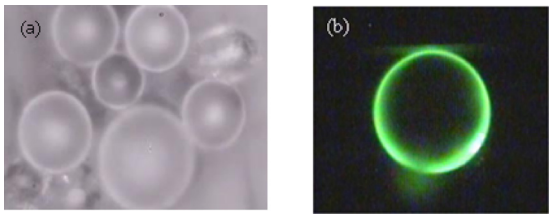


FIG. 3. (Color online) (a) Image of the microspheres made in the tube furnace, the largest sphere in the image is ~100 μm. (b) 55 μm microsphere laser pumped via a tapered optical fiber around 978 nm. The WGM is clearly evident and the tapered fiber coupler is in contact with the microsphere and at the top.

chimney effect. Optical images of the IOG-2 microspheres made in the tube furnace are shown in Fig. 3(a).

The spheres are visually inspected using an optical microscope; defect-free spheres in the Petri dish are selected and then glued to the 20 μm tip of tapered optical fiber using UV curing glue. The taper optical fiber glued to the sphere acts as a manipulation stem for the microsphere for ease of integration into experimental setups; the stem with sphere is attached to a three-dimensional stage so that the sphere can be manipulated in space. The microspheres are optically pumped at 978 nm via an adiabatically tapered optical fiber[18] with a waist diameter of 1 μm (measured using an optical resolution of 1 μm). The 978 nm light is converted to visible green light by the erbium ions in the glass in a process called upconversion.[19,20] An image of a 55 μm sphere under optical pumping at 978 nm is shown in Fig. 3(b). The 978 nm pump light also populates the $^4I_{15/2}$ metastable state of the erbium ions in the glass, resulting in light emissions around 1550 nm. These 1550 nm emissions are desired because they are used in the low loss transparency window of silica glass for optical telecommunications. The microspheres can be used, for example, as optical components in microphotonic circuits[21–23] or as multicolor light source micro-optical sensors.[24,25]

A typical single mode lasing emission from a 55 μm microsphere made in the tube furnace is shown in Fig. 4. The inset of Fig. 4 shows the WGM structure below lasing threshold. WGM propagation in circular geometry microresonators is a well-studied phenomenon.[1,23,26] In the experiments reported here, lasing thresholds of around

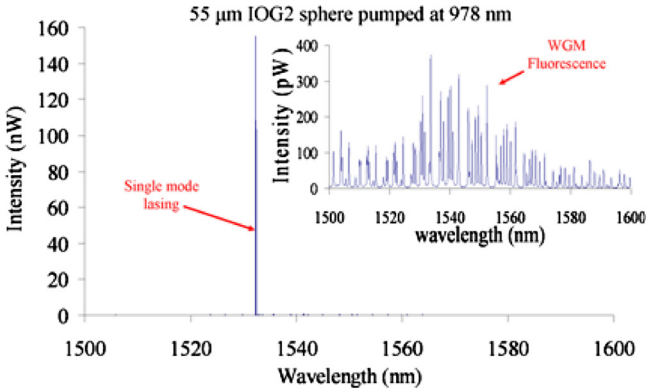


FIG. 4. (Color online) Single mode lasing at 1532 nm from a 55 μm IOG-2 microsphere pumped at 978 nm. Typical observed lasing thresholds are ~100 μW. Inset: WGM fluorescence from the same microsphere.

150 μW are routinely observed and the measured Q-factors ($Q=\lambda/\Delta\lambda$) of the microspheres of $\sim 1.5\times 10^5$ are limited by the resolution of the spectrum analyzer used (Anritsu MS9710B).

IV. CONCLUSION

In conclusion, we have designed a vertical tube furnace suitable for the fabrication of high quality microsphere lasers from crushed, doped laser glass. The tube furnace has a hot zone 133 mm long and many microspheres can be fabricated simultaneously. The system is easy to construct, inexpensive, and has the added advantage that it is very simple to operate compared to other systems.[11,15,17] We have demonstrated the functionality of the system by fabricating microspheres from IOG-2 laser glass, with a glass transition temperature of 375 °C and lasing in these spheres has been observed.

The temperature range achievable using the furnace design covers most of the doped laser glasses currently available commercially, e.g., heavy metal fluoride glasses,[27] chalcogenide glasses,[28] and phosphate glasses.[29] However, it should also be possible to make microspheres from glasses with a slightly higher transition temperature. By blowing the glass powder into the sealed tube furnace instead of dropping it through an open furnace there is no need to prefilter the particle size. It is sufficient to just crush the glass into a powder with a uniform particle size of around 1 μm, producing microspheres with diameters ranging from 10 to 400 μm. While many low quality microspheres are also produced during the process, the large number of spheres ensures that there are many of high quality fabricated and these can simply be selected postfabrication. It may also be worthwhile to mix different glass powders together and drop them simultaneously through the furnace to make a hybrid sphere composed of more than one type of doped glass. Future work will consider this aspect.

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