Evaluation Of Neodymium Doped Fluoride Glass Films Deposited By Pulsed Laser Deposition


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Abstract:

Pulsed laser deposition (PLD) has been evaluated as a technique for the realisation of neodymium doped fluoride glass waveguides. In contrast to other high energy techniques such as sputtering and molecular beam epitaxy, pulsed laser deposition appears to reliably reproduce the bulk stoichiometry of the doped glass in thin film form. However, characteristically for this deposition technique, the film topography is dominated by micron size particulates generated during fabrication. Film uniformity appears to be improved by depositing the film with a laser beam fluence close to the ablation threshold of the material. Unfortunately, this is at the cost of a significantly reduced film thickness which ultimately limits the usefulness of PLD for depositing waveguides from this material.

Introduction.

The development of the erbium doped fibre amplifier (EDFA) has discouraged the telecommunications industry from operating at the second communications window around 1300nm to the third window at 1550nm. Both wavelengths represent minima in the losses of standard telecommunications fibre due to hydroxyl absorption overtones in established silica-based fibre. However, the second window corresponds to the wavelength range of zero dispersion in which signals propagate with minimum distortion. Although more attractive for data transfer, industry has shifted emphasis away from propagation at 1300nm in favour of the third telecommunications window where the advantages of direct light amplification by the EDFA provide significant cost reductions. The FAST project (Fluoroaluminate Amplifier for the Second Telecommunications window) has concentrated on the development of a
neodymium doped fibre amplifier. Optimisation of the multi-component glass host has led to peak fluorescence and maximum gain at the zero dispersion wavelength of the conventional fibre network at 1317nm. Gain in excess of 30dB is expected with an inexpensive 100mW pump diode operating at 800nm. Due to the strong absorption of 170dB/m/1000ppm at the pump wavelength, fibre lengths of the order of centimetres, compared to several metres for praeosodymium hosts, are required. Such a short device length permits planar realisation not possible with other rare earth amplifiers.

Pulsed laser deposition (PLD) has been established as a method of producing crystalline and glass waveguides with the required complex composition. The report begins with a brief overview of the importance of PLD relative to other deposition techniques with regard to the reproduction of glass stoichiometry. The pulsed laser deposition procedure is described and introduces the experimental analysis undertaken for evaluation of PLD as a technique for realisation of the planar device.

Waveguide deposition techniques & pulsed laser deposition (PLD)

Glass waveguide lasers and amplifiers have been demonstrated using many deposition techniques based on sputtering and vapour phase processes. In general, such approaches are limited by the different vapour pressures of the components of the material being deposited making reproduction of complex bulk glass stoichiometry difficult. Sputtering of neodymium doped glass with nine components has previously been demonstrated but fluorescence spectra have revealed unacceptable red-shifting of the peak wavelength. Both chemical vapour deposition and molecular beam epitaxy have been employed but require a multitude of starting materials and expensive equipment.

Sol-gel and liquid phase epitaxy have not yet been considered, but rare earth doped waveguides have been achieved using both methods. Ion exchange, chemical etching and spin coating of molten glass are also being investigated as possible techniques for planar amplifier fabrication but are outside the scope of the report.

In contrast, pulsed laser deposition (PLD) overcomes the problems of non-congruent evaporation. The target material to be deposited is typically illuminated by a focused excimer laser beam operating at 248nm (KrF) or 193nm (ArF). Strong absorption by the glass network at these wavelengths leads to heating rates sufficient to vapourise the material in a time significantly less than that required for non-stoichiometric evaporation. Unfortunately, the application of PLD is hampered by two limitations: particulate formation and narrow deposition areas. Since the first demonstration of PLD the removal of particulates has proved a goal for workers and several experimental methods exist for their removal including: plume manipulation, rotating vane velocity filters, off-axis deposition, molten targets and target surface treatment.

Three main mechanism are proposed for particulate formation. Spherical
micron size particles are produced by subsurface boiling and the similar process of liquid layer expulsion by shock recoil\textsuperscript{11}. The third process, exfoliation\textsuperscript{12} is caused by the dislodging of features on the ablated surface and leads to irregular particulate shapes.

**Deposition of PLD films**

Two sets of films were deposited using pulsed laser deposition. The first set of films were used to evaluate the feasibility of the technique for the reproduction of glass stoichiometry with a range of deposition conditions. Detailed characterisation of fluorescence lifetime, fluorescence spectrum, EDAX analysis and SEM study of surface topography was performed on the first set of films.

The second set of films was deposited with precedent given to reduction of particulates and improvement of surface topography in order to lower the losses for films being deposited in waveguide format.

A summary of the conditions under which each of the first set of films was deposited is given in Table 1 and the geometry of the deposition conditions shown in Figure 0-a.

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**Figure 0-a Diagram of chamber geometry for pulsed laser deposition from preforms of glass**
Table 1 Summary of deposition conditions for preliminary films

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Substrate material</th>
<th>Substrate temp. (°C)</th>
<th>Base pressure (mBar)</th>
<th>Gas type</th>
<th>Deposition time (mins)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dep 1.1</td>
<td>CG</td>
<td>27</td>
<td>8.9×10⁻⁵</td>
<td>Vacuum</td>
<td>10</td>
</tr>
<tr>
<td>Dep 1.2</td>
<td>CG</td>
<td>290</td>
<td>3.7×10⁻⁵</td>
<td>Vacuum</td>
<td>10</td>
</tr>
<tr>
<td>Dep 1.3</td>
<td>CG</td>
<td>&gt;600</td>
<td>-</td>
<td>Oxygen</td>
<td>10</td>
</tr>
<tr>
<td>Dep 1.4</td>
<td>MS</td>
<td>&gt;600</td>
<td>-</td>
<td>Oxygen</td>
<td>10</td>
</tr>
</tbody>
</table>

† CG: Corning Glass Bariumaluminate-boro-silicate glass type.  
MS: Conventional washed microscope slide.

In order to reduce the number of particulates, the second set of films was deposited close to the ablation threshold of the material (estimated to be of the order of 1J/cm²) at temperatures between ambient room temperature and close to the glass transition temperature of the glass (430°C). Two substrate materials were used: the standard CG glass type used for the initial investigation and a modified fluoride glass material chosen for its matched material parameters and lower refractive index for use as a waveguiding substrate. A laser repetition rate of 10Hz was used throughout with a 20ns pulse duration and a target to substrate distance of 5cm.

Table 2 Summary of deposition conditions for reduced particulate films

<table>
<thead>
<tr>
<th>Substrate number</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
<th>11</th>
<th>12</th>
<th>13</th>
</tr>
</thead>
<tbody>
<tr>
<td>Excimer pulse energy (mJ)</td>
<td>280</td>
<td>280</td>
<td>210</td>
<td>400</td>
<td>150</td>
<td>263</td>
<td>263</td>
<td>263</td>
<td>263</td>
<td>350</td>
<td>350</td>
<td>395</td>
<td>395</td>
</tr>
<tr>
<td>Spot size (mm x mm)</td>
<td>6x2</td>
<td>6x2</td>
<td>6x2</td>
<td>5x2</td>
<td>6x2</td>
<td>8x3</td>
<td>8x3</td>
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<td>8x3</td>
<td>8x3</td>
<td>8x3</td>
<td>8x3</td>
</tr>
<tr>
<td>Energy density (J/cm²)</td>
<td>2.3</td>
<td>2.3</td>
<td>1.8</td>
<td>4.0</td>
<td>1.2</td>
<td>5</td>
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<td>1.1</td>
<td>1.1</td>
<td>1.5</td>
<td>1.5</td>
<td>1.6</td>
</tr>
<tr>
<td>Substrate temperature (°C)</td>
<td>250</td>
<td>20</td>
<td>400</td>
<td>20</td>
<td>400</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>400</td>
<td>400</td>
<td>400</td>
<td>40</td>
<td>20</td>
</tr>
<tr>
<td>(N₂ pressure) (10⁻² mBar)</td>
<td>5</td>
<td>5</td>
<td>200</td>
<td>10</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>5</td>
<td>4</td>
<td>4</td>
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<td>4</td>
</tr>
<tr>
<td>Deposition time (mins)</td>
<td>50</td>
<td>40</td>
<td>5</td>
<td>3</td>
<td>37</td>
<td>25</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>10</td>
<td>60</td>
<td>3</td>
<td>3</td>
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<tr>
<td>Comments†</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>P</td>
<td>NF</td>
<td>NF</td>
<td>NF</td>
<td>NF</td>
<td>NF</td>
<td>NF</td>
<td>NF</td>
<td>NF</td>
</tr>
</tbody>
</table>

† P: Particulates observed  
P: Thin film deposited (30nm)  
NF: No film deposited

**Analysis techniques**

Several techniques existed for investigating the quality and stoichiometry of the initial set of deposited films. As noted in the introduction, the composition of the glass was tailored to achieve maximum fluorescence and gain at 1317nm. Hence, the peak wavelength of fluorescence should correspond to that of the
Target and substrate were mounted in the chamber as shown, with a carbon dioxide laser or halogen lamps used for substrate heating as detailed. The chamber was evacuated and, depending on the choice of conditions, a back pressure of gas applied when a vacuum of the order of $10^{-4}$ mbar was achieved. To prevent thermal shock, the laser beam (operating at 248nm) was rastered over the preform surface by rotating the target as shown. Depositions were performed for 10 mins, after which the films were noted to be 'opaque' and removed from the chamber. Typical excimer energy density employed during the trials was 3.5 J/cm$^2$. 
Figure 0-a. Apparatus for determining fluorescence spectra of bulk and PLD deposited thin film samples

Results & Discussion

Comparison of the fluorescence spectra revealed a similar shape for both the 1050 and 1317nm transitions for the bulk and thin film samples except for the sample dep 1.4 deposited on a microscope slide at high temperature. In this case, the fluorescence spectra had been red-shifted by 9 ± 2nm for the 1317nm transition and 5 ± 2nm for the 1050nm fluorescence. The full-width half maximum (FWHM) of the fluorescence peaks provided a convenient method of comparing the shape of the spectra from bulk and thin film samples. The bulk glasses correlated well with each other: FWHM = 30 ± 1 nm. Similarly samples dep 1.1, 1.2 and 1.3 also showed FWHM consistent with the bulk glass. In contrast, as for the peak wavelength position, sample dep 1.4 showed a considerably reduced peak width at 1050nm. For the 1317nm transition, differences in FWHM were less discernible for dep 1.4 due to the difference in the shape of the spectra with a more prominent second feature appearing at approximately 1350nm. Since, for the majority of depositions, the shapes of the spectra and the values of the FWHM were very similar, it would appear that the changes responsible for the differences in sample dep 1.4 are occurring at the substrate, rather than in the ablated plume.
bulk glass. Two samples of bulk glass were used: 'bulk 1' an 'as provided' polished sample used for previous absorption measurements and 'bulk 2' which was a target remaining from the deposition of the films. Significant compositional changes in the glass were revealed by differences in the shape of the fluorescence curves at 1050 and 1317nm and a shift in the wavelength of peak fluorescence relative to the bulk. The lifetime of the upper level excited state in neodymium is again dependent on the host material and the concentration of neodymium via concentration quenching. By pumping the films with a square wave modulated pump beam produced by the chopper in the system, one was able to observe the decay of the $^4F_{3/2}$ excited state for comparison between bulk and thin film values.

Establishing the film composition was only one of the aspects to be considered in evaluating the PLD process since film uniformity is also essential in the production of low loss waveguides. Microscopic analysis of the films revealed particulate formation as described in section 2. Scanning electron microscopy (SEM) exhibits an increased depth of field relative to optical techniques which provided a convenient method of examining film topography. In conjunction, energy dispersive analysis of x-rays (EDAX) was available providing qualitative and semi-quantitative elemental analysis of the films produced.

**Fluorescence spectra**

*Experimental*

Fluorescence spectra were obtained using the standard configuration shown in Figure 0-a. The pump diode was operated at a maximum power of 35mW and a temperature of 16°C. Peak intensity occurred at 814nm but with a sufficiently broad emission to give good pumping of the 800nm neodymium transition in the glass. System design was simplified since absolute emission spectra were not required for comparison of the bulk and thin film samples. An InGaAs photodetector was used for detection of fluorescent signals in the region 1000-1400nm which was sufficient to compare the 1050nm and 1320nm transitions from the $^4F_{3/2}$ energy level of neodymium. The thin film samples were illuminated at grazing incidence to minimise the degree of pump light detected by the photodetector.
Figure 0-c. Photodetector output as a function of wavelength for bulk and thin film samples (1300-1400nm)

![Graph showing Photodetector output as a function of wavelength for bulk and thin film samples (1300-1400nm).](image)

Figure 0-d Photodetector output as a function of wavelength for bulk and thin film samples (1000-1100nm)

![Graph showing Photodetector output as a function of wavelength for bulk and thin film samples (1000-1100nm).](image)
Table 1 FWHM for thin film and bulk doped glass samples.

<table>
<thead>
<tr>
<th>Sample</th>
<th>1050nm FWHM (nm)</th>
<th>peak 1320nm FWHM (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk sample 1</td>
<td>30 ± 1</td>
<td>114 ± 2</td>
</tr>
<tr>
<td>Bulk sample 2</td>
<td>30 ± 1</td>
<td>109 ± 2</td>
</tr>
<tr>
<td>Dep 1.1</td>
<td>32 ± 1</td>
<td>117 ± 3</td>
</tr>
<tr>
<td>Dep 1.2</td>
<td>31 ± 1</td>
<td>119 ± 3</td>
</tr>
<tr>
<td>Dep 1.3</td>
<td>32 ± 1</td>
<td>115 ± 2</td>
</tr>
<tr>
<td>Dep 1.4</td>
<td>24 ± 1</td>
<td>114 ± 3</td>
</tr>
</tbody>
</table>

Figure 0-b. Photodetector output as a function of wavelength for bulk and thin film samples (1000-1400nm)
Results & Discussion

A typical digitised trace showing the temporal evolution of the fluorescent signal detected by the photodetector is shown in Figure 0-b. For a simple slow decay fluorescence signal, one would expect that the intensity on the photodetector would vary according to the expression:

\[ I = I_0 \exp\left(-\frac{t}{\tau_{\text{slow}}}\right) \]  

(Equation. 1)

By fitting the trailing edge of the intensity curve to this expression, one can determine the coefficient \( \tau_{\text{slow}} \) with an associated error incorporated within the uncertainty in the fitting parameter. Figure 0-c summarises the lifetime for each of the deposited films. Errors have been determined from the uncertainty associated with fitting to equation 1. No information on the fast decay component of the lifetime was possible within the resolution of the experiments as determined from a log-linear plot.

Figure 0-b. Typical digitised trace of photodetector output as a function of time for determining fluorescence lifetime of the 4F3/2 level of neodymium

![Digitised oscilloscope trace: photodetector output as a function of time for fluorescence lifetime determination](image)
**Fluorescence lifetime**

*Experimental*

Similar equipment to that used for characterising the fluorescence spectra of the samples was also applicable to determining the fluorescent lifetime of the $^4F_{3/2}$ level in the samples since the radiative transitions at 867, 1050 and 1317 nm all emanate from the $^4F_{3/2}$ level. Phase sensitive measurements used when producing fluorescence spectra from the samples, were not possible when measuring the fluorescence lifetime. This reduced the minimum signal to noise ratio which could be tolerated. Fortunately, the fluorescent signal available was increased by removing the monochromater and using all three radiative signals. One should be able to determine two contributions to the fluorescence decay curve if high resolution modulation is achievable. A fast decay component (FDC) is caused by cross relaxation and concentration quenching between neodymium ions. The slow decay component (SDC) is characteristic of the neodymium metastable energy level. If plotted on a log-linear scale, the SDC part of the curve is found to be purely linear due to the pure exponential decay whilst the FDC manifests as an increase in the magnitude of the gradient i.e. a faster decay. Equipment resolution was insufficient to resolve any fast decay component typically of the tens of microseconds but an acousto-optic modulator is available for future investigations.

**Figure 0-a. Apparatus for determining fluorescence spectra of bulk and PLD deposited thin film samples**
SEM and EDAX

Energy dispersive analysis of x-rays (EDAX) provides both qualitative and limited quantitative data about material composition using the same electron beam configuration incorporated in the scanning electron microscope (SEM). When the material surface is bombarded with energetic electrons, the excitation of electrons within the constituent atoms leads to the generation of x-rays characteristic of the energy separation of the atomic energy levels. By examining the characteristic x-ray peaks for their energy and the number of counts recorded, one can determine both qualitative and quantitative information on the elemental composition.
Figure 0-c. Comparison of fluorescence lifetimes of the $^{4}\text{F}_{3/2}$ excited state for bulk and thin film samples prepared by PLD.

Figure 0-c illustrates the differences in the slow decay rate of the $^{4}\text{F}_{3/2}$. As expected from the fluorescence spectra, sample dep 1.4 shows a significantly reduced lifetime relative to the other samples, but there is also considerable variation between the lifetimes in the other samples and even between the two bulk glasses.

In general, with a fluoride glass, the maximum phonon energy (MPE) is reduced so that non-radiative decay is suppressed. However, the loss of fluorine will make the glass less ionic and so raise the MPE of the material. In this situation, more non-radiative transitions will lower the radiative lifetime, i.e. it will decay more rapidly. Thus, in the case of the sample dep 1.4, with markedly different spectra, loss of fluorine or at least compositional change would be a likely explanation for the lower lifetime since sample dep 1.3, deposited under the same conditions as dep 1.4 but on the corning substrate shows no significant red-shifting.

In the case of the samples dep 1.1 to dep 1.3 there appears to be a decrease in lifetime with deposition number. One source of lifetime reduction is via concentration quenching but, as explained, this is evident in the first few microseconds of the decay. It is unlikely that the packing of the particles is a factor in determining the lifetime of the upper level. Instead, it is more likely that some compositional variation has occurred but is not detectable within the limits of the fluorescence and EDAX resolution. One possible explanation may be an evolution in lifetime with temperature. Such a dependence, caused by the substrate temperature increase in going from 1.1 to 1.3, may be caused by the quenching rate of the glass as it cooled on the substrates.
found with increasing deposition number.

Film dep 1.4. deposited on a microscope slide at high temperature showed a more continuous structure compared to that of the other films. The structure was consistent with a reflowing of the glass perhaps involving interaction by diffusion processes between the deposited glass and the substrate.

Figure 0-d Sample dep 1.3 cross-sectional view

Figure 0-e Sample dep 1.3 plan view

Figure 0-f Sample dep 1.4 cross-sectional view

Figure 0-g Sample dep 1.4 cross-sectional view

Sodium diffusion may have occurred at such elevated temperatures, but since the glass already contained sodium this was difficult to determine from the EDAX results presented in Figure 0-j. Sample dep 1.4 showed considerable cracking but with good adhesion. Such observations agree with some form of
The SEM photographs of Figure 0-a to Figure 0-c show particularly well the structure of the film dep 1.1 deposited at room temperature and under vacuum. From Figure 0-c, one can see that, rather than being continuous, the film is composed of a layer of closely packed particulates. Particle size is typically of the order of ten microns or less with spherical or ellipsoid geometry. From section 2, such particulate formation would be consistent with both subsurface boiling and shock recoil but such processes are normally confined to metallic or conducting targets\textsuperscript{13}. From the cross-sectional view of Figure 0-b, there is some evidence of amorphous particle formation. Furthermore, the surface of the ablated target showed structure which may possibly be susceptible to ‘splashing’ by exfoliation. Unfortunately, the high degree of splashing masks any background film produced by the true ablation process. Scratch tests have also demonstrated poor adhesion of the film to the substrate.

As the temperature of the substrate was increased in moving from samples 1.1 through to 1.3, an evolution in particle distribution and shape was seen. In general, the particulate distribution was reduced with increasing temperature with evidence of reflowing of the glass found in the re-melting of the glass to the substrates (seen in the cross-sectional view of Figure 0-d). Such reflowing of the glass was consistent with the improvement in film glass adhesion
Composition of bulk and thin film glass samples prepared by PLD determined by EDAX

Quantitative analysis of the glass samples using the EDAX technique was limited to a typical error of 5% on the determined weight percentage. In particular distinguishing Y, P and Sr was difficult due to the closeness of their emitted X-ray energies. A comparison of atomic percentage compositions determined using EDAX for both bulk and thin film samples is given in Figure 0-j. EDAX measurements were not able to produce quantitative data on the percentage of fluorine in the glass, the most common element to be lost during laser ablation.

Figure 0-h. Molar percentages of elements present in bulk glass 1 for different specimen areas
diffusion and fusion bonding processes occurring between film and substrate. Under these conditions, cooling of the substrate from high temperature would lead to cracking due to the poor thermal expansion matching of substrate and film.
In the case of sample dep 1.3, EDAX spectra were taken from both the centre of a droplet and in the surrounding area as indicated in Figure 0-e. The analysis technique was limited to a generation volume of the order of a micron to the extent that compositional data was only valid for features of dimension greater than this. Analysis revealed that the dark material in the photograph produced a signal consistent with the substrate used but did not discount the possibility of a thin, submicron film of the glass in addition to the surface spheroids.

**Reduction of particulates**

PLD has demonstrated its effectiveness in reproducing the complex stoichiometry of the bulk glass but, as typical of the technique, film topography was poor. This is particularly important for the use of PLD in optical waveguide applications since this raises the losses of the device to unacceptable levels over 10dB/cm as demonstrated by Alfonso et Al\textsuperscript{14}. The task lies in the removal of particulates to the point where analysis of background film may be disseminated from the influence of the material in the fragments. As for dielectrics where the degree of splashing was found to be reduced by decreasing the illuminating beam fluence\textsuperscript{15}. Experiments performed close to the laser threshold at \( = 1J/cm^2 \) reduced the number of particulates significantly but indicated that any background film deposition rate was slow. Deposition rate was improved by increasing the beam size whilst maintaining the beam fluence. A 30nm film was generated on both a Corning glass substrate and a ‘cladding’ glass substrate for the conditions indicated in . Fluorescence from the transition at 867nm was only detected from the ‘cladding’ film but this was determined to be due to contamination of the glass by neodymium during their manufacture.
Figure 0-1 Molar percentages of elements present in bulk glass 2 for different specimen areas

Figure 0-j Molar percentages of elements present in thin film samples prepared by PLD as compared to the theoretical composition

An indication of the relative errors associated with the molar percentages of elements in the films was produced from the computer algorithm uncertainty on the number of counts assigned to each characteristic peak.
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Conclusion

Pulsed laser deposition (PLD) has been evaluated as a technique for the realisation of neodymium doped fluoride glass waveguides. In contrast to other high energy techniques such as sputtering and molecular beam epitaxy, pulsed laser deposition appears to reliably reproduce the bulk stoichiometry of the doped glass in thin film form. However, characteristically for this deposition technique, the film topography is dominated by micron size particulates generated during fabrication. Film uniformity appears to be improved by depositing the film with a laser beam fluence close to the ablation threshold of the material. Unfortunately, this is at the cost of a significantly reduced film thickness which ultimately limits the usefulness of PLD for depositing waveguides from this material.

Acknowledgements

We are grateful for the opportunity of using the European Ultraviolet laser facility and would like to thank everyone at the Foundation for Research and Technology - Hellas for their support, advice and fruitful discussions.
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