#### **Response to Examiners**

#### External Examiner 1 (Prof. Dr. Bishnu Pal, IIT Dehli)

(All corrections of Prof. Dr. Bishnu Pal are highlighted in red in the corrected thesis)

# 1. Conclusion after each chapter is too long. Should be reduced significantly in consultation with thesis advisors

The conclusions have been shortened and summarised. The title of the section has also been changed, from 'Conclusion' to 'Summary and Conclusion', as it is felt that this would more appropriately reflect the content of the section (as a summary of the chapter and also its conclusion, leading into the next chapter). The summary and conclusion of each chapter is given as below

#### <u>Summary and Conclusion for Chapter 2</u>

In this chapter, the fabrication of the EDZF is first analysed. The fabrication process is carried out by using the same processes in which conventional fibres and EDFs are fabricated, with only a minor change being made during the solution doping process before the developed porous layer is sintered, collapsed and drawn into a fibre.

The process begins with a hollow silica tube with good optical transparency, relatively high drawing temperatures and good mechanical strength and non- hygroscopic properties as the base material. The tube is cleaned and then made to undergo the MCVD process, where O<sub>2</sub>, N<sub>2</sub> and CCl<sub>2</sub>F<sub>2</sub> gasses are fed through SiCl<sub>4</sub>, GeCl4 & POCl<sub>3</sub> in a liquid form in the bubbler to generate SiO<sub>2</sub>, GeO<sub>2</sub> & P<sub>2</sub>O<sub>5</sub> gases. The deposition process is carried out using an oxy-hydrogen burner at a temperature of 1250-1300°C along the direction of the reactant flow containing the SiCl4, GeCl4, POCl3 vapors. A dopant control program was used to adjust the NA values to within the range of 0.15-0.25 with a core diameter of between 5.0-6.0 µm, with the relative density of the soot layer estimated on the basis of RE concentration ranging between 0.30 to 0.50. Once the porous soot layer has been deposited on its inner layer, the tube is then made to undergo the solution doping process. An alcoholic-water mixture at a ratio of 1:5 with a suitable strength was used to form ErCl<sub>3</sub>.6H<sub>2</sub>O, ZrOCl<sub>2</sub>.8H<sub>2</sub>O & AlCl<sub>3</sub>.6H<sub>2</sub>O, with the strength of the ErCl<sub>3</sub>.6H<sub>2</sub>O was adjusted between 0.005 M to 0.010 M, while the strength of the ZrOCl<sub>2</sub>.8H<sub>2</sub>O & AlCl<sub>3</sub>.6H<sub>2</sub>O complexes adjusted to between 0.1 M to 1.0 M. Small amounts of Y<sub>2</sub>O<sub>3</sub> and P<sub>2</sub>O<sub>5</sub> are also added to serve as nucleating agents. Subsequently, the doped tube is now oxidized at 800°C to 1000°C before being dehydrated using a careful combination of temperature, Cl<sub>2</sub>:O<sub>2</sub> ratio and selected time period, and finally undergoing a sintering process at a temperature of between 1400 to 1600°C. The completed tube is then collapsed at a temperature of above 2000°C to form the preform. Fibre drawing is carried out using a drawing tower, and the drawn fibre is measured using a diameter measuring device, and coated with Desolite DP-1004 resin as the primary coating as well as Desolite DS-2015 resin as the secondary coating.

Two fibres are drawn, ZEr-A and ZEr-B, with different dopant concentrations. Visual inspection of the lateral surfaces of the both fibres shows a smooth polymer layer, indicating that no bubbles or other deformities present in the polymer coating and a very distinct core with a diameter of approximately 10  $\mu$ m under microscopic view. The core region morphology of selected ZEr-A and ZEr-B preform is studied FEGSEM, and the ZEr-B fibre is seen to have micro-crystallite structures with better defined boundaries than the ZEr-A fibre, owing to its higher ZrO<sub>2</sub> ion concentrations. The presence of the ZrO<sub>2</sub> rich micro-crystallites is further confirmed by TEM scanning. XRD analysis of the preform samples shows a small diffraction peak is detected at a 20 value of about 30°, indicating the formation of tetragonal ZrO<sub>2</sub> in the host matrix. An EPMA analysis of the preform samples gives an almost equivalent level of Al<sub>2</sub>O<sub>3</sub> dopants in both fibres, at about 0.24-0.25 mole%. However, the ZEr-B preform has significantly higher ZrO<sub>2</sub> and Er<sub>2</sub>O<sub>3</sub> dopant concentrations, at approximately 2.21 mole% and 0.155 mole% respectively, as compared to concentrations of only 0.65 mole% and 0.225 mole% for the same dopants in the ZEr-A fibre.

Spectral characterization of the EDZF, carried out with the use of a Bentham spectral loss system, gives details on the absorption coefficient, fluorescence and fluorescence decay curves of the fabricated fibres. The ZEr-A fibre shows a peak at 980 nm with a loss of about 16.5 dB/m as well as a second peak at 1550 nm with a spectral attenuation of around 27.5 dB/m. Similarly, the ZEr-B fibre sample also shows two attenuation peaks at the same wavelengths, with attenuation rates of around 22.0 dB/m and 53.0 dB/m at 980 nm and 1550 nm respectively and also a peak at 800 nm, with spectral attenuations of 6.0 and 8.0 dB/m. Measured fluorescence life-times are 10.93 and 10.86 ms for the ZEr-A and ZEr-B fibres respectively. The slightly shorter lifetime of the ZEr-B fibre, which can be attributed to the higher dopant concentrations of that fibre, is crucial as it indicates that the concentration-quenching phenomenon typical associated with high Er³+ ions concentrations is strongly reduced in the presence of high ZrO<sub>2</sub> doping levels. Examination of the refractive index profile of the ZEr-B, as well as the ZEr-A fibres gives a standard W-profile that is to be expected of the optical fibre.

In the following chapter, the application of the EDZF as an active medium will be examined. Due to the ZEr-B's higher dopant concentration, all subsequent research will be carried out using this fibre. Of particular interest would be to use the EDZF as a gain medium for the development of a compact SLM fibre laser, as the high dopant concentrations in the EDZF will allow the cavity length of the proposed laser to be reduced significantly, a crucial criteria for the generation of the SLM output. The next chapter will also examine the possible application of the EDZF as a non-linear medium, as the incorporation of the ZrO<sub>2</sub> rich micro-crystalline structures in the host matrix will allow non-linear phenomena to manifest in light signals of relatively low intensity, whereas the same phenomena will only manifest itself in conventional SMFs at much higher intensities. This makes the EDZF a particularly useful candidate for the development of exotic all-optical components such as wavelength converters.

#### Summary and Conclusion for Chapter 3

In this chapter, the various optical properties of the EDZF are investigated. This chapter begins with a detailed examination into the theoretical background of the EDF. Subsequently, the rate equations that are used to model these transitions are discussed, followed by a derivation of the equations that govern the ASE, gain and noise figure of the EDFA.

The chapter examines the ASE characteristics of the EDZF. The work is carried out with a 3 m long EDZF, which has a dopant concentration in excess of 3880 ppm/wt with an absorption ratio of about 22.0 dB/m at 980 nm and approximately 53.0 dB/m at 1550 nm. A conventional EDF of the same length is also examined under the same conditions, and the resulting spectra compared. The conventional EDF used has an Er3+ ion concentration of about 960 ppm and absorption ratio of between 11 to 13 dBm at 980 nm. The spectral shape of the EDZF's ASE output differs substantially from that of a conventional EDF, rising to a peak region at 1530 nm followed by a short 'plateau' afterwards before dropping back again. At the highest pump power, a peak power of -18.6 dBm is obtained at the 1530 nm region, while the plateau region gives an average power of -26.0 dBm. This is a very important characteristic, as it sets the operational boundaries of the EDZF and gives it a longer operating bandwidth as compared to the EDF. This is attributed to the high Er<sup>3+</sup> dopant concentrations in the fibre, which essentially allows the EDZF to act as an L-band amplifier. Additionally, the 1530 nm peak becomes more dominant at higher pump powers, recording peak powers of -24.9, -20.4 and -17.7 dBm at pump powers of 108.7, 140.0 and 170.1 mW while the plateau region remains rises at a much slower rate, increasing from an average of -30.0 dBm to -28.0 dBm over the range of the same pump powers. This is further confirmed by comparing the ASE from a 3 m long EDZF to that of a 2 m long EDZF that is pumped under the same conditions. Observation of the spectra obtained indicates that the shorter EDZF acts more like a conventional EDF, instead of its expected behavior when observing longer EDZF lengths.

As a gain medium, the EDZF exhibits characteristics that are typical of a highly doped EDF. For a -30 dBm signal, the EDZF-based EDFA is able to provide high gain of approximately 28.0 dB near the central region of 1530 nm in addition to a substantial gain of between 22.0 to 25.0 dB at the plateau region of the EDZF's ASE output. A relatively high NF is also observed, approximately 12.2 dB at 1530 nm before decreasing to around 2.8 dB at the 1550 nm wavelength region. At longer wavelengths though, the NF increases again, reaching 3.9 dB at 1590 nm. A 0 dBm input signal on the other hand experiences lower gains, averaging between 7.0 to 10.0 dB over a range of 1530 nm to 1570 nm and lower gains of 2.0 to 3.0 dB at longer wavelengths. In this configuration, the EDFA acts instead as a booster amplifier. The NF of experienced by the signal in this scenario is also higher, at approximately 14.1 dB before dropping to 7.7 dB at 1580 nm and subsequently rising slightly to 8.3 dB at 1590 nm.

As a fibre laser, the ability of the EDZF to act as the gain medium for an SLM laser is examined. This is because the high dopant concentrations in the fibre allow for a short cavity length to be realized, a necessary requirement for the SLM, while at the same time

being highly compatible with conventional optical fibres due to the fact that the EDZF is based on a conventional silica host. As an SLM fibre laser, a 0.5 m long EDZF with a dopant concentration of 3880 ppm/wt and absorption rate of about 21.0 dB/m at 980 nm serves as the gain medium. Due to the high dopant concentrations, the 0.5 m EDZF can provide the same performance as an EDF approximately 4 m long, thus significantly reducing the cavity length. Two short EDFs, approximately 3 cm and 6 cm long are also inserted in the cavity to act as saturable absorbers. The EDFs used are conventional EDFs, with an erbium ion concentration of about 900 ppm/wt and absorption rates of 5.0 dB/m at 1530 nm. A tunable C-band FBG provides the tuning mechanism. The SLM output of the laser has a tunable range of 11.2 nm, from 1533.8 nm to 1545.0 nm, as well as an average SNR of more than 50 dB with an output power of above -8.9 dBm. The output of the system is measured over a period of two and a half hours in 10-minute intervals, and during this period almost no fluctuations are observed in either the power or wavelength of the laser's output. Measurement with an RF-SA provides positive indication that the oscillation is in the single mode as the beating noise peaks observed in the trace are densely spaced, while measurements of the linewidth provide a value of about 0.2 MHz for the SLM output.

Another interesting characteristic of the EDZF is its non-linear properties. Although non-linear interactions in silica optical fibres are typically observed only when high intensity signals propagate through them, the presence of the  $ZrO_2$  micro-crystallites in the core region of the EDZF allow these interactions to occur at lower signal intensities. The most predominant, and useful non-linear effect that can be generated by the EDZF is the FWM effect, due to its multiple uses as wavelength sources and also in wavelength conversion. In investigating the FWM effect, a 4 m long EDZF is used to determine the non-linear characteristics of the fabricated fibre. FWM power levels of approximately -45 dBm are obtained at a wavelength of around 1565 nm, auguring well with the theoretical predicted values. Adjusting the pump and single wavelengths will also cause the idlers to shift in accordance with the theoretically predicted models. The EDZF has a non-linear coefficient of 14 W-1km-1 along with chromatic and dispersion slopes of 28.45 ps/nm.km and 3.63 ps/nm2.km that are well in accordance to the theoretical values.

It can be concluded that the EDZF does indeed succeed in meeting one of its main objectives, which is to develop an EDF with a high-dopant concentration without sacrificing the compatibility and strength of the SMF. The high dopant concentration of the EDZF allow for high gain levels to be obtained from compact EDFAs and also plays an important role in the development of SLM based fibre lasers, whereby the cavity length should be as short as possible. In both cases, the benefit of the silica host is evident - the fibre amplifiers and lasers are easily setup using conventional fibre components, thus demonstrating the real-world application prospects for the EDZF. The presence of the Zr³+ rich microcrystalline structures in the matrix of the silica host also allows for interesting non-linear phenomena to be observed, in particular the FWM effect, which has tremendous practical applications such as wavelength conversion.

The application of the EDZF as the gain medium for passive fast and ultra-fast pulsed lasers is of significant importance and is a key objective in this work. This is described in detail in the next chapter.

#### Summary and Conclusion for Chapter 4

In generating the fast and ultrafast pulses, the EDZF is configured to as a fibre laser working in conjunction with passive saturable absorbers formed from graphene or SWCNTs.

The formation of the saturable absorbers is described, with both saturable absorbers being formed by different means. The graphene based saturable absorber is formed by the optical deposition technique, using graphene flakes are obtained from Graphene Research Ltd suspended in an NMP solution. The flakes have an average particle size of 550 nm and average flake thickness of 0.35 nm. Taking advantage of the optical thermophoresis effect, the graphene layer is formed by first dipping a fibre ferrule in the aqueous solution containing the graphene flakes, before connecting it to another fibre ferrule and exposing the ferrules to an intense laser signal. After a period of time, the ferrules are disconnected and one cleaned, before being reconnected and the process repeated. The graphene based saturable absorber can be assumed to be formed once a reflected power of about 4.1% of the transmitted power is obtained, with the graphene layer reflecting approximately 0.1% of the signal and Fresnel reflection accounting for the remaining 4.0% of the reflected signal. Examination of the deposited graphene layer with a Renishaw InVia Raman spectrometer at 532 nm (2.33 eV) over a period of 10s with a grating value of 1800 lines/mm gives two intensity peaks at 1597 cm<sup>-1</sup> and 2684 cm<sup>-1</sup>. These peaks confirm the presence of the graphene layer, with the peak at 1597 cm<sup>-1</sup> corresponding closely to the expected 1580 cm<sup>-1</sup> G peak for graphene, while the peak at 2684 cm<sup>-1</sup> corresponds closely to the 2700 cm<sup>-1</sup> 2D peak for graphene. Furthermore, the ratio of G to 2D does not exceed 1 indicating that a nearly single layer of graphene has been obtained on the fibre ferrule, with the actual number of layers being probably 2 or 3.

The SWCNT saturbale absorber on the other hand is more easily fabricated and can be accomplished by simply hosting the nanotubes in a suitable host material. Typically, PVA is the preferred choice for a host material but in this research a new host material, PEO, is used to form the SWCNT saturable absorber. PEO has a number of advantages over PVA, such as a lower melting point and also easy dispersion in water, making the fabrication process easier while at the same time having no adverse effects on the performance of the SWCNTs as saturable absorbers. The saturable absorber itself is formed by sandwiching a SWCNT/PEO composite in between two fibre ferrules. The SWCNTs used are 99% pure with lengths of between 3 to 30 µm and average diameters of between 1 to 2 nm and are procured from Cheap Tubes Inc. The PEO solution has an average molecular weight of 1 x 106 g/mol while the SDS solution that is used to disperse the SWCNTs has an average molecular weight of 288.38 g/mol. The SWCNT is soaked in a 1% SDS solution before being ultrasonically dispersed at 50 W for a period of 30 minutes and is then mixed with the PEO before the entire solution is simply dropped onto the face of the fibre ferrule and allowing it to dry in air for 24 hours. Analysis by Raman spectroscopy shows G, D, and G' peaks at 1598 cm<sup>-1</sup>, 1362 cm<sup>-1</sup> and 2684 cm<sup>-1</sup>, indicating

the presence of the SWCNTs in the saturable absorber. Two RBM peaks at 186 cm<sup>-1</sup> and 287 cm<sup>-1</sup> are also observed.

As a Q-switched laser capable of generating fast pulses, a 3 m long EDZF lasers with an active ion concentration of approximately 3880 ppm/wt is used as the gain medium and the SWCNT/PEO composite saturable absorber as the Q-switching mechanism. The Q-switched pulse train is obtained above a threshold pump power of 92.5 to 95.0 mW, and at the maximum pump power of 141.8 mW a repetition rate of 14.20 kHz is obtained, with a corresponding pulse width of 8.6  $\mu$ s. The average output power of the fibre laser is 270.0  $\mu$ W at 141.8 mW, and the maximum pulse energy obtained is 19.02 nJ. Using the graphene based saturable absorber on the other hand gives Q-switched pulses with a pulse width of 4.6  $\mu$ s and a pulse energy and pulse peak power of 16.8 nJ and 3.6 mW respectively. The repetition rate of the Q-switched pulses is approximately 50.1 kHz, with a lasing threshold of 48 mW, and a Q-switching threshold of 56 mW.

When operating in the mode-locking regime using the graphene based saturable absorber, the EDZF fibre laser has a mode-locking threshold of 90 mW, with the generated pulses having an average output power, pulse energy and peak power of approximately 1.6 mW, 23.1 pJ and 31.6 W respectively as well as a pulse width of 730 fs. The repetition rate of the pulses is 69.3 MHz, corresponding to a channel spacing of 14.5 ns in the pulse train with a central wavelength of about 1563.0 nm. Replacing the graphene based saturable absorber with one formed from a SWCNT/PEO composite now allows the EDZF based mode-locked laser to generate pulses with a central wavelength of 1562.7 nm, although the obtained spectrum indicates the presence of some perturbations in the midpoint region. The mode-locked laser has an average output power of 180 µW, with a repetition rate and peak power of 17.74 MHz and 14.09 W respectively as well as a pulse energy of 0.01 nJ. The estimated pulse duration is approximately 720 fs at the FWHM point, with a time-bandwidth product of 0.48. Examination of the pulses using an RFSA confirms the repetition rate of the pulses to be 17.74 MHz, with an estimated peak-to-pedestal ratio of 35 dB. In all cases, the generated pulses are stable and consistent, and allow them to be deployed with a high degree of confidence and reliability in multiple practical applications.

#### 2. I have marked editorial corrections in several places.

I have made the corrections at the following points. These are higlighted in red in the main text as follows:

- Pg. 23, Line 27: 'Hockman' changed to 'Hockham'
- Pg. 24, Line 3: Added the word 'for' such that the sentence now reads '...2009 Nobel prize for groundbreaking achievement..'
- Pg. 24, Line 7: Changed the word 'was' to 'were' so that the sentence now reads '...more attention and resources were focused on optical...'

- Pg. 24, Line 9: Changed '1500' to '1550' so that the sentence now reads '...in the 1550 nm region...'
- Pg. 25, Line 6: The word 3R has been added so that the sentence now reads '...Amplified, Reshaped and Retimed (3R)...'
- Pg. 25, Line 11: The sentence has been modified so that it now reads 'However, 3R repeaters were bit rate sensitive, and as such needed to be replaced as and when transmission capacities needed to be boosted. This problem was further complicated with...'
- Pg. 25, Line 33: The sentence has been modified so that it now reads '...the third low-loss window of communications ...'
- Pg. 25, Line 31: The sentence has been modified so that it now reads '...by D. N. Payne and colleagues in 1987..'
- Pg. 26, Line 1: The sentence has been modified so that it now reads '...that are of relatively low-cost...'
- Pg. 26, Line 4: The sentence has been modified so that it now reads '...the EDFA has been...'
- Pg. 26, Line 7: The sentence has been modified so that it now reads '...spans of 100 to 200 km...'
- Pg. 27, Line 4: The sentence has been modified so that it now reads '...which require as short as ...'
- Pg. 27, Line 23: The sentence has been modified so that it now reads '...retaining a compact form-factor.'
- Pg. 29, Line 7: The sentence has been modified so that it now reads '...turn requires as short as possible...'
- Pg. 29, Line 7 to Line 11: The sentence has been divided into two shorter sentences so that it now reads 'Achieving this in turn requires as short as possible laser cavity, and the EDZF would thus be a highly suitable candidate for reducing the size of the laser cavity while at the same time allowing the other components of the fibre laser to be commercially procured. This keeps the cost of the laser low while still allowing it to operate at its optimum capacity.'

- Pg. 29, Line 28: The sentence has been modified so that it now reads '...typically Q-switched or mode-locked...'
- Pg. 32, Line 21: The sentence has been modified so that it now reads 'The obtained tube is then...'
- Pg. 34, Line 11: The sentence has been modified so that it now reads '...based on soft glass hosts...'
- Pg. 34, Line 16: The sentence has been modified so that it now reads '...incompatibility in splicing for example with...'
- Pg. 35, Line 4: The sentence has been modified so that it now reads '...during the fabrication of this...'
- Pg. 35, Line 17: The sentence has been modified so that it now reads '...prepare a porous glass host.'
- Pg. 35, Line 26 and 27: The sentence has been modified so that it now reads '...and preserve the pristine strength of the fibre.'
- Pg. 36, Line 7: The sentence has been modified so that it now reads '...as it has a number...'
- Pg. 36, Line 10: The sentence has been modified so that it now reads '...approximately 1550 nm.'
- Pg. 38, Line 1: The sentence has been modified so that it now reads '...result of the formation...'
- Pg. 40, Line 4: The sentence has been modified so that it now reads '...allowing it easier drawing [47].'
- Pg. 40, Line 31: The sentence has been modified so that it now reads '...for the Si4+ ions...'
- Pg. 42, Line 6: The sentence has been modified so that it now reads '...the host tube has been completed, the tube is cleaned...'
- Pg. 42, Line 25: The sentence has been modified so that it now reads '...above 1450°C to produce...'

- Pg. 43, Line 6: The sentence has been modified so that it now reads '...same time maintaining...'
- Pg. 52, Line 8: The sentence has been modified so that it now reads '...drawn into a fibre.'
- Pg. 53, Line 1: The sentence has been modified so that it now reads '...coated on-line with...'
- Pg. 63, Line 20: The sentence has been modified so that it now reads '...as well as the spectral...'
- Pg. 67: Equation 8 has been modified so that it now reads ' $NA = \sqrt{(n_1^2 n_2^2)}$  ......(.1)'
- Pg. 68, Line 5: The sentence has been modified so that it now reads '...the standard W-profile.'
- Pg. 71, Line 2 to Line 3: The sentence has been modified so that it now reads '...EDZF, we would like to reiterate that...'
- Pg. 71, Line 3: The sentence has been modified so that it now reads '...EDZF was to develop...'
- Pg. 71, Line 19: The sentence has been modified so that it now reads '...the late 1980s however...'
- Pg. 71, Line 20: The sentence has been modified so that it now reads '...are broadband optical amplifiers that allow a...'
- Pg. 71, Line 7 to Line 12: The sentence has been modified so that it now reads '...wherefrom these excited ions quickly relax to the lower 9l11/2 level where they stay for about 10 ms. This intermediate level forms a metastable state, and the energy difference between the 4l31/2 and the ground level of 4l51/2 correspond to a wavelength of 1550 nm. Due to the Stark effect, each energy level is split into multiple levels, and thus a band of signals around 1550 nm can stimulate a broadband emission while the excited ions fall back to the ground state.'
- Pg. 71, Line 19: The sentence has been modified so that it now reads '...divided into two...'
- Pg. 71, Line 27: The sentence has been modified so that it now reads '...and therefore exists...'

- Pg. 74, Line 3: The sentence has been modified so that it now reads '...active is due to...'
- Pg. 77, Line 6: The sentence has been modified so that it now reads '...transitions are possible...'
- Pg. 80, Line 11: The sentence has been modified so that it now reads '...be essentially represented...'
- Pg. 81, Line 12: The sentence has been modified so that it now reads '...the excited and...'
- Pg. 84, Line 4: The sentence has been modified so that it now reads '...occurs when an ...'
- Pg. 84, Line 18: The sentence has been modified so that it now reads '...power is a ...'
- Pg. 88, Line 2: The sentence has been modified so that it now reads '... as  $N_1$  and...'
- Pg. 91, Line 16: The sentence has been modified so that it now reads '...be first measured,'
- Pg. 93, Line 5: The sentence has been modified so that it now reads '...977 nm and...'
- Pg. 96, Line 5: The sentence has been modified so that it now reads '...that it allows...'
- Pg. 99, Line 14: The sentence has been modified so that it now reads '...which in turn ...'
- Pg. 102, Line 17: The sentence has been modified so that it now reads '...plateau region rises at...'
- Pg. 107, Line 17: The sentence has been modified so that it now reads '...external signal source...'
- Pg. 119, Line 1: The sentence has been modified so that it now reads '...many important applications ...'
- Pg. 119, Line 4: The sentence has been modified so that it now reads '...that it makes ...'

- Pg. 119, Line 20: The sentence has been modified so that it now reads '...with the phase delay...'
- Pg. 119, Line 21: The sentence has been modified so that it now reads '...can also be ...'
- Pg. 121, Line 17 to Line 19: The sentence has been modified so that it now reads 'The pump frequencies can be represented by  $\omega_1$  and  $\omega_2$  respectively, while the frequencies of the generated signals and idlers are designated as  $\omega_3$  and  $\omega_4$  respectively.'
- Pg. 121, Line 23 to Line 24: The sentence has been modified so that it now reads 'For degenerate FWM,  $\omega_1 = \omega_2$ .'
- Pg. 122, Line 11: The sentence has been modified so that it now reads 'The signal and idler frequencies...'
- Pg. 122, Line 14: The sentence has been modified so that it now reads '...encounter degenerate FWM ...'
- Pg. 123, Line 9: The sentence has been modified so that it now reads '...signals are propagating.'
- Pg. 127, Line 24: The sentence has been modified so that it now reads '...dispersion slope...'
- Pg. 131, Line 3 to Line 4: The sentence has been modified so that it now reads '...developed EDZFs can be...'
- Pg. 139, Line 13: The sentence has been modified so that it now reads '...high power laser...'
- Pg. 141, Line 17: The sentence has been modified so that it now reads '...demonstrated by Ippen...'
- Pg. 141, Line 8: The sentence has been modified so that it now reads '...oscillate independently in...'
- Pg. 147, Line 20: The sentence has been modified so that it now reads '...absorber, and hence creating...'
- Pg. 149, Line 21: The sentence has been modified so that it now reads '...using by...'
- Pg. 151, Line 1: The sentence has been modified so that it now reads '...conduction...'

- Pg. 158, Line 8: The sentence has been modified so that it now reads '...graphene, both...'
- Pg. 159, Line 11: The sentence has been modified so that it now reads '...spectroscopy to validate...'
- Pg. 169, Line 8: The sentence has been modified so that it now reads '...extrapolating...'
- Pg. 175, Line 58: The sentence has been modified so that it now reads '...seen that due...'
- 3. Normally one expects reduction in FWM efficiency in a fiber with increase in channel spacing (see e.g. Tynge Li, "Impact of amplifiers on long distance lightwave telecommunications," Proc IEEE vol 81, pp. 1568-1579 (1995) contrary non-dependence of FWM efficiency observed by the author here for variation in ch spacing from 30 GHz to 400 GHz as discussed on p. 129! Similar conclusions have been drawn in respect of Fig. 62 containing plot of nonlinear parameter 

  with channel spacing. During the viva voce this may be clarified or possible explanations given by the candidate.

Due to the nature of the fibre used in this work, it is believed that the higher conversion efficiency is a result of the channel spacing being narrow, thus allowing more energy to be transferred to the idlers generated. As the channel spacing increases, it can be seen that the conversion efficiency decreases as well, thus behaving as would be expected for the FWM effect.

This is given in Line 7 to 9 of Page 129 as follows:

'The initial high FWM efficiency can be attributed to the channel spacing being narrow, thus allowing for a larger transfer of energy from the pump and signal wavelengths to the generated idlers.'

4. On p.141 inversion factor is denoted by a parameter  $\gamma$ , which should be changed to another parameter as it clashes with nonlinearity parameter used earlier. Likewise parameter  $\psi$  used on p. 144 was used earlier!! On P. 145  $\Delta v$  should it be changed to  $\Delta f$ ? On. P. 150 parameter  $\gamma$  is again used to denote self-amplitude modulation coefficient!

I apologize for the oversight. The symbols have now been changed so as to not overlap each other, with the changes as follows:

γ replaced with Γ

- $\psi$  replaced with  $\Psi$
- Δv replaced with Δf

The changes made are highlighted in red.

#### **External Examiner 2 (Prof. Dr. Ajoy K. Kar, Herriot-Watt)**

#### (All corrections of Prof. Dr. Ajoy K. Kar are highlighted in purple in the corrected thesis)

 Abstract should be self-contained. The first sentence in the second paragraph should be deleted. The last paragraph does not make any sense and should be omitted. In addition the abstract describes many facts in great detail. it should be rewritten describing only the key results. Finally, acronyms must not be used in the abstract

The abstract has been rewritten as per the recommendation of the external examiner, with the notable changes being the removal of the first sentence of the second paragraph and also the last paragraph. The acronyms have also been removes, and the abstract made more concise. These changes were made for both the English and Bahasa Malaysia versions of the abstract.

#### Abstract (In English)

In this research, the fabrication and characterization of zirconia-yttria-alumino silicate glass-based erbium-doped fibres as well as their application as the gain media for compact fast and ultra-fast pulsed sources is presented.

The fabrication process is similar to that of a conventional erbium doped fibre, with the ZrO2 co-dopants incorporated into the silica host during solution doping. Morphological studies of the drawn fibres reveal a core with a diameter of approximately 10  $\mu$ m. Tunneling electron microscope scanning shows the presence of ZrO2 rich microcrystallites, while X-ray diffraction analysis indicates the formation of tetragonal ZrO2 structures. Spectral characterization of the fibres show attenuation peaks at 980 nm and 1550 nm, with sample designated ZEr-B having absorption rates of 22.0 dB/m and 53.0 dB/m at 980 and 1550 nm respectively as well as a fluorescence life-time of 10.86 ms, as well as a W-profile refractive index.

A 3 m long ZEr-B fiber with a dopant concentration of about 3880 ppm/wt is used to generate an amplified spontaneous emission spectrum. The fibres amplified spontaneous emission spectrum output differs substantially from that of a conventional erbium doped fibre of the same length, rising to a peak region at 1530 nm, followed by a short 'plateau' before decreasing. As a fibre amplifier a gain of around 28.0 dB near 1530 nm and a relatively flat gain of between 22.0 to 25.0 dB at the plateau region is obtained, together with a noise figure of approximately 14.1 dB for an input signal of -30 dBm. The fiber can also generate a single-longitudinal mode output, ranging from 1533.8 nm to 1545.0 nm at output powers of more than -8.9 dBm with an average signal-to-noise ratio of more than 50 dB. Additionally, the fiber allows non-linear interactions to occur at lower signal intensities than normal, with a four-wave-mixing output adhering to theoretically predicted models. The average four-wave-mixing power level is -45 dBm at approximately 1565 nm, with a non-linear coefficient of 14 W-1km-1 with chromatic and dispersion slopes of 28.45 ps/nm.km and 3.63 ps/nm2.km.

The erbium-doped zirconia fiber is also used in conjunction with gaphene and singlewalled carbon nanotubes based passive saturable absorbers to generate fast and ultrafast pulses is examined. Using a 3 m long ZEr-B with single-walled carbon nanotubes suspended in a polymer host generates Q-switched pulses with a repetition rate of 14.20 kHz and corresponding pulse width of 8.6 μs at a maximum pump power of 141.8 mW, as well as an average pulses output power of 270.0 µW and maximum pulse energy of 19.02 nJ. Using the graphene based saturable abosrber gives a 50.1 kHz pulse train with a pulse width, energy and peak power of 4.6 µs, 16.8 nJ and 3.6 mW respectively. When mode-locked, the ZEr-B combined with the graphene based saturable absorber generates ultrafast pulses with an average output power, pulse energy and peak power of approximately 1.6 mW, 23.1 pJ and 31.6 W respectively as well as a pulse width of 730 fs and repetition rate of the pulses is 69.3 MHz. Using the single-walled carbon nanotubes composite as a saturable absorber gives mode-locked pulses with a repetition rate and peak power of 17.74 MHz and 14.09 W as well as average output power of 180 µW and pulse duration of approximately 720 fs at the full-width at half maximum point, with a pulse energy of 0.01 nJ. The generated pulses are stable and consistent, and allow them to be deployed with a high degree of confidence and reliability in multiple practical applications.

#### Abstract (In Bahasa Malaysia)

Di dalam penyelidikan ini, pembuatan dan pencirian gentian berdopan erbium berasaskan gelas zirconia-yttria-alumino silicate serta aplikasinya sebagai bahan aktif bagi sumber berdenyut cepat dan ultra-cepat yang padat dibentangkan.

Proses pembuatan gentian ini adalah hampir sama seperti gentian berdopan erbium yang lazim, dengan penggabungan dopan ZrO2 ke dalam gentian silica semasa process pendopan cecair. Analisa morfologi bagi gentian yang ditarik menunjukkan bahagian teras dengan diameter sebanyak 10 µm. Analisa 'Tunneling Electron Microscope Scanning' menunjukkan kehadiran struktur kristal mikro yang kaya dalam ZrO2 manakala analisa penuraian X-Ray mengesahkan pembentukkan struktur ZrO2 berbentuk tetragon. Pencirian spectra gentian yang ditarik menujukkan puncak pelemahan pada 980 nm dan 1550 nm, dengan sampel ZEr-B mempunyai kadar penyerapan sebanyak 22.0 dB/m dan 53.0 dB/m pada 980 dan 1550 nm serta jangkamasa pendaflour selama 10.86 ms dan indeks biasan berprofil-W yang biasa.

Gentian ZEr-B sepanjang 3 m dengan ketumpatan dopan sebanyak 3880 ppm/wt digunakan untuk menghasilkan spektra amplified spontaneous emission. Spektra yang diperolehi daripada adalah amat berbeza daripada gentian berdopan erbium biasa, dengan puncak pada 1530 nm disertai bahagian datar yang pendek sebelum berkurang. Apabila digunakan sebagai pembesar optik, gentian EDZF berkebolehan untuk memberi penambahan sebanyak 28.0 dB di rantau 1530 nm, dengan penambahan yang agak malar di antara 22.0 hingga 25.0 dB di rantau mendatar, serta nilai hingar sebanyak 14.1 dB bagi isyarat masuk setinggi -30 dBm. ZEr-B juga dapat menghasilkan output jenis singlelongitudinal mode daripada 1533.8 nm hingga 1545.0 nm diperolehi pada kuasa -8.9 dBm

dan nisbah isyarat kepada hingar yang lebih daripada 50 dB. Di samping itu, ZEr-B juga membolehkan interaksi tidak linear berlaku pada kuasa isyarat yang rendah, dengan penjanaan. fenomena four-wave-mixing seperti yang dijangkakan dalam teori. Kuasa purata four-wave-mixing adalah sebanyak -45 dBm pada 1565 nm, dengan nilai pemalar tidak linear yang sebanyak 14 W-1km-1 serta cerun kromatik dan penguraian sebanyak 28.45 ps/nm.km dan 3.63 ps/nm2.km..

ZEr-B juga digunakan bersama graphene dan single-walled carbon nanotubes sebagai saturable absorber yang pasif bagi menjana denyutan yang cepat dan ultra-cepat. Menggunakan ZEr-B sepanjang 3 m dan single-walled carbon nanotubes yang diampaikan dalam polimer dapat menghasilkan denyutan Q-switched dengan kadar pengulangan 14.20 kHz serta lebar denyutan sepanjang 8.6 µs pada kuasa pam makisma sebanyak 141.8 mW, pada kuasa purata sebanyak 270.0 µW, dengan tenaga denyutan sebanyak 19.02 nJ. Penggunaan saturable absorber berasaskan graphene memberikan kadar denyutan sebanyak 50.1 kHz serta lebar denyutan, tenaga denyutan dan kuasa puncak denyutan sebanyak 4.6 µs, 16.8 nJ dan 3.6 mW. Apabila mode-locked, ZEr-B bersama saturable absorber berasaskan graphene memberikan kuasa output purata, tenaga denyutan dan kuasa puncak sebanyak 1.6 mW, 23.1 pJ dan 31.6 W serta lebar denyutan sebanyak 730 fs and pada kadar denyutan 69.3 MHz. Menggunakan saturable absorber jenis single-walled carbon nanotubes memberikan denyutan mode-locked dengan kadar denyutan dan kuasa puncak sebanyak 17.74 MHz dan 14.09 W pada kuasa purata sebanyak 180 μW dan masa denyutan 720 fs pada full-width at half maximum serta tenaga denyutan sebanyak 0.01 nJ. Denyutan yang dihasilkan adalah stabil dengan kuasa yang malar, dan ini membolehkannya digunakan dengan kadar keyakinan yang tinggi serta dapat diharapkan dalam pelbagai aplikasi yang praktik.

2. As the fibres were drawn and characterized at CGCRI Kolkata, during the viva the candidate must clarify his contributions in the Chapter 2. it must be clarified the contributions of CGCRI, whether the candidate was directly involved with the experiments.

Yes, the examiner is right – a major portion of the fabrication process was carried out at the CGCRI, Kolkatta, India by our collaborator (and my co-supervisor) Dr. Mukul. C. Paul. This was done as the University of Malaya lacked certain critical equipment that was required for the fabrication of the fibre. However, the testing and characterization work was carried out mainly at the University of Malaya as the necessary equipment was available.

This fact is highlighted in Line 13 to 15 of Page 35, as follows:

'Due to the availability of facilities, the fabrication of the fibers as well as certain characterization tests were carried out at the CGCRI, and some tests being carried out at the University of Malaya..'

3. The candidate is a single author only in one paper. He should also clarify his contributions in various other papers.

This has been clarified during the voice-viva.

4. I do not understand in the meaning of the publications resulting 'directly or indirectly' from the results presented in the thesis as mentioned in page 209. The direct and indirect aspects of the publications also have to be clarified during the viva.

I apologize for the words being used, as they have raised quite some confusion. In fact, I was involved directly in most of the papers. I have also added a list of conference proceedings which is also given in Appendix 2.

I have rephrased the sentence in Appendix 2 to avoid this confusion, and have also clarified this during the voice-viva.

The following are the publications that have resulted from this work.

#### **Book Chapters**

 Chapter 2: Nano-Engineered Glass-Based Erbium Doped Optical Fibers for Study of Multi-Channel Amplification and Four-Wave Mixing Phenomena, M. C. Paul, M. Pal, K. Thambiratnam, S. W. Harun, N. A. Awang, S. Das, S. K. Badhra, H. Ahmad and J. K. Sahu. Rare Earths: New Research (Chemistry Research and Applications: Materials Science and Technologies) (Zhaosen Liu (Editor)) Nova Science Publishers Inc. (1 July 2013, Hauppauge, New York, No. of pages: 289, ISBN-10: 162618996X).

#### **Publications in ISI Journals**

- 1. H. Ahmad, K. Thambiratnam, N. A. Awang, M. H. Jemangin, and S. W. Harun, "Stable Zirconia–Erbium Doped Multiwavelength Fibre Laser by Precise Control of Polarization States," *Las. Phys. Lett.*, vol. 22, pp. 982-985, 2012.
- 2. H. Ahmad, M. C. Paul, N. A. Awang, S. W. Harun, M. Pal and K. Thambiratnam, "Four-Wave-Mixing in Zirconia-Yttria-Aluminum Erbium," *J. Europ. Opt. Soc. Rap. Public.*, vol. 7, pp. 12011-1 12011-8, 2012.
- 3. H. Ahmad, N. A. Awang, M. Z. Zulkifli, K. Thambiratnam, M.C. Paul, S. Das, and S.W. Harun, "Supercontinuum from Zr-EDF using Zr-EDF mode-locked fibre laser," *Las. Phys. Lett.*, vol. 9, pp. 44-49, 2012.
- 4. H. Ahmad, K. Thambiratnam, N. A. Awang, Z. A. Ghani and S.W. Harun, "Four-wave mixing in zirconia-erbium doped fibre a comparison between ring and linear cavities," *Las. Phys. Lett.*, vol. 9, pp. 819-825, 2012.

- 5. H. Ahmad, K. Thambiratnam, M. C. Paul, A. Z. Zulkifli, Z. A. Ghani, and S. W. Harun, "Fabrication and application of zirconia-erbium doped fibres," *Opt. Mat. Expr.*, vol. 2, pp. 1690-1701, 2012.
- 6. H. Ahmad, A.Z. Zulkifli, K. Thambiratnam and S.W. Harun "Q-switched Zr-EDF laser using single-walled CNT/PEO polymer composite as a saturable absorber," *Opt. Mater.*, vol. 35, pp. 347-352, 2013.
- 7. K. Thambiratnam, H. Ahmad, , F. D. Muhammad, M. Z. Zulkifli, A. Z. Zulkifli, M. C. Paul and S. W. Harun, "Q-Switching and Mode-Locking in Highly-Doped Zr<sub>2</sub>O<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub>-Er<sub>2</sub>O<sub>3</sub> Doped Fibre Lasers using Graphene as a Saturable Absorber," *IEEE J. Select. Topics in Quant. Electron.*, vol. 20, pp. 1100108, 2013.

### **Proceedings / Presentations in National and International Conferences**

- H. Ahmad, K. Thambiratnam and S. W. Harun, Fabrication of Nano-Engineered Zirconia Yttria Aluminosilicate Glass Co-doped with Erbium and its Applications, 9th International Symposium on Modern Optics and Its Applications (ISMOA 2013), 24 Jun 2013 to 27 Jun 2013, Indonesian Optical Society (InOs) Physics of Magnetism and Photonics Group Institut Teknologi Bandung (International)
- 2. A. Zarif, H. Ahmad, K. Thambiratnam, M. C. Paul and S. W. Harun, Four-wave Mixing Effect in Zirconia-erbium Doped Fibers, International Conference on Materials for Advanced Technologies (ICMAT2013), 30 June 2013 to 5 July 2013, Materials Research Society (MRS) Singapore (International).
- 3. A. Z. Zulkifli, K. Thambiratnam, F. Ahmad, H. Ahmad and S. W. Harun, Single Walled CNT / PEO based Saturable Absorber with Zr-EDF for Passively Q- Switched Pulse Laser Generation, 7th Asia-Oceania Top University League on Engineering (AOTULE 2012) Student Conference, 24th to 25th November 2012, University of Malaya, Kuala Lumpur, Malaysia (Local).

## Internal Examiner (Dr. Rozalina Zakaria, Physics Dept.)

No major issues raised as in the examiner report.