Infrared-laser and upconversion luminescence in Ho3+-Yb3+ codoped tellurite glass microsphere

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In this letter, Ho3+-Yb3+ codoped tellurite glasses were fabricated using the melting-quenching method to produce optical microsphere cavities. A 980 nm excitation laser was coupled into the microsphere through a fiber taper, providing lasing at around 2.0 μm and upconversion luminescence in the visible (380-780 nm).

**OCIS codes:***(140.3460) Lasers; (190.7220) Upconversion; (140.3945) Microcavities; (160.4670) Optical materials.*

Rare-earth ions doped materials have been widely used in laser devices and fiber amplifiers owing to the numerous well-defined transition modes involving the 4f shell of their ions [1]. Among the rare-earth ions, Ho3+ and Tm3+ are excellent candidates for fabricating ~2.0 µm lasers, which have much potential in laser medical systems and sensing [2]. Compared with Tm3+, Ho3+ has an emission cross-section approximately five times larger, a longer radiative decay lifetime and a longer operating wavelength [3, 4]. However, Ho3+ cannot be pumped directly with readily available 980 or 808 nm commercial laser diodes, and sensitization using other rare-earth ions with strong absorption bands at 980 or 808 nm in the host glass has been investigated [5-7]. Yb3+ exhibits a large absorption cross section at 980 nm and its single 4f-4f electronic transition 2F5/2-2F7/2 matches the transitions among Ho3+ intermediate states, enabling an efficient energy transfer processes from Yb3+ to Ho3+. A Ho3+-Yb3+ codoped 2.05 µm fiber laser was reported in 2014 [8]. Ho3+-Yb3+ codoped silica microspheres have been synthesized using sol-gel method, resulting in a microsphere laser [9]. In addition, the Ho3+-Yb3+ codoped system is also suitable for upconversion luminescence [10, 11]: three different bands in the visible (380-780 nm) have been observed in Ho3+ doped glasses sensitized with Yb3+ under 980 nm laser excitation.

Tellurite glasses with good mechanical and thermal properties have an excellent transparency from visible to near-mid infrared region (400 nm-6 μm) [12]. Different from silica glass with easier occurrence of quenching of rare-earth ions, tellurite glasses that are based on the glass network former TeO2 are capable of dissolving a much higher concentration of rare-earth ions since the rare-earth ions replace network modiﬁers in the glass [13]. In addition, tellurite glass can efficient reduce the non-radiative losses since it has amongst the lowest phonon energies (650-800 cm−1) of all oxide glasses. Therefore, it is a type of desirable material for the near-mid infrared region lasing and efficient upconversion luminescence processes [14]. Some tellurite glass microsphere lasers have reported such as Tm3+ doped or Tm3+-Ho3+ codoped ~2.0 μm lasing [15-17] and Er3+ doped ~1.55 μm lasing [18].

In this work, lasing at ~2.0 μm and upconversion luminescence in the visible is the first time reported in Ho3+-Yb3+ codoped tellurite glass microspheres. Microsphere resonators provide high quality factors and small mode volumes to three-dimensional whispering gallery modes (WGM), resulting in low threshold and narrow linewidth lasers [19-21]. The Ho3+-Yb3+ codoped tellurite glass microsphere lasing at ~2.0 μm has significant potential in high-sensitivity chemical and biological sensing [22, 23], while the upconversion luminescence in the visible can be used in imaging.

Ho3+-Yb3+ codoped tellurite glass samples (72TeO2−20ZnO −5Na2CO3−2Y2O3−0.8Yb2O3−0.2Ho2O3) were prepared using a conventional melt-quenching method. 30 g of high-purity TeO2 (99.99%), ZnO (99.99%), Na2CO3 (99.99%), Y2O3 (99.9%), Yb2O3 (99.99%), and Ho2O3 (99.99%) powders were mixed and melted in a corundum crucible at 900 °C for 30 min, then poured into preheated stainless-steel molds and annealed around the glass transition temperature (~350 °C) for 3 h. The resulting glasses were then cut and polished for the optical property measurements. Fig. 1 (a) shows the absorption spectra of the Ho3+-Yb3+ codoped tellurite glass sample at room temperature. Two absorption bands are highlighted: one corresponding to the transitions from the 2F7/2 ground state to the 2F5/2 level of Yb3+; the other corresponding to the transition starting from the 5I8 ground state to the 5I7 level of Ho3+. The absorption spectra confirm that Yb3+ has a strong absorption at ~1.0 µm in the Ho3+-Yb3+ codoped tellurite glass. Fig. 1 (b) shows the emission spectra of the Ho3+-Yb3+ codoped tellurite glass sample at room temperature. On excitation by a 980 nm laser, a ~2.0 µm emission is observed, attributed to the characteristic 5I7→5I8 transitions of Ho3+ [24, 25]. It is well known that Ho3+ cannot directly absorb light at 980 nm. The ~2.0 µm emission originates from the energy transfer from Yb3+ to Ho3+. The inset of Fig. 1 (b) shows the energy-level diagram of Ho3+ and Yb3+ and relative transitions. Yb3+ ions are excited to the 2F5/2 level through the absorption of the 980 nm light; the energy transfers (ET), through a phonon-assisted process, to the 5I6 level of Ho3+, which is close to the 2F5/2 state of Yb3+ (the energy gap is about 1540 cm-1) [26]; finally, ions in the 5I6 level of Ho3+ non-radiatively decay to the 5I7 level and transfer to the 5I8 level with the ~2.0 µm emission.

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Fig. 1. (a) Absorption spectra and (b) Emission spectra of the Ho3+-Yb3+ codoped tellurite glass. Inset: energy-level diagram of Ho3+-Yb3+ codoped system and relative transitions resulting in the ~2.0 µm emission.

In order to prepare Ho3+-Yb3+ codoped tellurite glass microspheres, the Ho3+-Yb3+ codoped tellurite glass filaments were made from the molten glass using a glass rod tip, similar to the method described elsewhere [27]. A CO2 laser beam was used to cut the taper and the remaining glass tip was reheated to produce a Ho3+-Yb3+ codoped tellurite glass microsphere exploiting the surface tension of the molten glass. An 80 μm diameter microsphere is shown in Fig. 2 (a). The experimental setup used to measure the microsphere laser spectral characteristics is shown in Fig. 2 (b). The tapered fiber used for launching the pump light and collecting the output laser was fabricated by heating and stretching a strand of 1060XP single mode fiber. The tapered fiber waist diameter used in the experiments was controlled at ~2.0 μm. A 980 nm laser diode (OFLD1000, Ovlink, China) was used as pump source and an optical spectrum analyzer (OSA) (AQ-6375, Yokogawa, Japan) was used for spectroscopic analysis. The coupling process between the taper fiber and microsphere was accurately controlled using two 3-Axis stages (MBT616D, Thorlabs, U. S.) under a microscope.

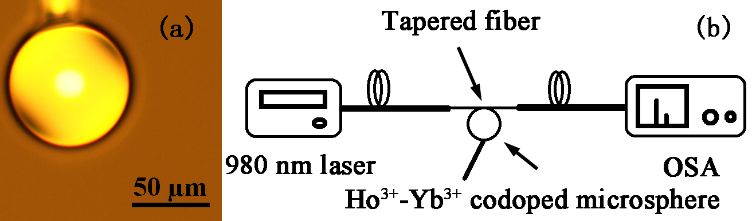


Fig. 2. (a) Optical image of the Ho3+-Yb3+ codoped tellurite glass microsphere (b) Schematic of the experimental setup.

As the 980 nm laser was coupled into the Ho3+-Yb3+ codoped tellurite glass microsphere, a multimode laser at ~2.0 µm was observed. The whispering gallery modes not only depend on the polarized state of light, but also depend on radial mode number (*r*), azimuthal mode number (*l*) and angular mode number (*m*). In the experiment, three laser peaks (λ=2065.0, 2069.4 and 2072.3 nm) can be observed in Fig 3 (a) when the pump power is 2.48 mW. For a microsphere resonator, the free spectral range (FSR) is defined as the wavelength spacing between two adjacent fundamental modes and it can be obtained by formula: FSR=λ2/(*πneff*D) [28]. Where λ is the resonant wavelength, *neff* is the effective refractive index, and D is the diameter of the microsphere. According to the equation, the FSR was calculated to be 7.3 nm at 2068 nm, assuming that the *neff* is about 2.3 [29] and the microsphere diameter is 80 μm. This value is exactly equal to the wavelength spacing between the first (λ=2065.0 nm) and third (λ=2072.3 nm) peaks. Therefore, the first and third peaks belong to different azimuthal mode numbers of the fundamental mode (*r*=1 and *l =* |*m*|). The second peak (λ=2069.4 nm) is the higher order mode lasing. The higher order modes laser was excited as the pump power reaches the threshold of higher order modes. However, when the coupling taper was placed further from the equatorial plane, the higher order angular modes laser was easily excited. In addition, the absorbed power of microsphere has a close relationship with the coupling gap. When the coupling distance approaches the critical coupling, more pump power into the microsphere and then the higher order modes laser will be more easily motivated.Yet, switching between single mode and multimode laser output can be realized by adjusting the relative position between taper and microsphere [30, 31]. Fig. 3 (b) shows the single mode operation when the taper is in physical contact at the microsphere equator. The central wavelength is 2071.4 nm and the power is 13.7 nW when the pump power reaches 1.55 mW. The single mode laser power as a function of pump power is show in Fig. 4. The laser threshold is less than 1.4 mW and the Ho3+-Yb3+ codoped tellurite glass microsphere laser shows a linear relationship with the pump power above threshold. In the experiment, the laser has maintained single mode operation without any sign of saturation even when the pump power was increased to 1.96 mW. The lasing efficiency is only about 0.006% since there is the phase mismatch as the refractive index is different between the tapered optical fiber (the refractive index is about 1.4 [29]) and the tellurite glass microsphere (the refractive index is about 2.3 [29]). In addition, limited by the experimental conditions in this investigation, critical coupling was not attained and this leads to low coupling efficiency. When a broadband light source (SC YSL SC-series, China) was used to illuminate the microsphere through the tapered fiber, the WGM spectrum was obtained from the OSA (AQ-6370, Yokogawa, Japan) with a resolution of 0.02 nm, providing a microsphere quality factor estimation as large as 105 at λ~1.5 μm. However, the OSA resolution limits the microsphere quality factor measurement accuracy. But it is clear that the true value or the values measured by instruments with higher resolution will not be lower than 105.



Fig. 3. (a) Multi-mode laser emission (pump power set to 2.48 mW) Inset: a zoom-in view of the multi-mode laser emission (b) Single mode laser emission (pump power set to 1.55 mW).



Fig. 4. Output power as a function of pump power.

Fig. 5 (a) shows the upconversion luminescence spectra of Ho3+-Yb3+ codoped tellurite glass microsphere, recorded at room temperature in the visible using an OSA (USB4000, Ocean Optics, U. S.). Three types of luminescence bands, peaking at 543, 657 and 750 nm were be observed under 980 nm laser excitation. To discuss the processes involved in the upconversion luminescence a simplified energy-level diagram of the Ho3+-Yb3+ codoped system is shown in Fig. 5 (b). The upconversion mechanism is as follows [10, 32]:

(a) Yb3+ is excited to the 2F5/2 level from 2F7/2 level through the absorption of the 980 nm light.

(b) The energy transfer (ET1) from the 2F5/2 level of Yb3+ to the 5I6 level of Ho3+ then occurs assisted by a phonon.

(c) An Excited State Absorption (ESA) process from the 5I6 level to the 5F4, 5S2 levels of Ho3+ occurs with the energy transfer (ET2) from the 2F5/2 level of Yb3+ to 5F4, 5S2 levels of Ho3+.

(d) The ions in 5F4, 5S2 levels of Ho3+ decay to the 5F5 level and transfer to the 5I8 level with the 657 nm emission.

(e) The 750 nm emission occurs with the transition from the 5F4, 5S2 levels to the 5I7 level of Ho3+.

(f) The 543 nm emission occurs with the transition from the 5F4, 5S2 levels to the 5I8 level of Ho3+.





Fig. 5. (a) Upconversion luminescence spectra of Ho3+-Yb3+ codoped tellurite glass microsphere when excited at 980 nm (b) Energy-level diagram of Ho3+-Yb3+ codoped system and relative transitions for upconversion emission.

There are many energy transfer and excited state absorption processes under the 980 nm laser excitation in the Ho3+-Yb3+ codoped system. However, the transfer efficiency is largely related to the ion doping concentration, the host glass phonon energy and temperature. As the back transfer efficiency increases with temperature, it is difficult to realize population inversion at room temperature [33]. Therefore, upconversion lasing has not been observed in this experiment.

In conclusion, a Ho3+-Yb3+ codoped tellurite glass microsphere lasing at ~2.0 μm has been demonstrated. Single mode and multimode laser operation are observed using a 980 nm laser diode as a pumping source and a fiber taper-microsphere coupling setup. Upconversion luminescence in the visible was observed and ascribed to the dopants cooperative behavior. This Ho3+-Yb3+ codoped tellurite glass microsphere system is expected to be used in imaging system, high-sensitivity chemical and biological sensing.

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