

Waveguiding and photoluminescence in Er³⁺ - doped Ta₂O₅ planar waveguides

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ABSTRACT

The optimization of erbium-doped Ta₂O₅ thin film waveguides deposited by magnetron sputtering onto thermally oxidized silicon wafer is described. Optical constants of the film were determined by ellipsometry. For the slab waveguides, background losses below 0.4 dB/cm at 633 nm have been obtained before post-annealing. The samples, when pumped at 980 nm yielded a broad photoluminescence spectrum (FWHM~50 nm) centered at 1534 nm, corresponding to ⁴I_{13/2}-⁴I_{15/2} transition of Er³⁺ ion. The samples were annealed up to 600 °C and, both photoluminescence power and fluorescence lifetime increase with post-annealing temperature and a fluorescence lifetime of 2.4 ms was achieved, yielding promising results for compact waveguide amplifiers.

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1. INTRODUCTION

Optical amplifiers are key components in optical telecommunications and in fully-integrated optical systems. Erbium doped materials are of particular importance in optical communications technology, due to their excellent performance as gain media for amplifiers and lasers at the telecommunications wavelength of $1.5 \mu\text{m}$. Low-cost, compact erbium doped waveguide amplifiers (EDWAs) are essential for local-loop optical systems, and fully-functional densely integrated planar lightwave circuits (PLCs) will rely upon gain in much the same way as electronic integrated circuits do at present. Thus far, many erbium doped materials with optical gain have also been demonstrated [1-4]. In recent years, erbium-doped high index contrast materials have generated great interest [5-8], and will allow strong confinement of light, ultra compact photonic devices, and non-linear processes at moderate power levels. Tantalum (Ta_2O_5) has already been used as a host for rare earth ions [9-11], with lasing being achieved only in $\text{Nd}:\text{Ta}_2\text{O}_5$ to date [9]. This, combined with high refractive index (>2.0), moderate phonon energy for high radiative efficiency [12], a large third order non-linearity [13], and high photosensitivity [14], makes it an ideal material for realising multifunctional PLCs.

In this paper, the deposition and optimisation of erbium-doped Ta_2O_5 ($\text{Er}:\text{Ta}_2\text{O}_5$) thin films using magnetron sputtering is presented. The spectroscopic properties of Er^{+3} ions in tantalum are provided through photoluminescence and fluorescence lifetime measurements. The effect of different annealing temperatures on the photoluminescence and lifetime measurements of $\text{Er}:\text{Ta}_2\text{O}_5$ films are also presented to evaluate this material's potential as a high index contrast host for erbium and as an EDWA.

2. EXPERIMENTAL

2.1 Thin film deposition

Slab waveguides were fabricated by magnetron sputter deposition (Plasma400 Oxford Instruments) of a powder pressed, Er:Ta₂O₅ target (150 mm diameter) onto an oxidized silicon substrate (oxide thickness $\sim 2.1 \mu\text{m}$). The target was doped with 1 wt. % of Er₂O₃ ($\sim 2.5 \times 10^{20}$ ions/cm³). The deposition was carried out in a vacuum chamber pumped to a base pressure of 10^{-8} Torr and backfilled with an Ar:O₂ ambient. The flow rate for both the gases inside the chamber was separately controlled. The chamber pressure was maintained at a constant value of 10 mTorr. In order to have optically good as-deposited films, optimization of the sputter deposition parameters is very important. The parameters optimized for the deposition were substrate temperature, magnetron power and O₂ gas flow. Deposition was carried out by varying substrate temperature and setting other parameters to a reasonable value. Once the deposition was complete, optical loss was measured and the value that gave the lowest loss and an acceptable deposition rate was used for optimizing the next parameter. This process was carried out until all the three sputter parameters, mentioned above, were optimized.

2.2 Optical constant measurement

The refractive index and the thickness of the sputtered tantala thin films were determined by ellipsometry. The apparatus consisted of a white light source emitting in the visible region (400-700 nm) and a detector which collects the polarized reflected light. The measurements were performed at a 70° angle of incidence. The Cauchy dispersion model [15] was used to fit the experimental data points using the tantala film thickness and the Cauchy parameters (A, B and C) as fit parameters. The Cauchy dispersion relation is given by:

$$n(\lambda) = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4}$$

Where, A, B, C are constants to be determined by fitting to the experimental data.

$k(\lambda) = 0$ and $n(\lambda)$ is assumed real, where λ is the wavelength, k is the extinction coefficient and n is the refractive index. Mean squared error (χ^2) was used to determine the quality of the fit and was minimised to find the index and thickness.

2.3 Loss measurements

The slab waveguide losses were measured at 633 nm (He-Ne laser) by directly capturing and monitoring the scattered light intensity normal to the waveguide plane, using an imaging system (CCD). The light was coupled into the waveguide using prism coupling with a rutile prism being used to couple light into the fundamental mode of the Er:Ta₂O₅ waveguide. The propagation loss was then determined by mapping the decay of scattered light (pixels captured by the imaging system) along the propagation length of the guide. The losses were estimated at several different places on a sample to check the homogeneity of the sample, and averaged.

2.4 Optical characterization

Photoluminescence measurements were performed at room temperature by pumping erbium ions into their ⁴I_{11/2} level using a Ti:Sapphire laser emitting at 980 nm. The thickness of the tantalum thin film was approximately 2 μm . The pump was directed onto the sample at 45 degrees to the surface normal. The pump power at the surface of the sample was about 180 mW and the spot size of the pump beam was < 1 mm in diameter. The pump was mechanically chopped at 25 Hz. The luminescence was analyzed using a grating monochromator normal to the sample surface, with a spectral resolution of 10 nm. A filter was used just before the monochromator to block

wavelengths below 1300 nm. The signal was detected using an InGaAs detector cooled to $-15\text{ }^{\circ}\text{C}$ and amplified using a lock-in amplifier. Lifetime measurements were performed with 0.2 ms resolution by monitoring the decay of the luminescence on an oscilloscope after pumping the erbium ions and mechanically chopping of the pump light source. The luminescence intensity was fitted to a single exponential decay, and the natural log of this decay, normalised with respect to the maximum intensity was plotted against time. The slope of the fit gives the luminescence decay time. Photoluminescence measurements were performed on both the annealed and non-annealed samples.

3. RESULTS AND DISCUSSION

3.1 Waveguide characterization

The deposition rate was determined by measuring the film thickness for various sputtering times, using a stylus profilometer. Figure 1 shows the thickness plotted against time, for the deposition conditions optimized below, with the average deposition rate found to be $\sim 2\text{ nm}\cdot\text{min}^{-1}$.

The optical loss variation with substrate temperature is shown in figure 2. The amount of oxygen in the layers increases with increasing substrate temperature and the microstructure of the sputtered layer becomes more regular and attains improved stoichiometry [16]. This in turn reduces the scattering losses. The sample sputtered at $200\text{ }^{\circ}\text{C}$ gave the lowest losses ($\sim 0.40\text{ dB/cm}$ at 633 nm) and an acceptable deposition rate ($\sim 2\text{ nm}\cdot\text{min}^{-1}$), so was chosen as the optimized value for the substrate temperature. The increase in the loss for temperature above $300\text{ }^{\circ}\text{C}$ may be due to micro-cracking of film due to local heating and stress at that temperature, or perhaps simply due to some unwanted and un-removable scattering centres in the film.

Figure 3 shows the variation of optical loss with magnetron power, with low loss being achieved at both 300 W and 400 W. The deposition rate at 400 W was almost twice that at 300 W. However, the photoluminescence results (described in detail below) showed that both luminescence intensity and lifetime were better with samples deposited at 300 W. Therefore, 300 W was chosen as the optimum magnetron power value.

Argon is used to start and maintain the plasma discharge and its flow rate is not critical, but the oxygen flow rate plays an important role in achieving low loss films as shown in figure 4. With the increase in the O₂ flow rate, the film approaches its stoichiometric composition and hence lowest possible loss, but a further increase will lead to increased oxidization of the target surface and an unacceptably low deposition rate. For our samples, a flow rate of 5 sccm achieved the lowest loss value and the average deposition rate ($\sim 2 \text{ nm}\cdot\text{min}^{-1}$).

The optimized deposition parameters for achieving low loss Er:Ta₂O₅ slab waveguides are 200 °C substrate temperature, 300 W magnetron power and 5 sccm oxygen flow rate with argon flow rate and chamber pressure maintained at a constant value of 20 sccm and 10 mTorr respectively. In order to further reduce the losses the samples were annealed at high temperatures (450 °C and above) in oxygen in a tube furnace. In the loss measurement for the annealed samples the scattered power was too low to measure, providing evidence of reduced loss. The apparatus for loss measurement by imaging scattered light was unable to measure losses below 0.4 dB/cm, and methods such as the cutback [17] or Fabry-Perot [18] techniques need to be employed on channel waveguides to accurately determine losses below this level.

Ellipsometry measurements were performed on the sputtered Er:Ta₂O₅ samples to determine the thin film refractive index in the visible region. The best fit using Cauchy dispersion model yielded $A=2.069$, $B=2.133$ & $C=1.075$ as the values for the Cauchy dispersion relation constants. Figure 5 shows the refractive index vs. wavelength for the fully optimized Er:Ta₂O₅ film. A refractive index of 2.13 was measured at 633 nm. At 1550 nm, where an EDWA would operate, a refractive index of 2.08 is estimated by extrapolating the Cauchy dispersion relation to longer wavelengths. The thicknesses of the films were within the experimental error of the value measured by the stylus profilometer.

3.2 Photoluminescence characterization

The photoluminescence spectra of annealed and non-annealed magnetron sputtered Er:Ta₂O₅ samples are shown in figure 6. The samples were annealed at 450, 500, 550 and 600 °C, respectively but higher temperatures were not employed as annealing above 600 °C is expected to result in a lossy polycrystalline film [19]. The emission spectra correspond to the transition between the $^4I_{13/2}$ - $^4I_{15/2}$ levels of the Er³⁺ ion and peak at 1534 nm. The bandwidth of the spectrum (FWHM) was measured to be 50 nm which is substantially broader than those obtained from non-tellurite glasses (~30 nm) [20] and comparable to high index contrast hosts such as tellurite glasses (n~2.1, 65 nm) [21] and alumina (n~1.69, 55 nm) [22] and thus shows potential for broadband applications. The photoluminescence intensity increases with annealing temperature to about 14 times that of the unannealed sample at 600 °C. Figure 7 shows the bandwidth of the normalised photoluminescence spectra to be constant for the annealed and unannealed Er:Ta₂O₅ samples. This suggests that there is an absence of phase change from amorphous to crystalline nature and consequently the narrowing of bandwidth

even at 600 °C. Therefore, it can also be said that because of the absence of phase change the losses should not increase at such high annealing temperatures (up to 600 °C). The luminescence lifetime of the erbium ions is shown in figure 8. It can be seen from the fit (bold dashed lines) in figure 8 that the decay is almost purely single exponential, and the quality of fit improves with the increasing annealing temperature consequently, the luminescence decay time was found to increase from 0.53 ms for the as-deposited sample to 2.4 ms for the sample annealed at 600 °C. The value of lifetime is smaller than those obtained from non-tellurite glasses (10-15 ms) [20] and alumina (6 ms) [22] but comparable to high index contrast hosts such as tellurite glasses (3.5 ms) [23] and zirconia ($n \sim 2.04$, 1.8 ms) [2].

4. CONCLUSIONS

The deposition of Er:Ta₂O₅ by magnetron sputtering has been optimized to yield low loss slab waveguides (as-deposited thin film), 0.4 dB/cm at 633 nm as measured by imaging scattered light method. It is believed that the losses reduced further upon annealing in oxygen at high temperatures (> 450 °C) [9] but alternative loss measuring techniques like cutback or Fabry-Perot need to be employed on channel waveguides to measure these lower losses. The refractive index of the thin film was determined over the wavelength range from 400 nm to 700 nm. A broad photoluminescence spectrum (FWHM~50 nm) peaking at 1534 nm was obtained, and a luminescence lifetime of 2.4 ms was measured for the erbium ions in the Er:Ta₂O₅ film for optimized sputtering and annealing conditions. The results obtained for the losses and radiative lifetime are promising, for realizing erbium-doped integrated amplifier/laser and multifunctional photonic circuits based on Er:Ta₂O₅.

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REFERENCES

- [1] R.N. Ghosh, J. Shmulovich, C.F. Kane, M.R.X. de Barros, G. Nykolak, A.J. Bruce, and P.C. Becker, "8-mW threshold Er^{3+} -doped planar waveguide amplifier," IEEE Photon. Tech. Lett., **8**, 518 (1996)
- [2] K. Hattori, T. Kitagawa, M. Oguma, H. Okazaki and Y. Ohmori, "Optical amplification in Er^{3+} -doped P_2O_5 - SiO_2 planar waveguides," J. Appl. Phys., **80**, 5301 (1996)
- [3] Y.C. Yan, A.J. Faber, H. de Waal, P.G. Kik and A. Polman, "Erbium doped phosphate glass waveguide on silicon with 4.1 dB/cm gain at $1.535 \mu\text{m}$," Appl. Phys. Lett., **71**, 2922 (1997)
- [4] A.Q. Le Quang, R. Hierle, J. Zyss, I. Ledoux, G. Cusmai, R. Costa, A. Barberis and S.M. Pietralunga, "Demonstration of net optical gain at 1550nm in an erbium doped polymer single mode rib waveguide," Appl. Phys. Lett., **89**, 141124 (2006)
- [5] G.N. van den Hoven, R.J.I.M. Koper, A. Polman, C. van Dam, J.W.M. van Uffelen and M.K. Smit, "Net optical gain at 1.53 microns in Er-doped Al_2O_3 waveguides on silicon," Appl. Phys. Lett., **68**, 1886 (1996).
- [6] R. Schermer, W. Berglund, C. Ford, R. Ramberg and A. Gopinath, "Optical amplification at 1534 nm in erbium doped Zirconia waveguides," IEEE J. Quantum Electron., **39**, 154 (2003).
- [7] C.C. Baker, J. Heikenfeld, Z. Yu and J. Steckl, "Optical amplification and electroluminescence at 1.54 micron in Er-doped zinc silicate germanate on silicon," Appl. Phys. Lett., **84**, 1462 (2004).
- [8] P. Nandi and G. Jose, "Erbium doped phospho-tellurite glasses for 1.5 micron optical amplifiers," Opt. Commun., **265**, 588 (2006).
- [9] B. Unal, M.C. Netti, M.A. Hassan, P.J. Ayliffe, M.D.B. Charlton, F. Lahoz, N.M.B. Perney, D.P. Shepherd, C.Y. Tai, J.S. Wilkinson and G.J. Parker, "Neodymium doped tantalum pentoxide waveguide lasers," IEEE J. Quantum. Electron., **41**, 1565 (2005).
- [10] H. Rigneault, F. Flory, S. Monneret, S. Robert and L. Roux, "Fluorescence of Ta_2O_5 thin films doped by kilo-electron-volt Er implantation: application to microcavities," Appl. Opt., **35**, 5005 (1996).

- [11] N. Maeda, N. Wada, H. Onoda, A. Maegawa, K. Kojima, "Preparation and optical properties of sol-gel derived Er³⁺-doped Al₂O₃-Ta₂O₅ films," *Opt. Mater.*, **27**, 1851 (2005).
- [12] P.S. Dobal, R.S. Katiyar, Y. Jiang, R. Guo and A.S. Bhalla, "Raman scattering study of a phase transition in tantalum pentoxide," *J. Raman Spectrosc.*, **31**, 1061 (2000).
- [13] C.Y. Tai, J.S. Wilkinson, N.M.B. Perney, M.C. Netti, F. Cattaneo, C.E. Finlayson and J.J. Baumberg, "Determination of nonlinear refractive index in a Ta₂O₅ rib waveguide using self-phase modulation," *Opt. Exp.*, **12**, 5110 (2004).
- [14] C.Y. Tai, C. Grivas and J.S. Wilkinson, "UV photosensitivity in a Ta₂O₅ rib waveguide Mach-Zender interferometer," *IEEE Photon. Tech. Lett.*, **16**, 1522 (2004).
- [15] J.A. Thornton, "Influence of apparatus geometry and deposition conditions on the structure and topography of thick sputtered coatings," *J. Vac. Sci. Tech.*, **11**, 666 (1974).
- [16] A. Rickman, G.T. Reed, B.L. Weiss and F. Namavar, "Low-loss planar optical waveguides fabricated on SIMOX material," *IEEE Photon. Tech. Lett.*, **4**, 633 (1992).
- [17] T. Feuchter and C. Thirstrup, "High precision planar waveguide propagation loss measurement technique using a Fabry -Perot cavity," *IEEE Photon. Tech. Lett.*, **6**, 1244 (1994).
- [18] P.C. Joshi and M.W. Cole, "Influence of postdeposition annealing on the enhanced structural and electrical properties of amorphous and crystalline Ta₂O₅ thin films for dynamic random access memory applications," *J. Appl. Phys.*, **86**, 871 (1999).
- [19] P.M. Peters, D.S. Funk, A. P. Peskin, D.L. Veasey, N.A. Sanford, S.N. Houde-Walter, and J.S. Hayden, "Ion-exchanged waveguide lasers in Er³⁺/Yb³⁺ codoped silicate glass," *Appl. Opt.*, **38**, 6879 (1999).
- [20] R. Rolli, M. Montagna, S. Chaussement, A. Monteil, V.K. Tikhomirov and M. Ferrari, "Erbium-doped tellurite glasses with high quantum efficiency and broadband stimulated emission cross section at 1.5 μm ," *Opt. Mater.*, **21**, 743 (2003).
- [21] G.N. van den Hoven, E. Snoeks, A. Polman, J.W.M. van Uffelen, Y.S. Oei, and M.K. Smit, "Photoluminescence characterization of Er-implanted Al₂O₃ thin films," *Appl. Phys. Lett.*, **62**, 3065 (1993).
- [22] H. Yamauchi, G.S. Murugan, and Y. Ohishi, "Optical properties of Er³⁺ and Tm³⁺ ions in a tellurite glass," *J. Appl. Phys.*, **97**, 043505 (2005).

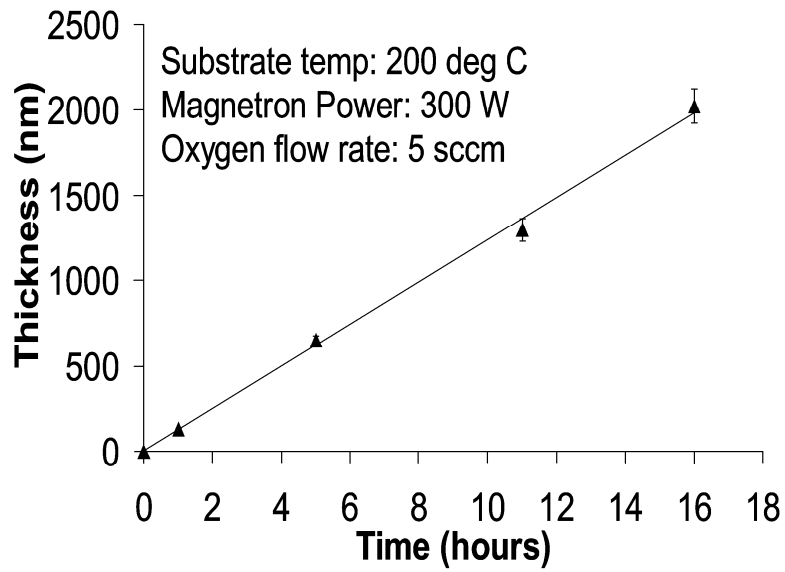


Figure 1 Deposition rate for sputtered Er:Ta₂O₅ thin film

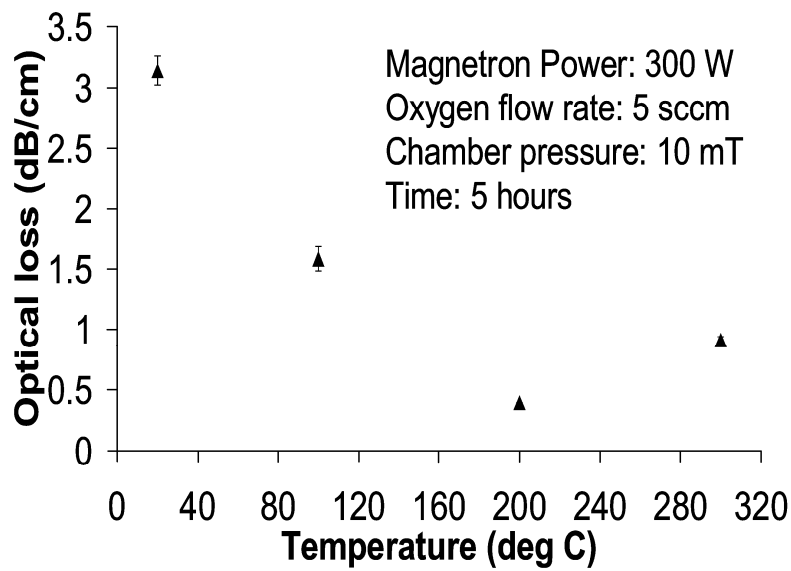


Figure 2 Optical loss at 633nm vs. substrate temperature for sputtered Er:Ta₂O₅ film

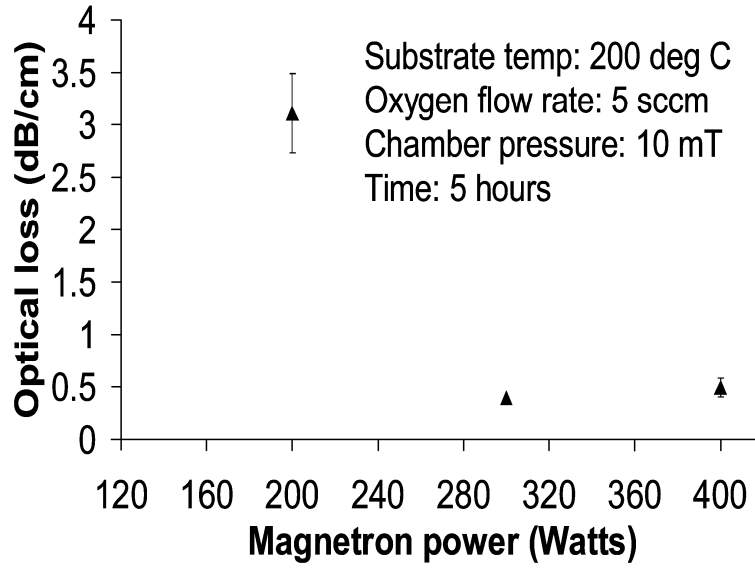


Figure 3 Optical loss at 633nm vs. magnetron power for sputtered Er:Ta₂O₅ film

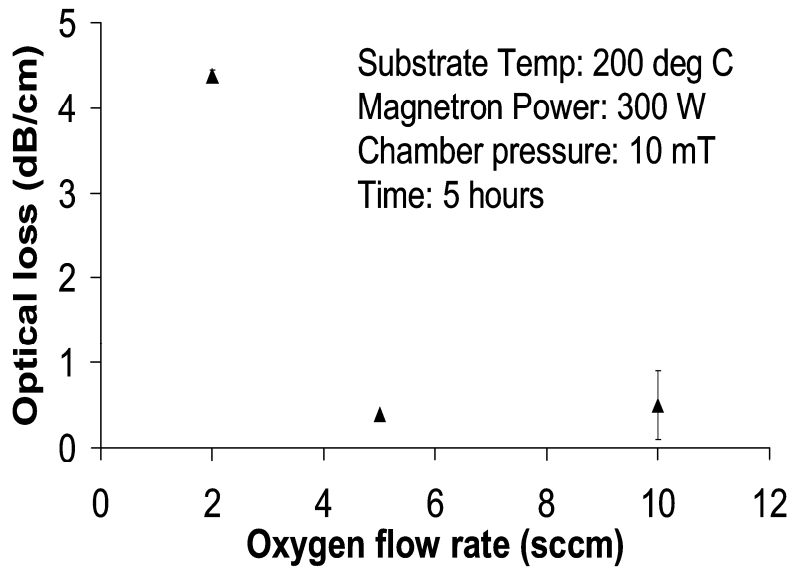


Figure 4 Optical loss at 633nm vs. oxygen flow rate for sputtered Er:Ta₂O₅ film

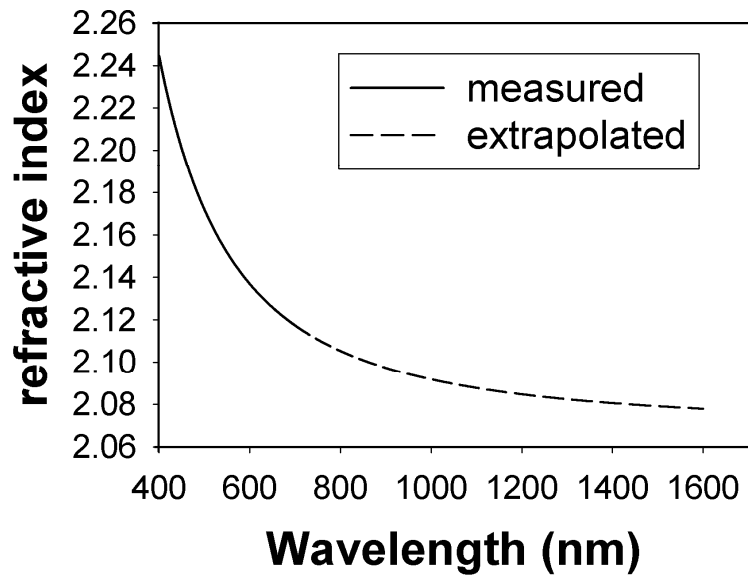


Figure 5 Refractive index of sputtered Er:Ta₂O₅ film determined by ellipsometry in the visible wavelength region

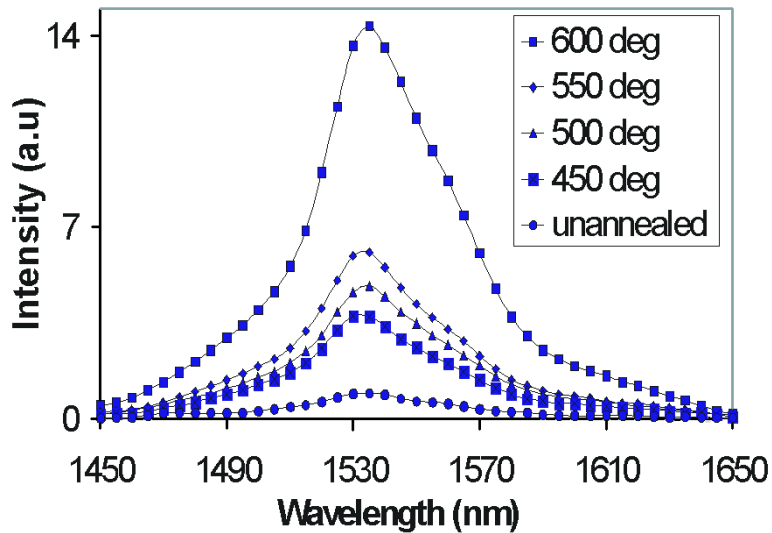


Figure 6 Photoluminescence spectra for unannealed and annealed Er:Ta₂O₅ films. The spectrum peaks at 1534nm. The annealing temperatures used were 450, 500, 550 & 600 °C, respectively (colour online).

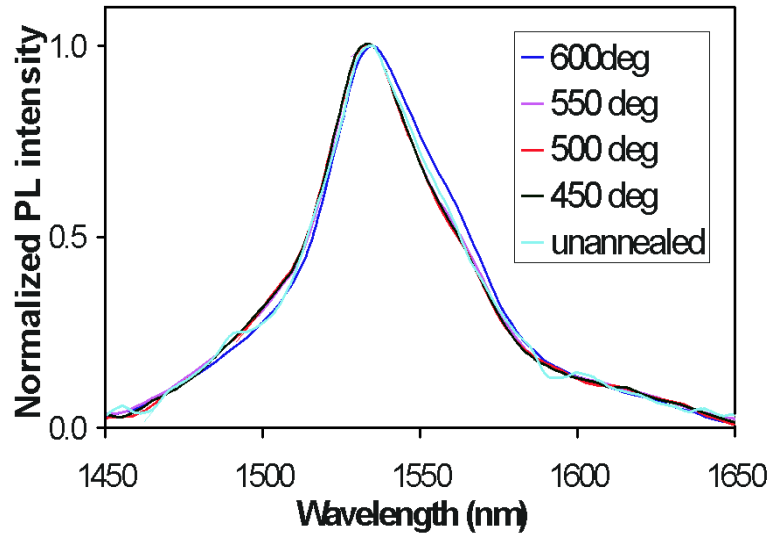


Figure 7 Normalized photoluminescence spectra for annealed and unannealed Er:Ta₂O₅ films peaking at 1534nm. The annealing temperatures used were 450, 500, 500 and 600 °C respectively. Bandwidth remains constant at 50nm with annealing temperature (colour online).

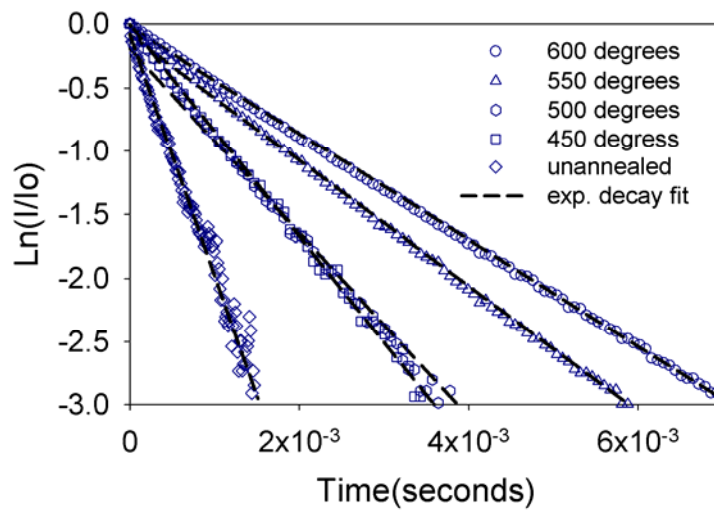


Figure 8 Comparison of luminescence decay of annealed and unannealed Er:Ta₂O₅ films. Ln(I/I₀) is the normalized intensity. A lifetime of 2.4ms was calculated for sample annealed at 600 °C (color online)