

# Emitting 1530 nm light from mixture of Er<sub>2</sub>O<sub>3</sub>, P<sub>2</sub>O<sub>5</sub> nanoparticles and spin-on-glass on silicon

Kuo-Jui Sun<sup>a</sup>, Yi-Shin Su<sup>a</sup>, and Ching-Fuh Lin<sup>\*ab</sup>

<sup>a</sup>Graduate institute of Electro-Optical Engineering, National Taiwan University,  
No.1, Sec. 4, Roosevelt Road, Taipei, 106, Taiwan, R.O.C

<sup>b</sup>Graduate institute of Electronics Engineering, and Dept. of Electrical Engineering

## ABSTRACT

We introduce a new fabricating technique that forms a light emitting layer on silicon wafer. This emitting layer could emit light at 1530nm. We mix the Er<sub>2</sub>O<sub>3</sub> nanoparticles with spin-on glass, then deposit this mixed solution onto silicon wafer. It emits 1530nm light because Er<sub>2</sub>O<sub>3</sub> nanoparticles release Er<sup>3+</sup> into glass at high temperature. The manufacturing process is very simple and low cost. We also add P<sub>2</sub>O<sub>5</sub>, Si, Al, and Ag nanoparticles in order to modify the physical characteristics and light emission efficiency.

**Keywords:** Erbium-doped optical fiber amplifiers, Er<sub>2</sub>O<sub>3</sub> nano-particles, nanotechnology, P<sub>2</sub>O<sub>5</sub> nano-particles, rare earth compounds, optical communications, optical interconnect.

## 1. INTRODUCTION

### 1.1 Erbium-doped fiber amplifier

The amount of data transmission continues to grow, so the advantage of high transmission capability of optical fiber is of great worth. In order to transmit signals over long distances, the first generation of optical systems used periodic electrical repeaters which detected, regenerated and re-transmitted the optical channel. The link was thus composed of small optical transmission segments of several tens of km length. The main constraint in terms of cost, capacity and flexibility was therefore due to the electrical regenerators. Indeed, if such regenerators could process signals with relatively high modulation speed (100 MHz–10 GHz), their cost dramatically increases with the signal bit-rate. In addition, the bit-rate, the modulation format and the data protocol are fixed for the whole system lifetime (e.g., 15–25 years). In-line regeneration by optical amplification was introduced to avoid such expensive electrical regeneration. Optical amplification also brought the potential of regenerating several WDM channels at once. Thus, the Er-doped optical fiber amplifier becomes the core technique of modern optical communication systems [1-4].

### 1.2 Our novel approach

However, Er-doped optical fiber has several disadvantages. For example, it requires a quite long distance to acquire optical gain and has expensive fabricating cost. In addition, it is difficult to monolithically integrate with electronics. Thus, we introduce a new manufacturing technique to create a light emitting layer on Si wafer. It emits light at 1530 nm. The fabrication process is very simple and with very low cost, compared with ion implantation technique. Ordinary erbium-doped optical fiber requires a quite long distance to acquire significant light emission from Er<sup>3+</sup> ions. Relatively, our emitting layer is able to show the signals only within 0.5μm due to the higher concentration of Er<sup>3+</sup> and surface effect of nano-particles. In addition, it can be possibly integrated with mature IC manufacturing process widely used today and realizes monolithic integration. Therefore, we can extend the applications of Si such as taking the Si optoelectronic integrated circuits (OEIC) for optical communication systems.

## 2. METHODOLOGY

### 2.1 Manufacturing process

Here, a description of the manufacturing process of forming the emitting layer on Si wafer is given. It will be seen that the process is very simple and easy to be applied in mass production.

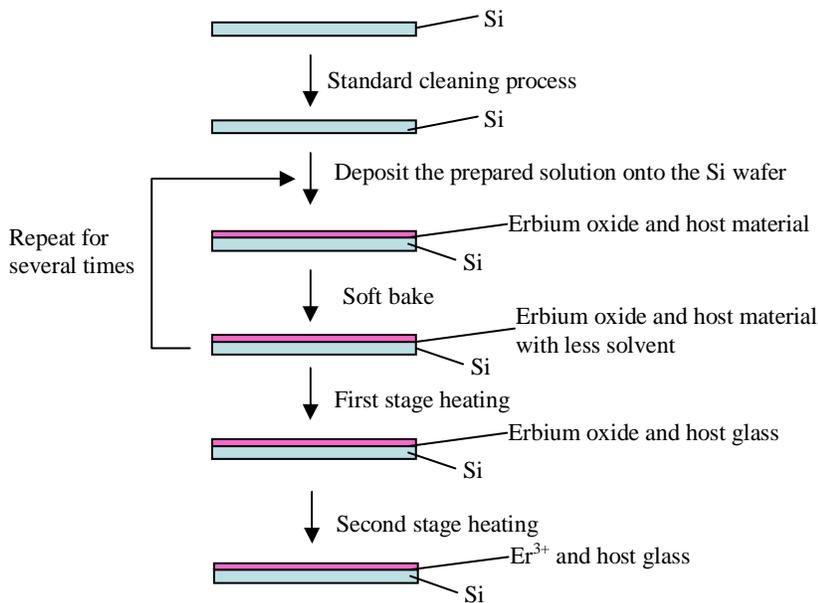


Fig. 1 Sample manufacturing process.

### 2.1.1 Preparing the solution for emitting layer

$\text{Er}_2\text{O}_3$ ,  $\text{P}_2\text{O}_5$ , Si, Al and Ag nanoparticles are mixed into spin-on glass (SOG), which is taken as the host material. The nanoparticles are uniformly distributed in the solution by means of the ultrasonic agitation. This solution will be deposited onto the Si substrate.

### 2.1.2 Steps of forming emitting layer

The detail steps are schematically shown in Fig.1. A piece of silicon wafer is cleaned with standard procedure [5]. Then, we deposit the solution on the silicon wafer and soft bake it at  $80^\circ\text{C}$  for 1 minute to remove part of solvent. This step is repeated a few times to obtain a thick enough emitting layer. The thickness of emitting layer will affect the photoluminescence intensity. In the final step, heat the sample at  $880^\circ\text{C}\sim 1000^\circ\text{C}$  for 30~150 minutes in order to make the  $\text{Er}_2\text{O}_3$  release  $\text{Er}^{3+}$  and react with SOG. The heating temperature has to be  $880^\circ\text{C}$  or above so that  $\text{Er}_2\text{O}_3$  would be reactive [2]. The heat treatment is an important experimental parameter. The final heating step significantly dominates the light emission efficiency of emitting layers. This important parameter will also be discussed in Sec. 3.

The different sets of  $\text{Er}_2\text{O}_3$ ,  $\text{P}_2\text{O}_5$ , Si, Al, and Ag mixture with different ratio were experimented. The different compositions result in samples with variable properties, such as conductivity and formation of cluster. Table I lists these different compositions.

## 2.2 Photoluminescence setup

Fig.2 shows the measurement setup. Take 980nm laser diode as pumping source, and focus it on the sample after a collimator and a mirror. InGaAs detector receives the light emitted by the sample through two lenses and CM110 monochromator. Finally, lock-in amplifier SRS SR830 that is triggered by optical chopper MC1000 amplifies the signals from detector, and transfers the amplified signals to the computer. Following measurements of photoluminescence are under optimized alignment.

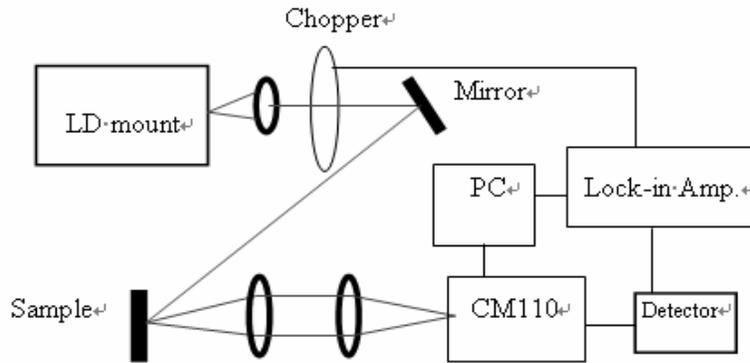


Fig. 2 Photoluminescence spectra of Set A and B.

### 3. DATA & RESULTS

#### 3.1 Spin-on glass mixed with $\text{Er}_2\text{O}_3$ only

Fig. 3 illustrates the photoluminescence spectrum of the sample that only  $\text{Er}_2\text{O}_3$  and SOG were mixed in its emitting layer. Although the compositions are quite simple, there is already an emitting peak at 1530 nm. The signal to background ration is quite poor, but it is still an encouraging result to make us move ahead with this promising work.

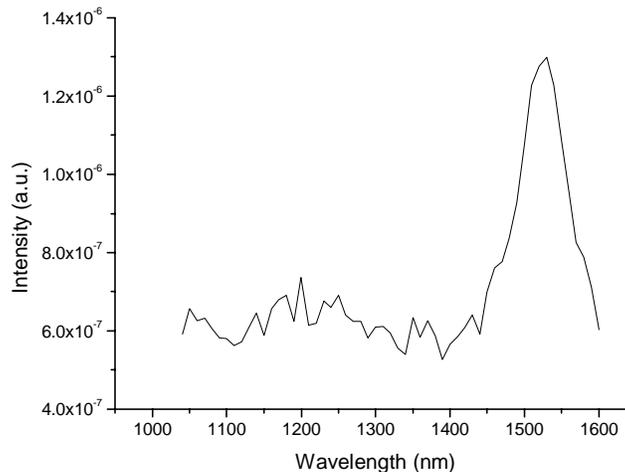


Fig. 3 Photoluminescence spectrum of the sample that only  $\text{Er}_2\text{O}_3$  and SOG were mixed in its emitting layer.

#### 3.2 Spin-on glass mixed with $\text{Er}_2\text{O}_3$ and $\text{P}_2\text{O}_5$

A large  $\text{Er}^{3+}$  doping concentration in silica glass results in concentration quenching caused by ion clusters. Phosphate and silicate glasses are superior to silica glass for the realization of high concentration erbium-doped optical amplifiers. Due to the high solubility of rare-earth ions without cluster formation, phosphate glasses are of great interest as a gain medium for compact erbium-doped optical amplifiers [3, 6-8]. Therefore, we add  $\text{P}_2\text{O}_5$  nanoparticles to the solution of light emitting layer to reduce cluster formation.

TABLE I  
SUMMARY OF COMPOSITIONS

Set	Weight ratio of nanoparticles and spin-on glass					
	Er <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	Si	Al	Ag	SOG
A1	1	0.5				4
A2	1	0.5				7
C	1	1			0.5	1
D1	1	0.5				7
D2	1	0.5	0.1			7
E	1	0.5		0.05		4
F1	1	2	1			2
F2	1	2	1			2

Current Er<sup>3+</sup> doped fiber amplifiers typically utilizes approximately 20 m of silica fiber doped with a few hundred ppm weight Er<sup>3+</sup> ions. A larger Er<sup>3+</sup> doping concentration in silica glass results in concentration quenching caused by ion clusters. However, an Er<sup>3+</sup> doping concentration greater than 2 wt% is required to decrease the length of the active fiber to a few centimeters for integrated devices. This is achievable with phosphate and silicate glasses which are superior to silica glass for the realization of high concentration erbium-doped optical amplifiers [3].

In these sets, P<sub>2</sub>O<sub>5</sub> nanoparticles are added to the solution of emitting layer, and then apply it to the Si substrate as Fig. 1 illustrated. The composition and the heat treatment are listed in TABLE I and TABLE II.

It can be seen that the signal to background level ratio has been greatly improved due to lowering the Er<sup>3+</sup> cluster formation. Comparing the PL results of Set A1 and Set A2 (TABLE III), peak intensity of Set A1 is about 2.5 times of that for Set A2. This tells us that the composition can influence the emission efficiency.

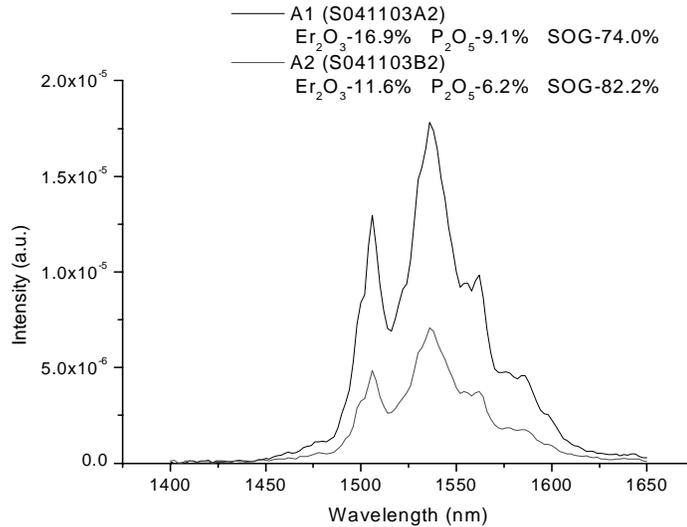


Fig. 4 Photoluminescence spectra of P<sub>2</sub>O<sub>5</sub> nanoparticles doped samples.

### 3.3 Effect of P<sub>2</sub>O<sub>5</sub> and Ag nanoparticles

The effect of P<sub>2</sub>O<sub>5</sub> and Ag nanoparticles was investigated with samples prepared using a similar procedure (Fig. 1). We added P<sub>2</sub>O<sub>5</sub> nanoparticles and Ag nanoparticles to the solution of Set C. Fig. 5 illustrates the photoluminescence spectrum of Set C. These samples have different physical properties and all of them emit light at 1530nm with different light emission characteristics of spectrum. Set C shows highest signal level among all the sets. Although it has quite high signal level, the layer thickness of sample C is not uniform.

TABLE II  
SUMMARY OF HEAT TREATMENT

Set	Soft bake at 80°C (min)	300°C Duration (min)	1000°C Duration (min)
A1	1	30	90
A2	1	30	90
C	1		90
D1	1		90
D2	1		90
E	1		90
F1	1		30
F2	1		90

The original motivation of adding Ag nanoparticles to the solution of the emitting layer is to improve conductivity. However, we discovered an extra benefit of increasing light emission efficiency. Fig. 5 shows the measured spectrum with the high peak intensity of photoluminescence for the sample with the Ag nanoparticles (Set C). To explain this phenomenon, we presume that Ag atoms play the role that transfers energy to  $\text{Er}^{3+}$  by free carrier absorption. Therefore,  $\text{Er}^{3+}$  ions are more easily to be excited for efficiency increase.

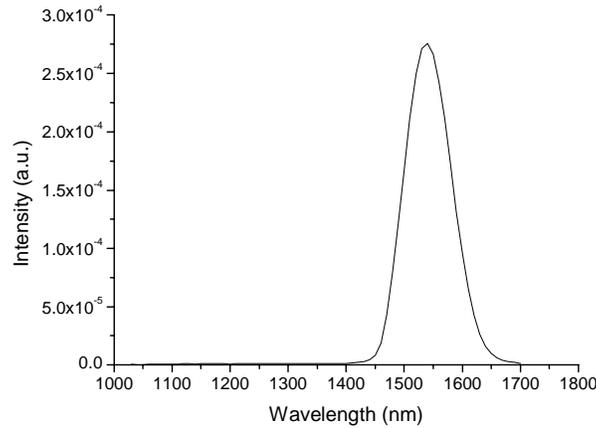


Fig. 5 Photoluminescence spectrum of Set C shows high intensity at 1530 nm.

### 3.4 $\text{Er}_2\text{O}_3$ , $\text{P}_2\text{O}_5$ , and Si mixed with spin-on glass

According to our experiments, Si nanoparticles could also improve the emission at 1530 nm by 47% (Fig. 6). Like Ag nanoparticles does in Set C, Si nanoparticles play the role that absorb energy from 980 nm pumping source and transfer energy to  $\text{Er}^{3+}$ , so the emission efficiency is increased.

### 3.5 $\text{Er}_2\text{O}_3$ , $\text{P}_2\text{O}_5$ , and Al mixed with spin-on glass

The samples with Al nanoparticles are also prepared. Fig. 7 is PL spectrum with the sample pumped by different pumping power. It shows relatively high signal level (13.7dB) and smooth spectrum curves. Al nanoparticles are possibly oxidized to form  $\text{Al}_2\text{O}_3$  in the heating process and it has been known that  $\text{Al}_2\text{O}_3$  can improve the solubility of rare earth ions.

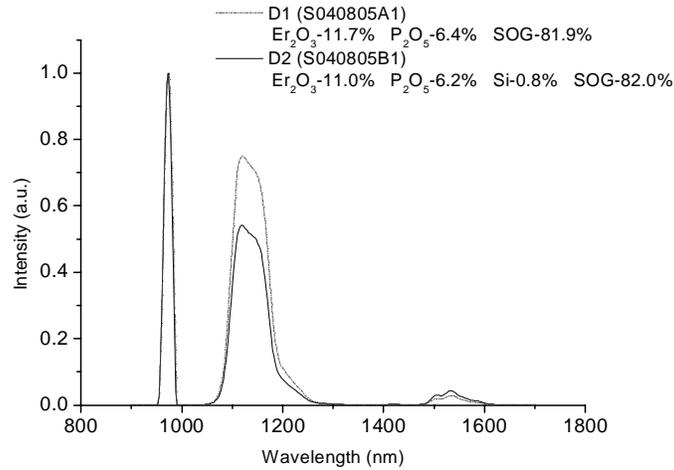


Fig. 6 Photoluminescence spectra of Set D1 & D2 pumped by 980 nm laser.

It was found  $\text{Al}_2\text{O}_3$  improves fluorescent lifetime of  $\text{Er}^{3+}$  ions, but the integrated absorption cross-section of  $\text{Er}^{3+}$  ions decreases with the increase of  $\text{Al}_2\text{O}_3$  concentration. Lower  $\text{Al}_2\text{O}_3$  content, and 0.2 – 0.4 mol%  $\text{Er}_2\text{O}_3$  are preferred for LD pumped microchip laser application. OH groups in glass greatly affect fluorescent intensity and lifetime of  $\text{Er}^{3+}$  [9]. When the intermediate oxide  $\text{Al}_2\text{O}_3$  in the glass is replaced by  $\text{Er}_2\text{O}_3$ , the number of non-bridging oxygen atoms stayed as constant. However, due to the higher polarizability of Er, the refractive index increases rapidly [3].

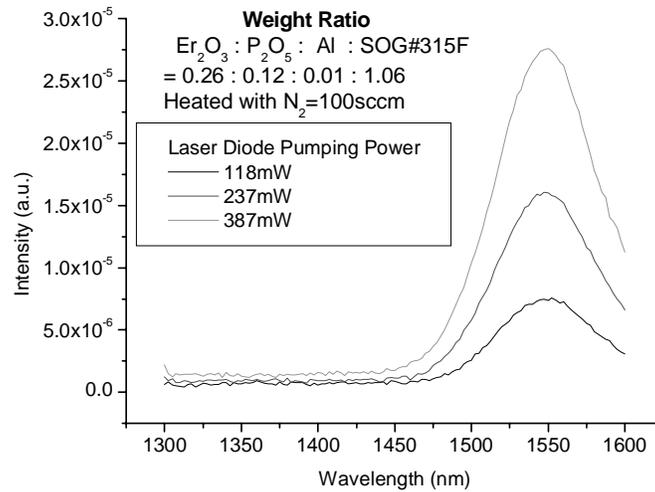


Fig. 7 Photoluminescence spectra of Set E with Al nanoparticles under different pumping power.

### 3.6 Effect of the thickness of emitting layer

As mentioned in Sec. 2.1.2, we thicken the emitting layer because the thickness of emitting layer affects the photoluminescence intensity. Fig. 8 illustrates that the sample with thicker emitting layer has stronger emission at 1530 nm. The reason is that the thicker layer has longer active region for optical pumping. However, in the electroluminescence case, to benefit electron tunneling, the emitting layer would be thin for current conduction.

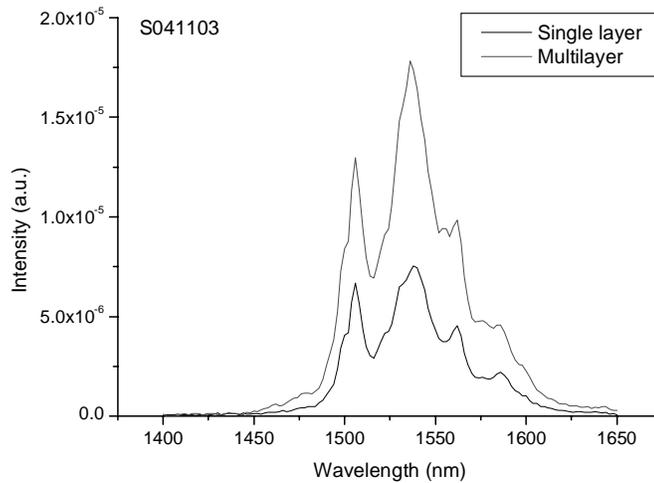


Fig. 8 Comparison of multi/single emitting layer.

### 3.7 Effect of heating duration

To reveal the effect of heating duration, we compare the difference between set E1 and set E2, which was heated for 30 and 150 minutes respectively. Their manufacturing processes are the same as other samples (Fig. 1), and compositions are listed in TABLE I. Fig. 9 shows the photoluminescence for Set E1 and Set E2. The comparison between E1 and E2 shows that samples with a longer heating duration (E2) have more emission than samples with a shorter heating duration (E1). Their signal to background level ratio is listed in TABLE III.

The heat treatment is also an important parameter of the experiment. In our experiment, samples without heat treatment of high temperature cannot emit light around 1530 nm because  $\text{Er}_2\text{O}_3$  is inertial below  $880^\circ\text{C}$ . Experiments show that light emission increases with the heating duration (Fig. 9). The peak intensity of Set E2 is about five times than that of Set E1.

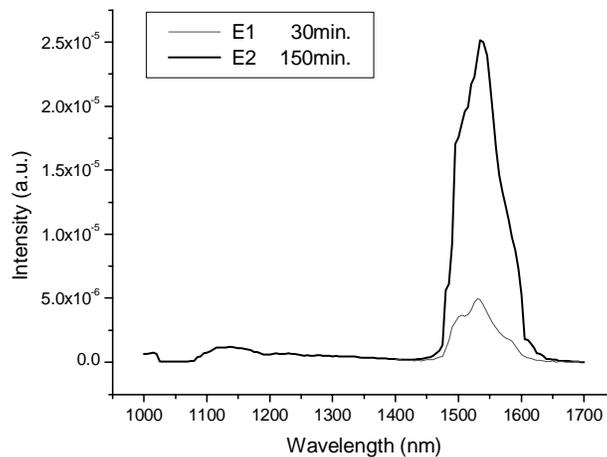


Fig. 9 Photoluminescence spectra of Set E1 and E2 show the effect of heating duration.

TABLE III  
SUMMARY OF SIGNAL TO BACKGROUND LEVEL RATIO

Set	Signal Peak Values (a.u.)	Background Level (a.u.)	Peak to Background Ratio (dB)
A1	$1.78 \times 10^{-5}$	$5.3 \times 10^{-8}$	25.3
A2	$7.07 \times 10^{-6}$	$5.3 \times 10^{-8}$	21.3
C	$2.76 \times 10^{-4}$	$9.2 \times 10^{-7}$	24.9
D1	$4.11 \times 10^{-5}$	$1.1 \times 10^{-6}$	15.7
D2	$8.53 \times 10^{-5}$	$9.1 \times 10^{-7}$	19.7
E	$2.76 \times 10^{-5}$	$1.5 \times 10^{-6}$	12.6
F1	$4.97 \times 10^{-6}$	$4.9 \times 10^{-7}$	10.1
F2	$2.51 \times 10^{-5}$	$4.9 \times 10^{-7}$	17.2

## 5. CONCLUSIONS

Emitting 1530 nm light on Si wafer is very useful because 1530nm is an important band in optical fiber communication. We demonstrated a simple process to form light-emitting layer on Si. The properties of samples can be varied through controlling the composition. A very valuable part of the process is that it can be possibly integrated with IC manufacturing process. In the future, building electrical and optical systems together on Si will be an expected achievement.

## REFERENCES

1. Dominique Bayart, "Erbium-doped and Raman fiber amplifiers" *C. R. Physique*, 4, pp. 65–74, 2003
2. S. Kennou, S. Ladas, M. G. Grimaldi, T. A. Nguyen Tan, J. Y. Veuillen, "Oxidation of thin erbium and erbium silicide overlayers in contact with silicon oxide films thermally grown on silicon" *Applied Surface Science*, 102, pp. 142-146, 1996.
3. Karine Seneschal, Frederic Smektala, Shibin Jiang, Tao Luo, Bruno Bureau, Jacques Lucas, Nasser Peyghambarian, "Alkaline-free phosphate glasses for ultra compact optical fiber amplifiers at 1.5  $\mu\text{m}$ " *Journal of Non-Crystalline Solids*, 324, pp. 179-186, 2003.
4. Michel J. F. Digonnet, *Rare Earth Doped Fiber Lasers and Amplifiers*, Dekker, 1993
5. Xiao, *Introduction to Semiconductor Manufacturing Technology*, Prentice Hall, 2001
6. William J. Miniscalco, "Erbium-Doped Glasses for Fiber Amplifiers at 1500 nm" *Journal of Lightwave Technology*, Vol. 9, No. 2, February 1991.
7. Emmanuel Desurvire, *Erbium-doped Fiber Amplifiers*, John Wiley & Sons, 1994
8. Zhuping Liu, Changhong Qi, Shixun Dai, Yasi Jiang, Lili Hu, "Spectra and laser properties of  $\text{Er}^{3+}$ ,  $\text{Yb}^{3+}$ :phosphate glasses" *Optical Materials*, 21, pp. 789 – 794, 2003
9. P.W. France, *Optical Fiber Lasers and Amplifiers*, Blackie, 1991

\*cflin@cc.ee.ntu.edu.tw; phone 886 2 3366-3540