行政院國家科學委員會專題研究計畫　成果報告

子計畫二:奈米粒子與光學晶體上之光電元件製作和研究

(2/2)

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行政院國家科學委員會專題研究計畫成果報告
奈米粒子與光學晶體上之光電元件製作與研究 (2/2)
Fabrication and investigation of optoelectronic devices based on nano-particles and photonic crystals

計畫編號：NSC 92-2120-E-002-003
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一、　中文摘要

在今年度的研究中，我們成功製作出將氧化鉺 (erbium oxide)與氧化磷 (phosphorous oxide)混合旋塗玻璃 (spin-on glass) 溶液，佈塗在矽基板 (silicon substrate)上，製作簡單且低成本的發光層，發射波長在光通訊使用的 1530 nm。

關鍵詞：奈米科技、光通訊、稀土元素、氧化鉺、氧化磷。

英文摘要

We introduce a new manufacturing technique that lays a light emitting layer on silicon wafer. This layer is formed with the mixture of Er2O3 nanoparticles and spin-on glass. It emits 1530nm light because Er2O3 releases Er3+ at high temperature. The process is simple. We also add P2O5, Al and Ag nanoparticles to improve light emission.

Key words: Nanotechnology, optical communication, optical pumping, rare earth compounds.

二、前言與研究目的

The amount of data transmission continues to grow, so the advantage of high transmission capability of optical fiber is of great worth. The demand for optical components also rockets up. Erbium-doped optical fiber amplifier is the core technique of modern optical communication systems. However, erbium-doped optical fiber has several disadvantages. For example, it 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 1530nm. The fabrication process is very simple and nearly costless, compared with ion implantation technique. In addition, it can be possibly integrated with mature IC manufacturing process widely used today and realizes monolithic

integration. Moreover, we can extend the applications of Si such as taking the Si optoelectronic integrated circuits (OEIC) for the light source of optical communication systems.

三、文獻探討

Erbium doped fiber is the cone technology of modern optical communication systems [1]. Semiconductor manufacturing processes have been extensively studied [2]. With different nanoparticles size, quantum size effect on optical properties can be observed [2]. Different co-dopant can change the property of erbium doped fiber [3-7]. In this work, optical pumping and photoluminescence of emitting layer of Er2O3 nanoparticles are demonstrated. It was found Al_2O_3 improves fluorescent lifetime of Er^{3+} ions, but the integrated absorption cross-section of Er^{3+} ions decreases with the increase of Al_2O_3 concentration [8].

四、研究方法

1. Preparation of emitting layer

The manufacturing process is schematically shown in Fig.1. A piece of silicon wafer is cleaned with standard procedure. Er_2O_3, P_2O_5, Al and Ag nano-particles are mixed into spin-on glass (SOG), which is taken as the host material. The Er_2O_3 nanoparticles are uniformly distributed in the solution. Then, we deposit the solution on the silicon wafer and soft bake in 80^{o}C for 1 minute. Repeat this step for a few times to obtain a thick emitting layer. In the final step, heat the sample in 880^{o}C~1000^{o}C for 30~150 minutes in order to make the Er_2O_3 release Er^{3+} and react with SOG. The heating temperature has to be 880^{o}C or above so that Er_2O_3 would be reactive.

We also used high energy ultraviolet laser to anneal the samples. The energy is so high that the Si substrate could be melted and then re-crystallizes. This post heat treatment can help the emitting layer gets more uniform and promote the light emission efficiency.

Though, we have to make some compromise. If the energy density is high, the emitting would be spluttered. Therefore, we lower the energy density to avoid the phenomenon, and have Er^{3+} better bounded with glass structure in the emitting layer.

A large 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.

TABLE I
SUMMARY OF COMPOSITIONS AND HEATING TREATMENT

	Compositions (Volume Ratio)				Heating Temperature and Duration	
Set	Er_2O_3	SOG	P_2O_5	Ag	Temperature (^oC)	Duration (min.)
A	1	1			880	90
B	1	1	1		1000	90
C1	2	2	2	1	1000	90
D1	1	2	2	1	1000	30
D2	1	2	2	1	1000	150

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. Therefore, we add P_2O_5 nanoparticles to the solution of light emitting layer to reduce cluster formation.

The different sets of Er_2O_3, P_2O_5, and Ag mixture with different volume 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. 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 monochromater. 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.

3. Setup of measuring optical gain

Fig. 8 illustrates the setup of optical gain measurement. The specimen is pumped with the 980 nm laser diode because Er^{3+} has strong absorption around this wavelength. Pumping light emitted out from the laser diode is collimated by the first lens. Afterwards, it is re-focused by the cylindrical lens and shaped into a narrow light stripe. This light stripe is cast onto the specimen with a movable block placed in front of it. We can change the length of the pumping stripe on specimen by moving the block. To prevent the pumping source (980 nm) entering the monochromator, a low pass filter is set in front of the monochromator. The measurements of photoluminescence are done with optimized alignment.

五、結果與討論

A. Er_2O_3, and SOG Only

In Set A, only Er2O3 and SOG were mixed. Although the compositions are quite simple, there is already an emitting peak at 1530nm (Fig. 3).

B. Effect of P_2O_5 and Ag Nanoparticles

The effect of P2O5 and Ag nanoparticles was investigated with samples prepared using a similar procedure (Fig. 1). We added P2O5 nanoparticles to the solution of Set B, and Ag nanoparticles to the solution of Set C1. Fig. 3 illustrates the photoluminescence spectra of Set A and Set B. Fig. 4 illustrates the photoluminescence spectrum of Set C1. These samples have different physical properties and all of them emit light at 1530nm with different light emission characteristics of spectrum. Set C1 shows highest signal level among all the sets. Although it has quite high signal level, the layer of sample C1 is somehow badly uniform.

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. 4 illustrates the high peak intensity of photoluminescence for the sample with the Ag nanoparticles (Set C1). To explain this phenomenon, we presume that Ag atoms play the role that transfers energy to Er^{3+} by free carrier absorption. Therefore, Er^{3+} ions are more easily to be excited for efficiency increase.

C. Effect of Heating Duration

To reveal the effect of heating duration, we compare the difference between set D1 and set D2, 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. 5 shows the photoluminescence for Set D1 and Set D2. The comparison between D1 and D2 shows that samples with a longer heating duration (D2) have more emission than samples with a shorter heating duration (D1). Their signal to background level ratio is listed in TABLE II.

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 1530nm because

TABLE II
SUMMARY OF SIGNAL TO BACKGROUND LEVEL RATIO

Set	Signal Peak Values (a.u.)	Background Level (a.u.)	Peak to Background Ratio (dB)
A	1.30×10^{-6}	6.2×10^{-7}	3.2
B	3.49×10^{-6}	4.9×10^{-7}	8.5
C1	2.76×10^{-4}	9.2×10^{-7}	24.9
D1	4.97×10^{-6}	4.9×10^{-7}	10.1
D2	2.51×10^{-5}	4.9×10^{-7}	17.2

Er_2O_3 is inertial below 880°C. Experiments show that light emission increases with the heating duration (Fig. 5). The peak intensity of Set D2 is about five times than peak intensity of Set D1.

D. Effect of Al Nanoparticles

The samples with Al nanoparticles are also prepared. Fig. 6 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 Al_2O_3 in the heating process and it has been known that Al_2O_3 can improve the solubility of rare earth ions.

Ordinary erbium-doped optical fiber requires a quite long distance to acquire significant light emission from Er^{3+} ions. Relatively, our emitting layer is able to show the signals only within 0.5μm due to the higher concentration of Er^{3+} and surface effect of nano-particles.

It was found Al_2O_3 improves fluorescent lifetime of Er^{3+} ions, but the integrated absorption cross-section of Er^{3+} ions decreases with the increase of Al_2O_3 concentration. Lower Al_2O_3 content, and $0.2 - 0.4$ mol% Er_2O_3 are preferred for LD pumped microchip laser application. OH groups in glass greatly affect fluorescent intensity and lifetime of Er^{3+}. [8]

D. Effect of Si Nanoparticles

A large 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 [2]. Therefore, P_2O_5 nanoparticles are added to the solution of light emitting layer to reduce cluster formation. According to our experiments, the heating temperature and duration are important parameters. Si nanoparticles could also improve the emission at 1530 nm by 47% (Fig. 7). Si nanoparticles play the role that absorb energy form 980 nm pumping source and transfer energy to Er^{3+}, so the emission efficiency is increased.

E. Observation of Optical Gain

The signal at 1530 nm is observed to super-linearly proportional to the pumping length, while the pumping power is linearly proportional to the pumping length, as show in Fig. 9. This is evidence that the emitting layer reveals optical gain.

六、計畫結果自評

Emitting 1530nm 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.

七、參考資料

[1] Michel J. F. Digonnet, *Rare Earth Doped Fiber Lasers and Ampilifers*, Dekker, 1993
[2] Xiao, *Intooduction to Semiconductor Manufacturing Technology*, Prentice Hall, 2001
[3] William J. Miniscalco, "Erbium-Doped Glasses for Fiber Amplifiers at 1500 nm" *Journal of Lightwave Technology, Vol. 9, No. 2, February 1991.*
[4] Emmanuel Desurvire, *Erbium-doped Fiber Amplifiers*, John Wiley & Sons, 1994
[5] P.W. France, *Optical Fiber Lasers and Amplifiers*, Blackie, 1991
[6] 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.
[7] Karine Seneschal, Frederic Smektala, Shibin Jiang, Tao Luo, Bruno Bureau, Jacques Lucas, Nasser Peyghambarian, "Alkaline-free phosphate glasses for ultra compact opticalfiber amplifiers at 1.5 μm" *Journal of Non-Crystalline Solids*, 324, pp. 179-186, 2003.
[8] Zhuping Liu, Changhong Qi, Shixun Dai, Yasi Jiang, Lili Hu, "Spectra and laser properties of Er^{3+}, Yb^{3+}:phosphate glasses" *Optical Materials*, 21, pp. 789 – 794, 2003

八、圖表

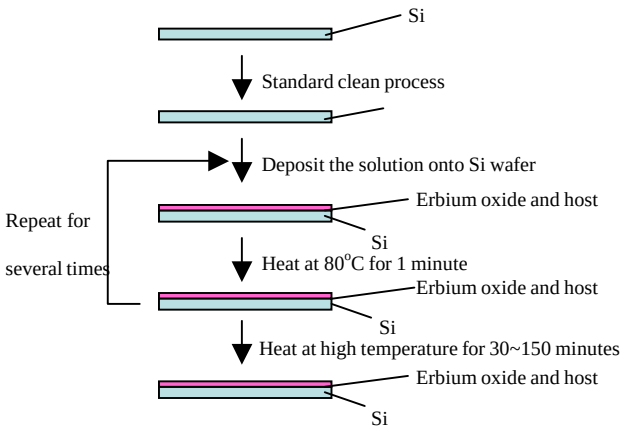

Fig. 1: Sample manufacturing process.

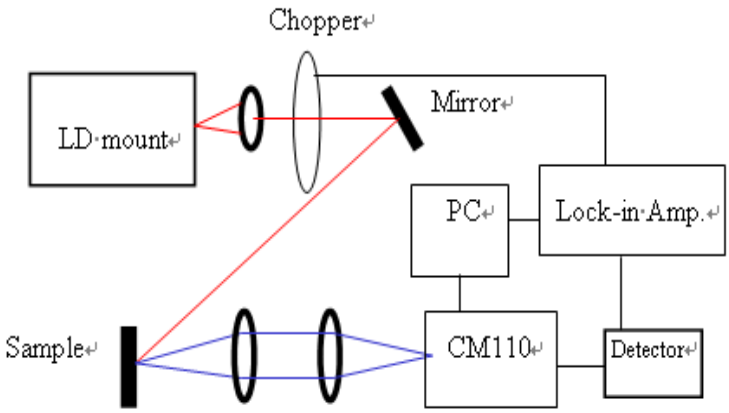

Fig. 2: Photoluminescence measurement setup

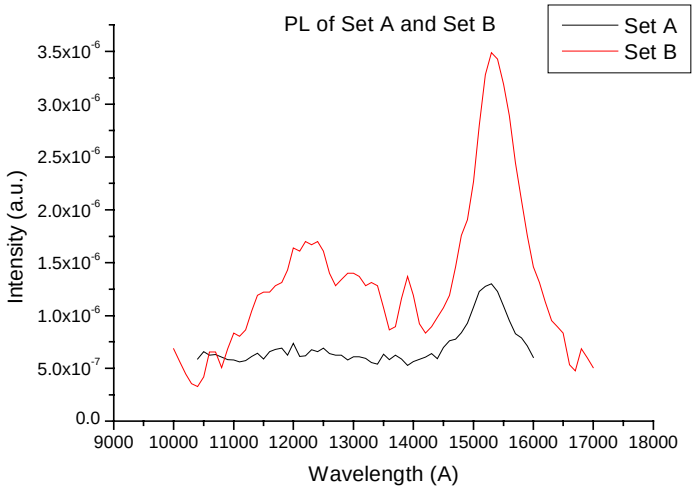

Fig. 3: Photoluminescence spectrum of Set A and B.

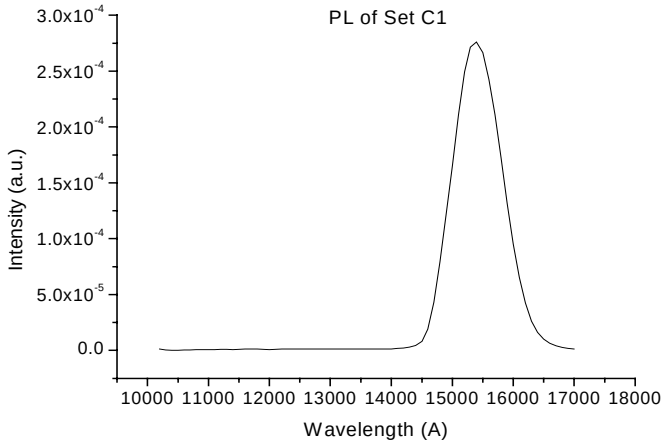

Fig. 4: Photoluminescence spectrum of Set C1 shows high intensity at 1530nm.

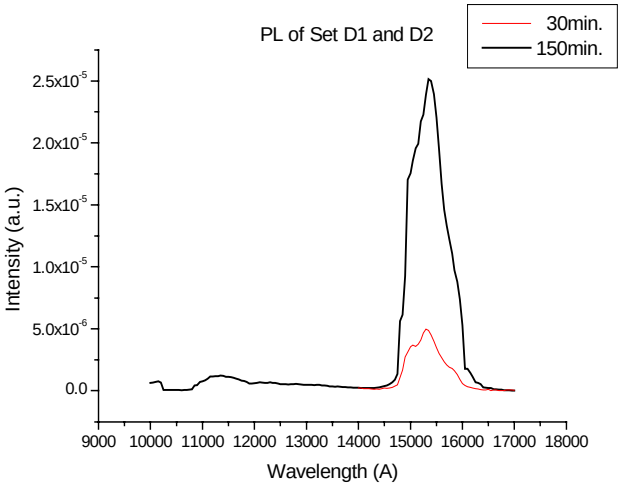

Fig. 5: Photoluminescence spectra of Set D1 and D2 show the effect of heating duration.

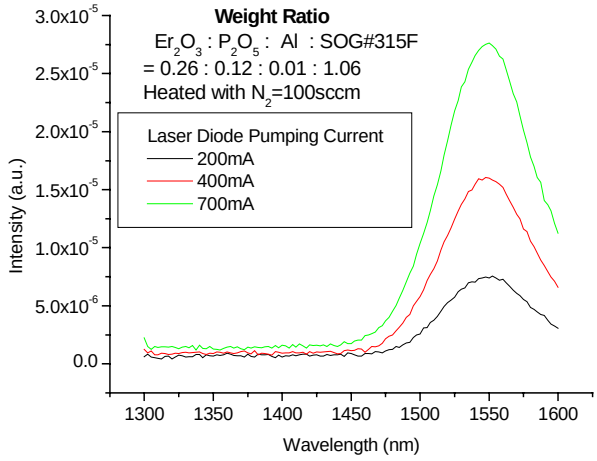

Fig. 6: Photoluminescence spectra of sample with Al nanoparticles under different pumping power

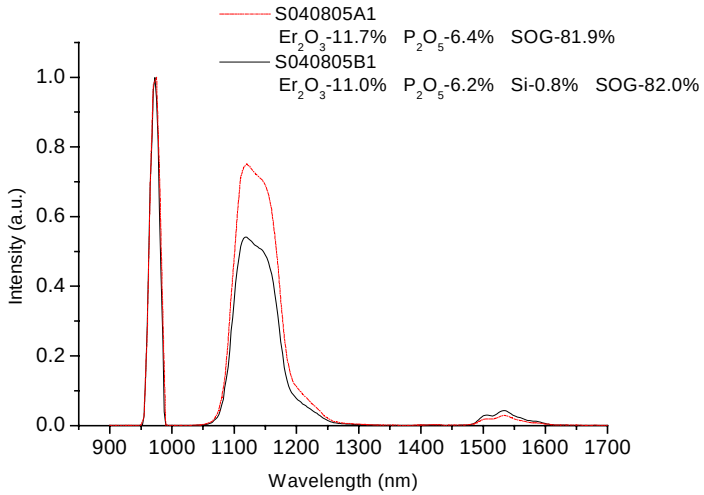

Fig.7 Photoluminescence spectra pumped by 980 nm laser. Reveal the effect the Si nanoparticles.

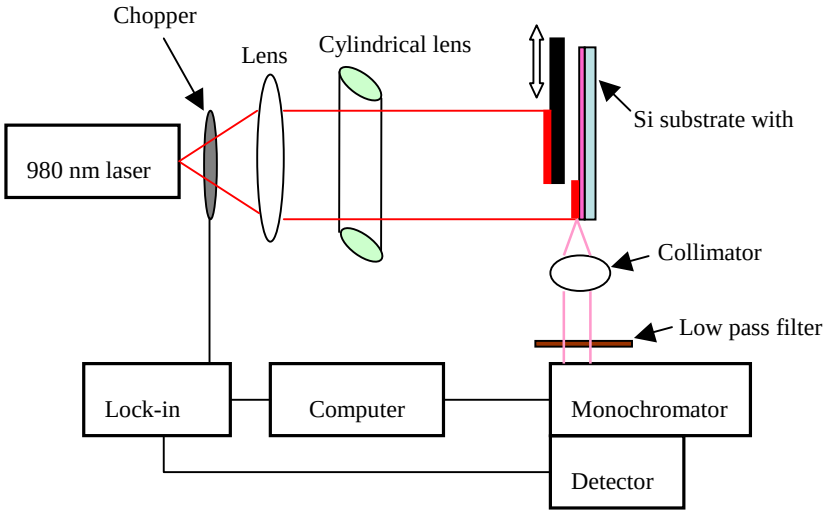

Fig. 8. Setup of measuring optical gain

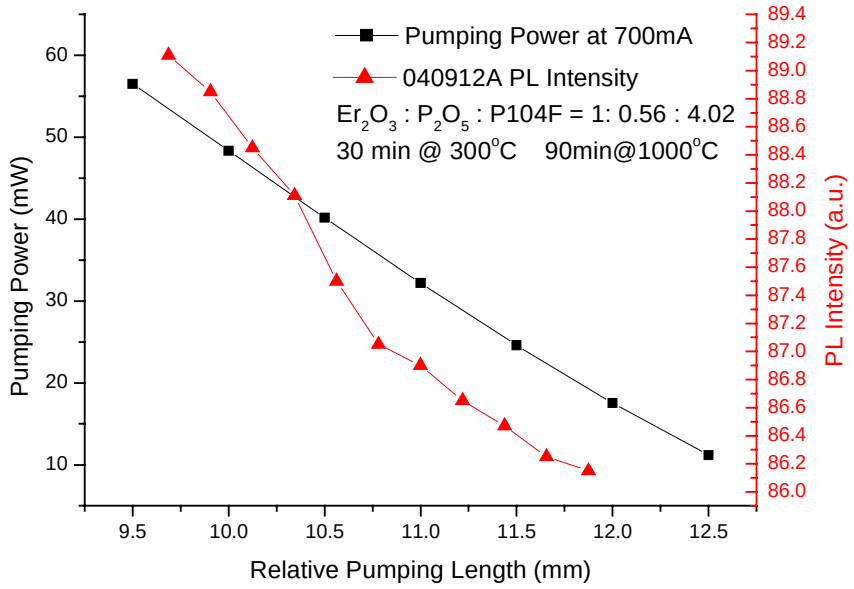

Fig. 9 The pumping power at 980 nm and the optical signal at 1530 nm vs. pumping length.