# Fluoride fiber lasers

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### **ABSTRACT**

Simultaneous laser oscillation near  $2\mu m$  and  $2.3\mu m$  on the  $^3H_4$  -  $^3H_6$  and  $^3F_4$  -  $^3H_5$  transitions respectively has been demonstrated in a thulium-doped fluorozirconate fiber when pumping at 791nm. Diode-pumped operation of the  $^3H_4$  -  $^3H_6$  transition has also been achieved.

Pulsed laser emission at  $1.72\mu m$  has been observed on the  $^4S_{3/2}$  -  $^4I_{9/2}$  transition in an erbium-doped fluorozirconate fiber when pumping at 488nm or 514nm.

## 1. INTRODUCTION

The low phonon energy fluoride glasses which hold out the prospect of ultralow loss communications systems also make interesting host glass matrices for rare earth laser activator ions. Compared with a silica fiber host many more fluorescing metastable multiplets are available, some of which have  $\approx 100\%$  radiative quantum efficiency, reflecting the higher order of multiphonon emission processes. Rare earth doped fluoride fibers thus offer the prospect of new laser transitions, low thresholds and also the efficient generation of upconverted fluorescence by successive excitation steps between long-lived metastable levels. This paper contains a discussion of recent results on  $Tm^{3+}$  and  $Er^{3+}$ -doped fluorozirconate fibers which illustrate some of these features.

Laser sources operating in the near infra-red can be expected to find applications in areas such as the sensing of gas molecules and also have uses in medicine. Thulium is a particularly attractive ion for such applications since it emits at wavelengths between  $\approx 1.6 \mu m$  and  $\approx 2.1 \mu m$  and so covers absorption bands of water vapour (1.88  $\mu m$ , 1.91  $\mu m$ ), liquid water (1.94  $\mu m$ ), carbon dioxide (1.96  $\mu m$ , 2.01  $\mu m$  and 2.06  $\mu m$ ) and methane (a C-H bond overtone is centred at 1.668  $\mu m$ ). A further attraction of Tm<sup>3+</sup> is that there is a strong ground state absorption near 790 nm which means that it is possible to use an AlGaAs semiconductor diode laser as the pump source. We have previously studied the performance of Tm<sup>3+</sup>-doped silica fiber lasers and have shown that they may be efficiently pumped at wavelengths near 800 nm and offer a tuning range in excess of 250 nm near  $2 \mu m$  as well as offering high power operation (1W output power) when pumped with a Nd:YAG laser.

A problem with the silica host is that the upper level suffers from fast non-radiative decay (the radiative quantum efficiency of the  ${}^3H_4$  level is less than 20%) leading to a higher threshold than for other fiber lasers such as Nd<sup>3+</sup>. For this reason a fluorozirconate fiber was considered as an alternative host since non-radiative decay rates via multiphonon emission are less than for oxide glasses such as silica. It is the reduced multiphonon emission rate from the  ${}^3F_4$  level in a fluorozirconate host which allows laser operation at  $0.8\mu m^8$  ( ${}^3F_4 - {}^3H_6$ ),  $1.47\mu m^8$  ( ${}^3F_4 - {}^3H_4$ ) and  $2.3\mu m^{8,9}$  ( ${}^3F_4 - {}^3H_5$ ) to take place. Additionally the reduced non-radiative decay rates make upconversion more efficient in the fluorozirconate glass which has allowed a krypton ion laser (operating in the red) pumped blue upconversion laser to be demonstrated. 10

Erbium is another attractive laser ion since in addition to operating at  $1.55\mu m$  in the lowest loss window for silica telecommunications fiber it also has many other radiative transitions when doped into a fluorozirconate host allowing many possible laser transitions. To date laser oscillation in  $Er^{3+}$ -doped fluorozirconate fiber has been obtained at  $0.85\mu m^{11}$ ,  $0.99\mu m^{12}$ ,  $1.55\mu m^{13}$ ,  $2.7\mu m^{14}$  and recently at 1.66 and  $1.72\mu m^{15}$ . The work on erbium described in this paper concentrates on laser oscillation at  $1.66\mu m$  and  $1.72\mu m$ .

wavelengths near the C-H bond overtone of methane which suggests that laser sources using Er<sup>3+</sup>-doped fluorozirconate fiber as the active medium may find applications in sensing.

# 2. THULIUM-DOPED FLUOROZIRCONATE FIBER LASERS OPERATING NEAR 2μm PUMPED AT WAVELENGTHS AROUND 800nm

The fiber used in these experiments was of the standard ZBLANP composition and was fabricated using a casting technique<sup>16</sup>. The fiber was doped with 740ppm by weight  $Tm^{3+}$  ions and had core and cladding diameters of 40 and  $80\mu$ m respectively.

Initial spectroscopic measurements were made using a styryl 9M dye laser operating at 800nm as the pump source corresponding to absorption from the  ${}^{3}H_{6}$  ground state to the  ${}^{3}F_{4}$  excited state as illustrated in the partial energy level diagram, Fig. 1. Emission at around 2.3 $\mu$ m and 1.47 $\mu$ m was observed (corresponding to decay from the  ${}^{3}F_{4}$  level to the  ${}^{3}H_{5}$  and  ${}^{3}H_{4}$  levels respectively) and at around 1.8 $\mu$ m (decay from the  ${}^{3}H_{4}$  level to the  ${}^{3}H_{6}$  ground state. An emission spectrum of light escaping radially from the fiber for wavelengths between 1.2 and 2.2 $\mu$ m is shown in Fig. 2. Since this light has not been guided by the fiber it is not distorted by reabsorption losses which would be particularly significant for the quasi-three-level  ${}^{3}H_{4}$  -  ${}^{3}H_{6}$  transition. We attribute the ripple on this spectrum to noise although Allain et al<sup>8</sup> who excited the  ${}^{3}H_{4}$  -  ${}^{3}H_{6}$  transition with a krypton laser observed ripple which was repeatable and suggested that it was not caused by noise. The lifetimes of the  ${}^{3}F_{4}$  and  ${}^{3}H_{4}$  levels were measured to be 1.1ms and 6.4ms respectively which compare to calculated radiative lifetimes of 1.3ms and 6.8ms confirming the low non-radiative decay rates from these levels in a fluorozirconate host.

The pump source used for obtaining diode-pumped operation of the  $^3H_4$  -  $^3H_6$  transition was a Sony SLD303 broad stripe diode operating at 795nm with a nominal maximum output power of 500mW. The diode output was collected by a 8mm focal length diode collimating lens and reshaped by a 45mm focal length cylindrical lens. Light was launched into the fiber by means of a x10 microscope objective, with the positions of the optics being determined by mode-matching calculations. The launch efficiency of light from the diode was determined by launching into undoped fiber of similar core diameter and numerical aperture to the  $Tm^{3+}$ -doped fiber.

A standard Fabry-Perot cavity was constructed by butting the cleaved ends of a 30cm length of fiber against dielectric mirrors of >99% reflectivity at the lasing wavelength with index matching fluid being used to minimize the butt losses at the fiber/mirror interface. The fiber length chosen was a compromise between ensuring good absorption of the pump light and minimizing reabsorption losses at the lasing wavelength. For the 30cm length of fiber used, approximately 50% of the launched light was absorbed.

The launched power required to obtain continuous-wave laser oscillation at  $1.972\mu m$  was found to be approximately 40mW (20mW absorbed). A maximum output power of  $\approx 200\mu W$  was measured for the maximum available launched power of 100mW (Fig. 3). This corresponds to a slope efficiency of approximately 0.3% with respect to launched power (0.6% with respect to absorbed power). Attempts were made to obtain laser oscillation near  $2\mu m$  when using higher values of output coupling. However, it was found that threshold could not be reached even when using output couplers of only 5% transmission. For this reason experiments were carried out using a Ti:sapphire laser as the pump source since the higher excitation intensities afforded by the pump source allowed threshold to be reached with output couplers with transmission as high as 10%. Additionally the tunability of this source meant that the excitation wavelength could be tuned over the entire  ${}^{3}H_{6} - {}^{3}F_{4}$  absorption band unlike the diode laser where temperature tuning only allowed pump wavelengths as short as 795nm to be used.

The greater power which could be launched into the core from the Ti:sapphire pump source meant that longer fiber lengths could be used for obtaining oscillation near  $2\mu m$ . Various lengths of fiber were used in a Fabry-Perot cavity as described above with the highest output powers being obtained for a 1m fiber length. The pump wavelength was set to 791nm since this wavelength gave maximum output power from the fiber

laser. The output power as a function of pump power (Fig. 4) was measured for output couplers of <1%, 5% and 10% transmission at the laser wavelengths which were measured to be  $2.000\mu m$ ,  $1963\mu m$  and  $1.960\mu m$  respectively. All of these laser wavelengths fall outside the tuning range ( $1.84\mu m$  to  $1.95\mu m$ ) reported for this transition by Allain et al<sup>8</sup>. The reason for the laser wavelength being dependent upon the output coupler used was that the 5% and 10% output couplers had a minimum reflectivity near  $1.9\mu m$  where as the high reflector had a flat transmission band between  $1.7\mu m$  and  $2.1\mu m$ . As expected the highest slope efficiency (3.2% with respect to incident power, 5.3% with respect to launched power, 5.9% with respect to absorbed power) was obtained when using the 10% output coupler although the highest output power (7.9mW) was recorded when using the 5% output coupler as a consequence of a lower threshold.

The slope efficiencies measured for the  $Tm^{3+}$ -doped fluorozirconate fiber laser operating on the  $^3H_4$  -  $^3H_6$  transition are much lower than for a  $Tm^{3+}$ -doped silica fiber laser<sup>5</sup>. This is in part a consequence of the reduced multiphonon emission rates in the fluorozirconate host. In silica nearly all of the ions excited to the  $^3F_4$  level decay via multiphonon emission to the  $^3H_4$  upper laser level. However, in the fluorozirconate host the main decay route ( $\approx 90\%$  based on the data of Guery et al<sup>17</sup>) from the  $^3F_4$  level is through the  $^3F_4$ -radiative transition with the emission of photons near 800 nm. Thus only a small fraction of the ions excited to the  $^3F_4$  level give rise to the emission of photons corresponding to the  $^3H_4$  -  $^3H_6$  transition. However, the threshold for the  $Tm^{3+}$ -doped fluorozirconate and silica fiber lasers may be expected to be comparable (if the fiber core diameters and numerical apertures are the same) since below threshold most  $Tm^{3+}$  ions excited to the  $^3H_4$  level ( $\approx 80\%^5$ ) in a silica host decay non-radiatively unlike in the fluorozirconate host where essentially all ions excited to this level decay radiatively.

The best way of enhancing the pumping efficiency of the  ${}^3H_4$  level in the fluorozirconate host would be by using a much higher dopant concentration (>1%). At higher dopant levels a cross-relaxation mechanism between neighbouring  $Tm^{3+}$  ions becomes efficient which gives rise to two ions being excited to the  ${}^3H_4$  upper laser level for each pump photon absorbed. This scheme is illustrated in Fig. 5. However, since  $Tm^{3+}$ -doped fibers of high concentration were not available an alternative scheme for improving the performance of a laser operating on the  ${}^3H_4-{}^3H_6$  transition needed to be considered. The method used was to have simultaneous laser oscillation on the  ${}^3F_4-{}^3H_5$  transition and use this stimulated emission to increase the branching ratio from  ${}^3F_4$  to  ${}^3H_4$ .

A Fabry-Perot cavity was constructed using an 86cm length of fiber (the same piece of fiber as used above but with recleaved ends) butted against a mirror of >99% reflectivity at wavelengths between 1.8  $\mu m$  and 2.4  $\mu m$  at the input end of the fiber and an output coupler of  $\approx 3\%$  transmission between 1.8  $\mu m$  and 2.4  $\mu m$ . The Ti:sapphire pump wavelength used was again 791nm. Using this arrangement laser oscillation was observed on both the  $^3H_4-^3H_6$  and  $^3F_4-^3H_5$  transitions at wavelengths of 1.94  $\mu m$  and 2.305  $\mu m$  respectively. A plot of the fiber laser output power at the two wavelengths as a function of launched pump power is shown in Fig. 6. The fact that the threshold for the two transitions was similar is entirely coincidental; for example, when a longer fiber length was used the threshold for the  $^3H_4-^3H_6$  transition increased as a consequence of increased reabsorption losses while the threshold for oscillation on the  $^3F_4-^3H_5$  transition decreased since more pump power was absorbed. The slope efficiencies at 1.942  $\mu$  and 2.305  $\mu$  were measured to be 8.3% and 18.8% respectively with respect to launched power (5.0% and 11.3% with respect to incident power). Thus it can be seen that laser oscillation on the  $^3H_4-^3H_6$  transition was found to be more efficient when simultaneous oscillation took place on the  $^3F_4-^3H_5$  transition. However the improvement in efficiency was such that it may have resulted from the construction of a cavity of lower loss. Further experiments are required to examine the effect of simultaneous oscillation on the two transitions, ideally on monomode fiber where both transitions may be pumped many times above threshold. In passing it is interesting to note that despite using higher excitation intensities than Allen and Esterowitz9, no saturation of output power at 2.3  $\mu$  was observed. Allen attributed the saturation of output power to a build-up of population in the  $^3H_4$  level which in the work described here is not a problem, since laser emiss

# 3. ERBIUM-DOPED FLUOROZIRCONATE FIBER LASERS OPERATING AT 1.66μm AND 1.72μm

The Er³+-doped fiber used for this work was of the standard ZBLANP construction and was fabricated by a casting technique¹6. The core diameter was  $40\mu$ m and the Er³+ concentration 977ppm by weight. Pump light at 514nm (corresponding to absorption from the ⁴ $I_{15/2}$  ground state to the  ${}^2H_{11/2}$  excited state as shown in Fig. 7) was mechanically chopped and launched into the fiber by means of a x5 microscope objective. As noted by Brierley et al¹¹, nearly 20 separate transitions are apparent. A fluorescence spectrum for wavelengths between 1.4µm and 1.9µm is shown in Fig. 8. The three peaks at 1.55µm, 1.66µm and 1.72µm are believed to correspond to transitions between the  ${}^4I_{13/2}$ - ${}^4I_{15/2}$ ,  ${}^2H_{11/2}$ - ${}^4I_{9/2}$  and  ${}^4S_{3/2}$ - ${}^4I_{9/2}$  levels respectively. Fig. 8 is for light which has been guided by a 40cm length of fiber and so is distorted by the reabsorption of short wavelength emission from the  ${}^4I_{13/2}$ - ${}^4I_{15/2}$  transition. When looking at light emitted radially from the fiber the peaks at 1.66µm and 1.72µm were not as apparent. Fluorescence decay from the  ${}^4S_{3/2}$  level was found to be exponential with an e-¹ decay time of approximately 500µs. The lifetime of the  ${}^4I_{9/2}$  level was estimated to be approximately 1ms by studying emission at 785nm from this level to the ground state, although since this level was populated through other metastable levels (e.g.  ${}^4S_{3/2}$ ) this measurement may be inaccurate.

To investigate laser behaviour a 40cm length of fiber with cleaved ends was butted at either end against dielectric mirrors of >99% reflectivity between  $1.65\mu m$  and  $2.1\mu m$  and which had a transmission of 85% at 514nm. Index matching fluid (liquid paraffin) was used to minimize butt losses at the fiber/mirror interfaces. Pulsed laser action at  $1.72\mu m$  was observed with a threshold pump power of 200mW incident on the launch objective (150mW was launched into the fiber and 90mW absorbed). This wavelength corresponds to the  $^4S_{3/2}$ - $^4I_{9/2}$  transition. Laser action at  $1.66\mu m$  has also been observed and this may correspond to the  $^2H_{11/2}$ - $^4I_{9/2}$  transition since the  $^2H_{11/2}$  and  $^4S_{3/2}$  levels are in thermal equilibrium  $^{18}$ . Alternatively the transition may be from the  $^4S_{3/2}$  level to a lower Stark level of the  $^4I_{9/2}$  level than for laser emission at  $1.72\mu m$ . Pulsed laser emission at  $1.72\mu m$  has also been observed when pumping at 488nm, with a similar threshold to that at 514nm.

Half the fiber was then immersed in liquid nitrogen to cool the fiber to 77K. Pulsed laser emission at  $1.66\mu m$ ,  $1.68\mu m$  and  $1.72\mu m$  was observed with a threshold pump power of 400mW. Continuous-wave emission at  $1.60\mu m$  ( $^4I_{13/2}$ - $^4I_{15/2}$  transition) was also observed after the pulsed emission had terminated. The threshold for all transitions was found to be approximately 400mW of pump power incident on the launch objective.

The reason for pulsed, self-terminating emission at  $1.72\mu m$  is that the fluorescence lifetime measurements suggest that the lower laser level has a longer lifetime than the upper laser level. One possible way of enforcing continuous-wave operation would be to co-dope with a different ion and remove rapidly population from the lower laser level through energy transfer. Alternatively a pump wavelength which suffers from excited state absorption from the  ${}^4I_{9/2}$  level could be used to remove population, a technique which has allowed cw operation on the normally self-terminating  ${}^4I_{11/2}-{}^4I_{13/2}$  transition  ${}^{14}$ . From a practical viewpoint, it would be desirable to pump the laser with an AlGaAs diode laser near 800nm. It was found that for an excitation power of 100mW at 800nm, the efficiency of populating the  ${}^4S_{3/2}$  level was approximately 20% of that under direct excitation from 514nm light. It may, therefore, be possible to use upconversion to pump the  ${}^4S_{3/2}-{}^4I_{9/2}$  transition in a monomode fiber with a laser diode.

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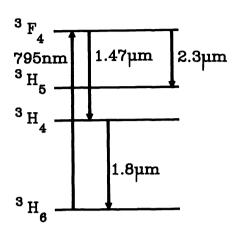


Fig. 1 Partial energy level diagram for Tm<sup>3+</sup>-doped fluoro-zirconate fiber

# fluorescence intensity

Fig. 2 Fluorescence spectrum for Tm<sup>3+</sup>-doped Fluorozirconate fiber excited at 800nm

wavelength (nm)

1800

1600

1200

1400

2000

<del>22</del>00

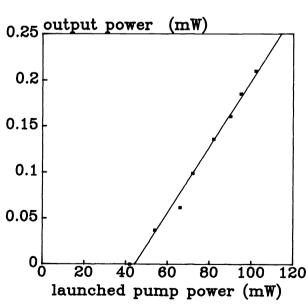


Fig. 3 Output power at 1.972µm against launched pump power for a diodepumped Tm<sup>3+</sup>-doped fluorozirconate fiber laser

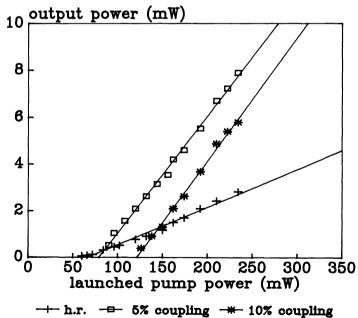


Fig. 4 Output power against launched pump power for  $Tm^{3+}$ -doped fluorozirconate fiber lasers operating near  $2\mu m$ 

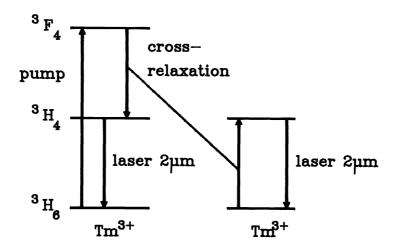


Fig. 5 Possible Tm<sup>3+</sup>-Tm<sup>3+</sup> cross relaxation process

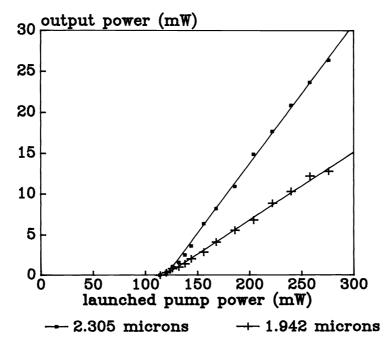


Fig. 6 Output power at  $1.94\mu m$  and  $2.30\mu m$  for  $Tm^{3+}$ -doped fluorozirconate fiber laser operating simultaneously on the  $^3H_4-^3H_6$  and  $^3F_4-^3H_5$  transitions

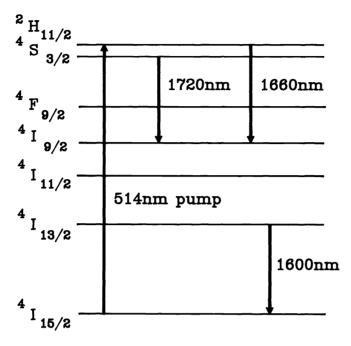


Fig. 7 Partial energy level diagram for Er<sup>3+</sup>-doped fluorozirconate fiber

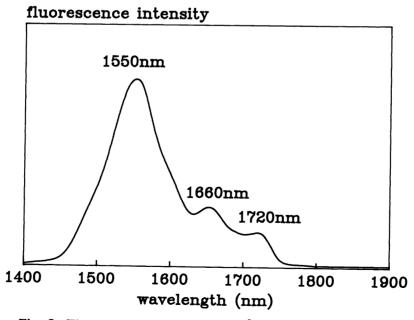


Fig. 8 Fluorescence spectrum for Er<sup>3+</sup>-doped fluorozirconate fiber excited at 514 nm