

# Optical Amplifiers and Lasers in Infrared Fibres

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## ABSTRACT

We are currently investigating two infrared glasses for active applications. Gallium lanthanum sulphide (GLS) glass is investigated as a potential host material for rare-earth doped mid-infrared fibre lasers. We have fabricated gallium lanthanum sulphide glass by melt quenching and drawn it into fibres using the rod-in-tube technique. Fluoroaluminate glasses (ALF) are being prepared in planar form by spin coating and clad waveguides have been achieved. The quality of waveguides from both these materials is gradually being improved as methods to eliminate transition metals and other impurities, understand crystallization and reduce the imperfections at the core/clad interface are developed. Although initially motivated by the demand for a practical 1310 nm amplifier, interest has now extended further into the infrared. We describe recent progress in these glasses, their properties and applications.

**Keywords:** Infrared, Optical Fibre, Chalcogenide, Fluoride, Glass, Laser, Amplifier

## 1. INTRODUCTION

Silica glass fibre is clearly the ultimate optical waveguide, with millions of kilometres now installed and an infrastructure that is firmly established. Silica, however, is not an infrared material; transmission over long distances is limited to about 2 microns, beyond which multiphonon absorptions result in a glass which is opaque. For this reason, as far back as the early 1960's, infrared transmitting glasses, not based on traditional oxide glasses, have been investigated as passive waveguides for infrared transmission<sup>1</sup>. In the 1980's, following the failure of the fluoride glass ZBLAN to meet theoretical predications of a lower loss than silica<sup>2</sup>, a turning point was reached and activity shifted to active applications of these glasses.

Silica based lasers and amplifiers are limited to only a fraction of the possible transitions that can be achieved through rare earth doping of the glass. The success of the erbium-doped amplifier operating at 1.5 microns is an exception. The majority of other wavelengths are either quenched through non-radiative vibrations or for the case of emission above 2 microns, simply cannot be transmitted. An optical fibre amplifier operating at 1.3 microns was the original motivation for turning to new materials. The same property that reduced multiphonon absorptions, a low phonon energy, also extends the transmission range further into the infrared. Thus at the same time, glasses which provide new opportunities for infrared passive waveguides also open up new active applications<sup>3</sup>.

Glasses which provide a 1.3 micron amplifier include both the fluoride and sulphide families. Since 1991 we have studied both and while interest remains in achieving a practical 1.3 micron optical amplifier, the breadth and direction of our work has expanded to new applications. In doing so, new fabrication methods have also developed in parallel. In this paper, we describe our current work on lasers and amplifiers in these two types of infrared materials.

## 2. GLASS PROPERTIES

Application of a glass as the host for an active optical fibre device is driven by the glass properties. The stability, inertness and low intrinsic loss of silica have helped to establish its use in telecommunications system. For active applications such as lasers and amplifiers, again the glass properties are of key importance. In contrast to applications for long distance communications with operation over kilometre distances, active waveguide device lengths range from a few metres to as short as a few millimetres. As a result, loss tolerances drop by several orders of magnitude and other glass properties such as those described here become significant and can be exploited.

### 2.1 Fluoride Glasses

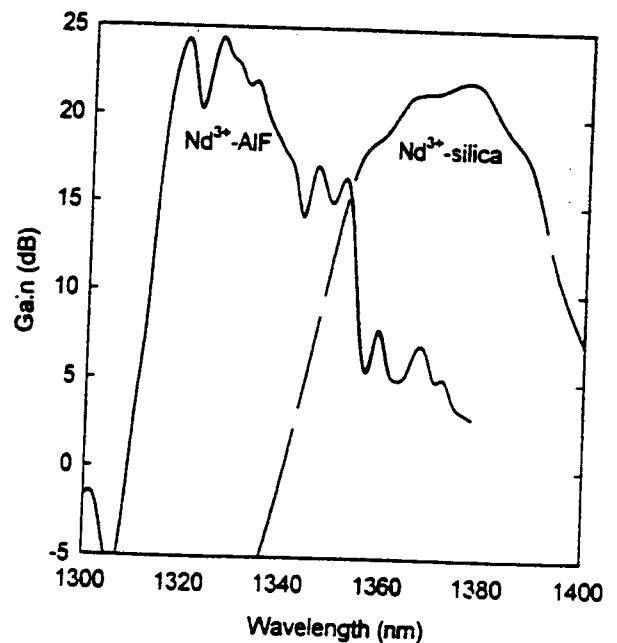
The key requirement of glasses in our pursuit of a  $\text{Nd}^{3+}$ -doped 1.3 micron amplifier is the reduction of excited state absorption (ESA). A strong signal ESA severely reduces the amplifier efficiency and shifts the gain peak to longer wavelengths such that the overlap with the telecom window becomes relatively poor<sup>4</sup>. Using the results of absorption measurements coupled with a Judd-Ofelt analysis, a criterion to determine the best possible glass composition has been established. For AIF glasses, the key property is the ionicity which is inversely proportional to the refractive index. The more ionic the glass, the smaller the ESA effect. Sample AIF glass compositions are given in table 1. Key properties for these glasses are presented in table 2.

**Table 1: Composition and Refractive Index for AIF Glasses**

Sample	Composition (mole %)	$n_d$
AIF 70	37AIF <sub>3</sub> :12MgF <sub>2</sub> :15CaF <sub>2</sub> :9SrF <sub>2</sub> :6BaF <sub>2</sub> :14YF <sub>3</sub> :6NaPO <sub>3</sub>	1.432
AIF 117	39AIF <sub>3</sub> :7MgF <sub>2</sub> :30CaF <sub>2</sub> :7SrF <sub>2</sub> :7BaF <sub>2</sub> :9LiF	1.402
AIF 123	39AIF <sub>3</sub> :6MgF <sub>2</sub> :22CaF <sub>2</sub> :6SrF <sub>2</sub> :6BaF <sub>2</sub> :10LiF:10NaF	1.397

Based on refractive index measurements, this novel family of non-toxic AIF glasses have been identified as the most promising hosts for a  $\text{Nd}^{3+}$ -doped glass. Through compositional modifications, refractive indices and thus ESA have been reduced. Figure 1 shows the results of gain measurements around 1.3 microns clearly demonstrating the shift towards shorter wavelengths obtained through the use of AIF glass<sup>5</sup>.

Other properties of significance include the thermal characteristics. Like other fluorides, crystallization is a concern<sup>2</sup>. By increasing the temperature gap between the fibre drawing temperature and the temperature for onset of crystallization, fibre loss due to crystallization is minimized. For both fibre drawing and spin coating applications, knowledge of the glass viscosity as a function of temperature is important. In our own experience, the relatively high thermal expansion coefficient of this family of glasses again benefits from characterization of thermal behaviour. These thermal properties have been reviewed in reference 6.



**Figure 1.  $\text{Nd}^{3+}$ -doped AIF and silica glass operating within the standard 2<sup>nd</sup> telecommunications window.**

## 2.2 Sulphide Glasses

We have worked primarily with the binary composition  $70\text{Ga}_2\text{S}_3:30\text{La}_2\text{S}_3$  with substitution of the  $\text{La}_2\text{S}_3$  with up to 30%  $\text{La}_2\text{O}_3$  to improve thermal stability. As a passive fibre GLS offers a transmission window from the visible region of the spectrum to 5 microns. This window, unlike in other sulphide glasses, is not dominated by a large sulphur-hydrogen ( $\text{SH}^-$ ) absorption at 4.03 microns. GLS glass is also non-toxic as it does not contain the usual arsenic based glass former. Moreover, its temperature stability is enhanced, with a melt temperature around  $1000^\circ\text{C}$ , well above other candidates<sup>7</sup>. The challenge of drawing fibre without crystallization means that the thermal properties of the glass are most important. Figure 2 shows a typical differential thermal analysis result and the proximity of the fibre drawing temperature to the crystallization peak. Key properties are listed in table 2.

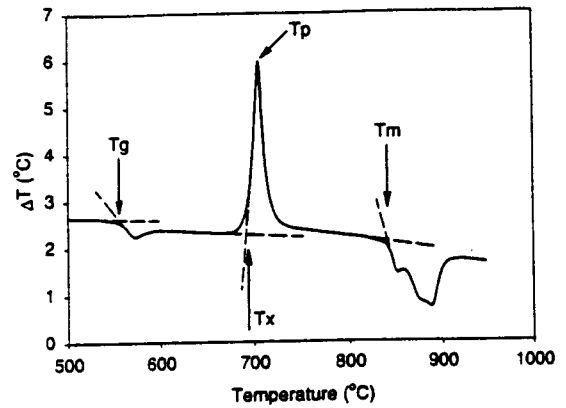


Figure 2. DTA trace for GLS glass indicating glass transition ( $T_g$ ), onset and peak of crystallization ( $T_x$ ,  $T_p$ ) and onset of melting ( $T_m$ )

Table 2: Key Properties of Fluoride and Sulphide Glasses and Comparison with Silica Glass

	Silica	Fluoride	Sulphide
Composition	$\text{SiO}_2$	AlF	GLS
<b>Thermal</b>			
Glass Transition ( $^\circ\text{C}$ )	1175	430	560
Melting Temperature ( $^\circ\text{C}$ )	>2200	692	842
Specific Heat (cal/g- $^\circ\text{C}$ )	0.179	0.103	0.109**
<b>Mechanical</b>			
Expansion Coefficient ( $^\circ\text{C}^{-1} \times 10^{-6}$ )	0.55	17.0	10.6
Density (g/cm <sup>3</sup> )	2.20	3.6	4.04
Poisson's Ratio	0.17	0.30*	0.24
Elastic moduli (Gpa): Youngs	73.1	58.3*	59.0
Shear	31.2	20.5*	23.0
Bulk	36.7	47.7*	24.5
<b>Optical</b>			
Refractive Index at 0.589 $\mu\text{m}$	1.458	1.432	2.4833
Abbe Number	68	76	13.7
Zero Material Dispersion Wavelength	1.3	1.6*	n/a
Non-linear index ( $n_2$ ) (esu $\times 10^{-11}$ )	1	0.85*	300
Temp. coefficient of refractive index (dn/dT) ( $^\circ\text{C}^{-1}$ )	$+11.9 \times 10^{-6}$	$-14.75 \times 10^{-6}$ **	n/a
Approximate transmission range ( $\mu\text{m}$ )	0.16 - 2.0	0.22 - 4.0*	0.53 - 5.0

\*approximated by value for ZBLAN, \*\* approximated by value for  $\text{As}_2\text{S}_3$  glass

Current interest in this glass rests strongly on its application as an optically active material. Early work showed that it could be readily doped to high levels with any of the lanthanides simply by substitution. Unlike a silica glass host where rare earth ion clustering limits concentrations to a few hundred parts per million, we have prepared glasses with concentrations of hundreds of thousands of parts per million without clustering. It is possible to obtain a glass with even 40% by weight erbium sulphide. The ease of doping with rare earth ions combined with its infrared transparency make this glass an ideal candidate for infrared sources and lasers. We are now pursuing a variety of sources in the 3-5 micron region for a range of applications.

### 3. WAVEGUIDE FABRICATION

Waveguide fabrication in both the gallium lanthanum sulphide and fluoroaluminate glasses is not a straightforward task. Conventional fibre drawing techniques were initially attempted for both families of glasses. As our experience with these glasses broadened so did our range of fabrication possibilities. We are currently focussing on a spin coating technique for planar waveguides and a spin casting or rod in tube technique for fibre waveguides.

#### 3.1 Planar Waveguides

At temperatures above the liquidus temperature, several soft glass families, such as the fluorides, sulphides and phosphates, have viscosities comparable to those of polymeric spin coating solutions. Using techniques developed in-house, AlF glass has successfully been spin coated into a waveguide directly from its liquid phase. This glass, with a viscosity of 0.1 poise at 1000°C, is ideally suited for short planar devices fabricated in this way.

Nd-doped waveguides were fabricated using the apparatus shown in figure 3. A 25 mm diameter AlF glass substrate of refractive index 1.417 was preheated to its glass transition temperature of 430°C in an upper annealing furnace. In the lower furnace, a 30 g melt of Nd-doped AlF glass of refractive index 1.432 was heated to 1000°C. When the temperatures of the two furnaces had stabilised, a positioning system was used to transfer the molten glass between furnaces. Accurate control of the melt position resulted in the surface of the substrate being dipped a fraction of a millimetre into the molten glass. The computer was used to synchronise the withdrawal and spinning of the substrate up to a maximum speed of 6000 rpm. The as-deposited waveguides were then annealed in the upper furnace at 430°C for three hours, followed by a slow ramp down to room temperature at 30°C/hour<sup>8</sup>.

Scanning electron microscopy was used to examine the cross-section of the films. The waveguides exhibited excellent thickness uniformity (0.1µm/mm) over the central area of the substrate, with some curvature of the surface observed at the edges due to re-melting of the underlying substrate glass. The high quenching rates achieved by coating a thin layer of molten glass onto a much larger cooled substrate produced a crystal-free glass/waveguide interface, as seen in SEM micrographs presented in figure 4.

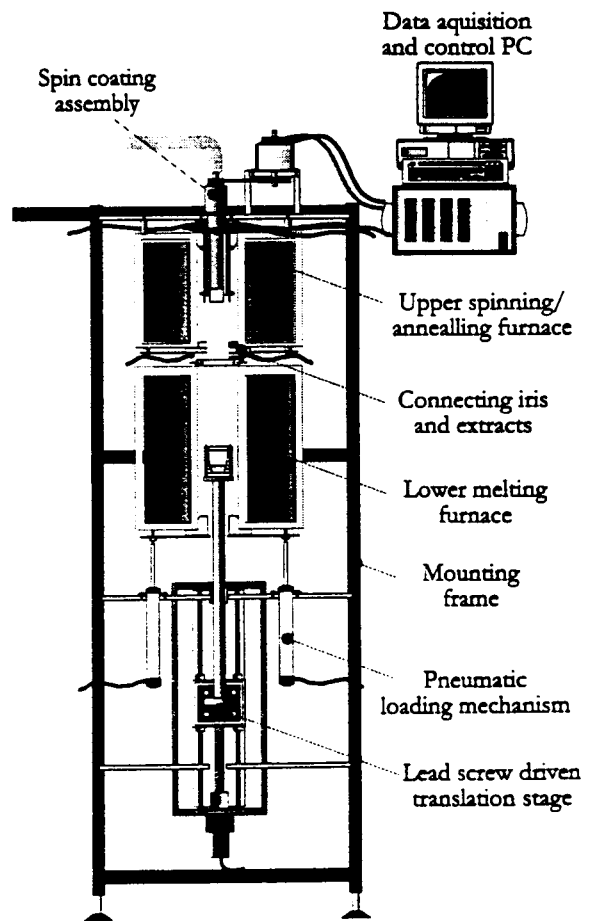


Figure 3. Apparatus for spin coating of waveguides using

### 3.2 Fibre Drawing

There are two basic methods of drawing fibre, from a preform or from a melt<sup>7</sup>. Both techniques have been used in practise for chalcogenide fibres but the preform method is most suited to GLS glasses. The high melt temperature, which provides an advantage for fibre stability, makes drawing these fibres at temperatures above the liquidus, ie crucible drawing, problematic.

Historically, the preforms have been prepared by a rod-in-tube method. Ingots of glass are melted in carbon boats in an argon atmosphere. These are polished into rods typically 3 mm in diameter and 110 mm long or tubes, 10 mm outer diameter, 3-4 mm inner diameter and 110 mm long. A double-rod-in-tube collapse to fibre results in a typical core-clad ratio of about 1:6, as shown in figure 5. Details of this procedure have been described in reference 9. We have achieved single mode fibre through a quadruple collapse. A single mode fibre with core diameter 1.8 microns, is shown in figure 6.

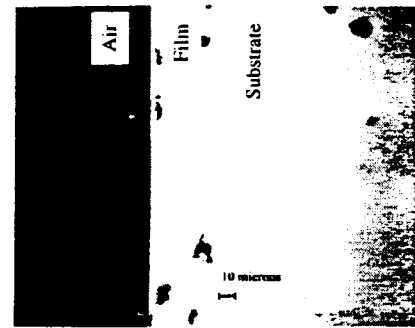


Figure 4. Cross sectional SEM micrograph of 40 micron thick planar waveguide structure obtained by spin coating.

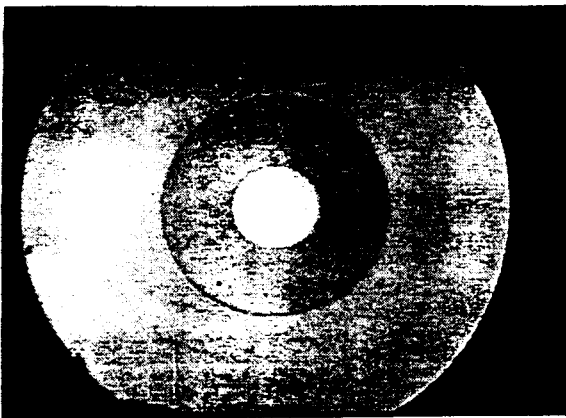


Figure 5. GLS fibre obtained by double collapse shown illuminated from above.

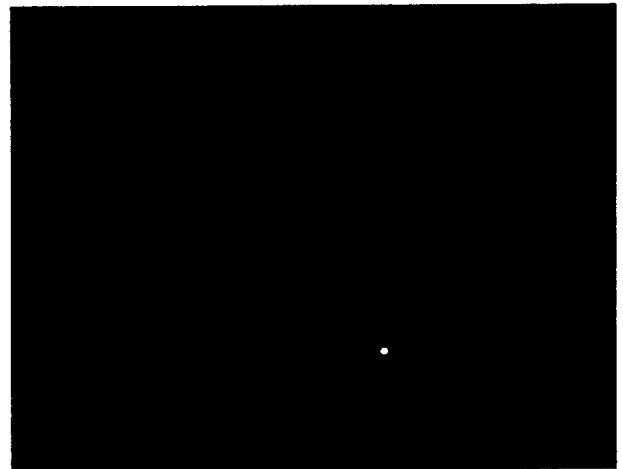


Figure 6. GLS fibre single mode at 1.5 microns shown guiding light in the core.

## 4. WAVEGUIDE PROPERTIES

As new materials are developed for optical waveguide applications, regardless of their key properties, the transmission loss is of primary concern. Whether the glass provides new laser transitions or high efficiency amplification, if loss requirements cannot be met, the material has little practical use.

### 4.1 Planar Waveguides

In order to measure the propagation losses, a 2 mW HeNe laser was end-coupled into a 20 micron thick parallel end-polished

sample. A silicon CCD camera was then used to image the scattered radiation from the waveguide surface. An exponential fit to the scattered intensity along the length of the waveguide gave a loss of  $\sim 0.5\text{dB/cm}$ . This was confirmed by direct measurement of the transmission through the guide. Such a loss figure is adequate for planar device applications and is expected to improve as higher purity raw materials are obtained.

#### 4.2 Fibre Waveguides

The fibre loss was measured using a standard cutback method with a Fourier transform infrared spectrometer, Perkin-Elmer System 2000 and a HgCdTe detector for  $1\mu\text{m}-10\mu\text{m}$ . This apparatus was adapted from that described in reference 2. The procedure works well for multimode fibre. However cladding modes guided along with core modes makes measurement difficult. This is a consequence of the high index which prevents the coating from stripping cladding modes. We are currently preparing apparatus which will allow single-mode fibre characterization.

A typical loss plot is shown in figure 7. We note that the loss minimum is around 4.0 microns as predicted by intrinsic loss calculations<sup>10</sup>. At the present time, loss levels are limited by OH<sup>-</sup>, transition metals and other impurities, crystallization and imperfections at the core/clad interface.

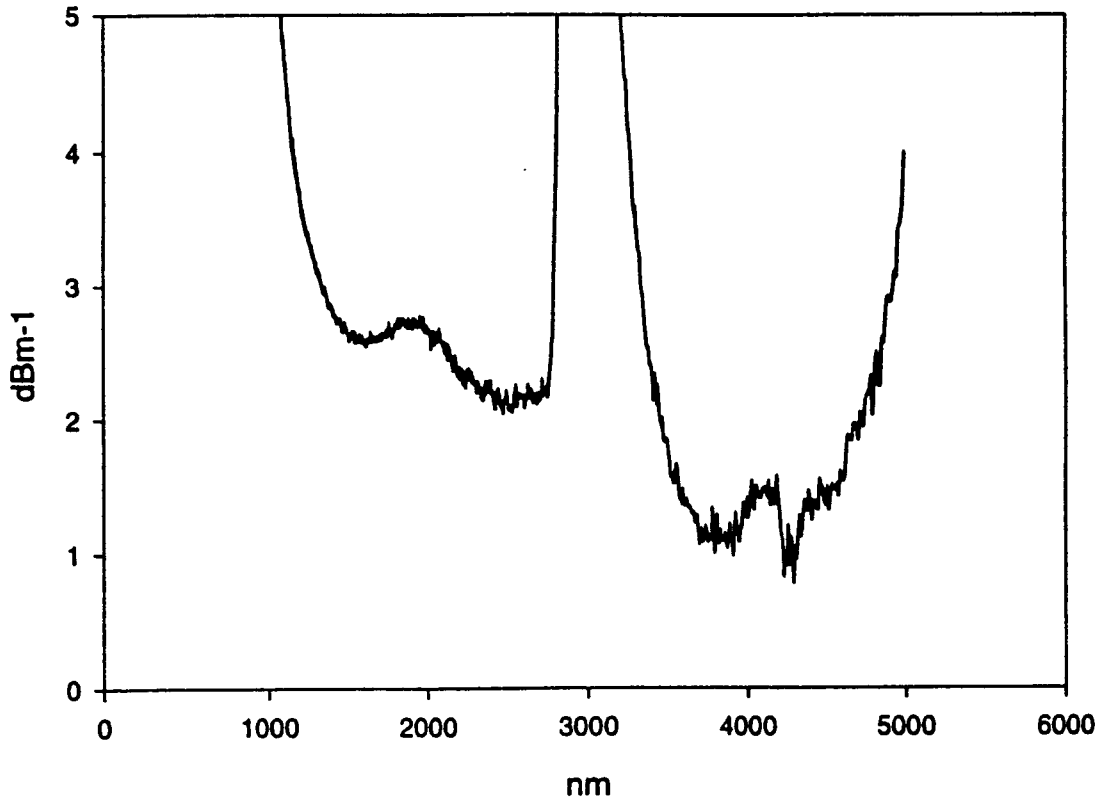


Figure 7. Transmission spectrum for GLS fibre.

### 5. APPLICATIONS

#### 5.1 Laser operation around 1050 nm

With  $\text{Nd}^{3+}$ -doped AIF, we have the potential for an efficient and inexpensive amplifier. It has a convenient pump wavelength at 800 nm where inexpensive laser diodes are commercially available. The strong absorption of  $\text{Nd}^{3+}$  at 800 nm ( $170\text{ dB/m/1000 ppm}$ ) and the strong solubility of the ion in AIF glass (up to  $4 \times 10^4\text{ ppm}$ ) suggest a short device of  $\sim 2\text{ cm}$  is practical.

A laser cavity was formed in planar AIF by the attachment of plane mirrors to the polished, parallel end faces of a 20 micron thick, 5 mm long waveguide. The chopped output from a Ti:sapphire laser operating around 800 nm was end-launched into the waveguide using a 50 mm focal length microscope objective. The input coupler was highly reflecting (HR) at 1050 nm, and

highly transmitting at 800 nm. A 3.5% output coupler at 1050 nm was used which was HR at 800 nm in order to double pass any unabsorbed pump.

Laser output was observed at several wavelengths between 1045 nm and 1065 nm. Laser peaks correspond to the positions of maximum reflectivity in the Fabry-Perot cavity formed between the surface of the mirrors and the back of their respective substrates. The maximum slope efficiency was measured to be 10% with respect to incident power for a corresponding threshold of 170 mW as shown in figure 8. This represents, we believe, the first demonstration of laser action in a fluoride glass waveguide. Lasing was also demonstrated in neodymium doped GLS glass and glass fibre at 1080 nm, representing the first reported laser action in a rare earth doped chalcogenide material. The strong thermal lensing in the bulk glass rules out the use of GLS glass as a bulk laser material. The effect was eliminated by using a fibre geometry showing the advantages and necessity of GLS glass fibre<sup>11</sup>.

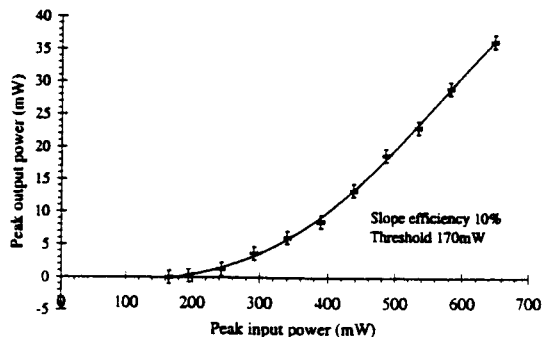


Figure 8. Output power at 1050 nm as a function of pump power at 800 nm for an Nd-doped AlF waveguide laser.

### 5.2 Sources for the 3-5 micron Region

Over the past four years, detailed spectroscopic measurements have been performed on rare earth doped GLS glasses. This has been fully described in reference 12. Absorption, fluorescence, and lifetime measurements have been performed for eleven lanthanide ions for wavelengths ranging from the visible to the mid-infrared with our focus on mid-infrared transitions in the 3 to 5 micron transmission window. The results of the measurements were used to study the multiphonon decay in GLS glass and to obtain important laser parameters such as the absorption and emission cross sections, branching ratios, and quantum efficiencies from the Judd-Ofelt theory, the Fuchtbauer-Ladenburg equation, and the McCumber theory. Twenty-one transitions with peak emission wavelengths longer than 2 microns were identified, seven of which have not been reported in a glass before. Some transitions overlap with the fundamental absorption bands of environmentally important gases such as the 3.4 microns (hydrocarbons) and 4.7 microns (carbon monoxide) praseodymium transitions and the 4.3 micron (carbon dioxide) dysprosium transitions, whereas the 3.8 micron thulium and the 3.9 micron holmium transitions coincide with an atmospheric transmission window. Table 3 describes the key transitions under investigation. Co-doping schemes such as thulium/terbium, praseodymium/ytterbium, and dysprosium/erbium which offer more favourable absorption bands through ion-ion energy transfer and thus allowing diode laser pumping are also being studied.

Table 3. Pump and emission wavelengths for rare-earth doped GLS

Rare Earth Ion	Pump Wavelengths (microns)	Emission Wavelength (microns)
Praseodymium	1.02, 1.57, 2.0	3.4, 4.7
Neodymium	0.815, 0.89, 1.65	5.1
Terbium	0.8, 2.0	4.8
Dysprosium	0.81, 0.91, 1.1, 1.3, 1.7	4.3
Holmium	0.65, 0.76, 0.9	3.9, 4.9
Erbium	0.66, 0.805	3.6, 4.5
Thulium	0.7, 0.8, 1.22	3.8

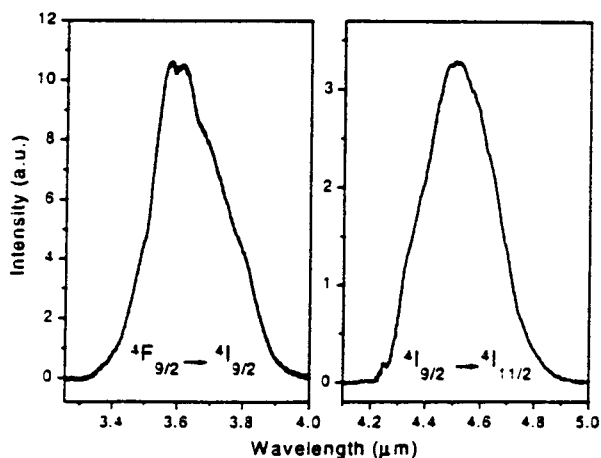


Figure 9. Emission in infrared from Er<sup>3+</sup>-doped GLS fibre pumped at 660 and 810 nm.

## 6. CONCLUSIONS

We are currently investigating two infrared glasses for active applications, GLS glass as a potential host material for rare-earth doped mid-infrared fibre lasers and AIF glass for application as a planar waveguide amplifier.

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