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**DESIGN AND FABRICATION OF Pr<sup>3+</sup>-DOPED  
FLUORIDE GLASS OPTICAL FIBRES  
FOR EFFICIENT 1.3  $\mu$ m AMPLIFIERS**

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## **Abstract**

Two novel glass compositions aimed at improving the quantum efficiency and gain of the 1.3  $\mu\text{m}$  praseodymium-doped fibre amplifier (PDFA) were developed and investigated. The two glasses were compositions based on cadmium halide and gallium fluoride. Both bulk glass samples and core-clad fibre structures were fabricated and studied. A variety of measurements have been carried out in order to assess the potential of these glasses as hosts for PDFA. The results were used to perform a detailed modelling of the projected amplifier characteristics. Both glasses are judged to be promising candidates for the second generation of 1.3  $\mu\text{m}$  PDFA's.

## 1. Introduction

Following the development of the erbium-doped optical fibre amplifier (EDFA) which successfully amplifies signals in the 1.5  $\mu\text{m}$  telecommunications window<sup>[1]</sup>, the search is now on for a similar device which would operate in the 1.3  $\mu\text{m}$  window of the terrestrial networks. The search has focused on praseodymium-doped fibre amplifiers (PDFA) based on low-phonon energy fluoride glasses<sup>[2]</sup>. Low phonon energy is a crucial requirement for a PDFA glass host, since the phonon energy of the host is the most important parameter determining the PDFA performance. Both the quantum efficiency and gain of a PDFA are proportional to the lifetime of the  $^4\text{G}_5$  metastable level which is dominated by non-radiative transitions<sup>[2]</sup> (see Figure 1). In a high purity low-doped glass non-radiative transitions are due almost entirely to multiphonon relaxations. The multiphonon decay rate is known to decrease exponentially with each additional phonon required to bridge the energy gap to the underlying level<sup>[3]</sup>; hence low phonon energy is indispensable for achieving an efficient, high gain PDFA. Prototype PDFA's based on ZBLAN have been extensively studied over the recent years. State-of-the-art small-signal amplifiers utilizing a single laser diode pump source provide gains of up to 23 dB<sup>[4]</sup>. Fully engineered power amplifiers, pumped by Nd:YLF lasers and yielding outputs of 70 mW, are now becoming commercially available<sup>[5,6]</sup>. These devices have been made possible by the solution of many of the engineering problems associated with high NA fluoride fibres. However, the fundamental problem of low quantum efficiency and gain has not been overcome. For this reason, new glass hosts are being studied with a view to improving the amplifier performance.

Two families of fluoride glasses possessing lower phonon energy than ZBLAN have lately attracted much attention as possible hosts for a second generation of PDFA's. These new glasses are based on cadmium halide and gallium fluoride. Cadmium halide glass promises much improved performance, but is highly susceptible to moisture and requires environmental protection. It is also relatively difficult to fabricate into low-loss fibre. Gallium fluoride is environmentally stable and requires little protection. Its fibre-drawing parameters are not far

removed from those for ZBLAN fibres, for which the technology is well established. In this paper we describe the performance of cadmium halide and gallium fluoride glasses in both bulk and fibre geometry, and discuss their projected amplifier characteristics and their prospects for realising an improved PDFA device.

## 2. Experimental methods

The cadmium halide family of glasses were based on the composition  $\text{CdX}_2:\text{BaX}_2:\text{NaX}$ , where X denotes either F or Cl<sup>[7]</sup>. The gallium fluoride glasses were  $\text{GaF}_3:\text{MF}_2:\text{YF}_3$ , where M stands for a divalent alkaline metal, and were similar in composition to indium fluoride-based glasses<sup>[8]</sup>. The glasses were prepared and melted in dry nitrogen atmosphere and cast into brass moulds. Preforms were manufactured by rotational casting, the typical size being 120 mm length and 10 mm diameter. Fibre drawing was carried out in an RF tower under dry nitrogen.

Fluorescence lifetime was measured using a Ti-Sapphire laser with an acousto-optic modulator and an InGaAs detector connected to a digital storage oscilloscope. Emission spectra were obtained using the same laser and an optical spectrum analyzer. Raman spectra were observed using a Jobin-Yvon Micro-Raman instrument in conjunction with an  $\text{Ar}^+$ -ion laser. Absorption spectra were obtained by a Perkin-Elmer spectrophotometer. Fibre loss was measured by standard cut-back techniques, using a white light source and a spectrum analyzer, on sections of fibre of over 1 m length.

## 3. Results and discussion

The phonon energies of cadmium halide and gallium fluoride glasses were established using Raman scattering. The cadmium halide composition was found to possess two Raman peaks, at  $250\text{ cm}^{-1}$  and  $370\text{ cm}^{-1}$ , due to Cd-Cl and Cd-F vibrations respectively. Since the probability for multiphonon relaxations decreases exponentially with the number of phonons participating, the process is dominated by the highest energy phonons present in the glass

structure. Therefore in cadmium halide glass multiphonon decay is expected to be mediated by the 370 cm<sup>-1</sup> phonons. This fact was recently confirmed<sup>[9]</sup> in an experiment in which the phonon energy was inferred from the temperature dependence of the decay rate. The gallium fluoride composition was found to possess one peak at 525 cm<sup>-1</sup>, corresponding to the Ga-F vibration.

Given the energy of the phonons mediating the multiphonon relaxation process, it is possible to estimate the expected lifetime. This can be done by using an approximate empirical formula for multiphonon decay rate which requires only the phonon energy ( $\hbar\omega$ ) and the energy gap ( $\Delta E$ ) as its inputs<sup>[10]</sup>:

$$W_{mp} = A (1+n)^p \exp[-\beta(p-2.4)]$$

$$n = [\exp(\hbar\omega/kT) - 1]^{-1}$$

$$p = \Delta E/\hbar\omega \quad (1)$$

$$A \sim 10^7 \text{ s}^{-1}$$

$$\beta \sim 4.0$$

where A and  $\beta$  are constants, n is the phonon occupation number, and p is the number of phonons participating in the process. Table 1 below shows the estimated multiphonon lifetimes together with radiative lifetimes calculated using standard Judd-Ofelt methods. Also given in Table 1 are the total expected lifetimes and the actual lifetimes measured in bulk glass. The discrepancy may be accounted for by the presence of impurities.

Table 1: calculated and measured lifetimes of the <sup>1</sup>G<sub>4</sub> level of Pr<sup>3+</sup>.

Glass	$\tau_{\text{radiative}}, \mu\text{S}$	$\tau_{\text{non-radiative}}, \mu\text{S}$ (estimated)	$\tau_{\text{total}}, \mu\text{S}$ (estimated)	$\tau_{\text{total}}, \mu\text{S}$ (measured)
Cd-halide	2870	640	520	325
Ga-fluoride	3000	310	280	200
ZBLAN	2880	110	105	110

The total non-radiative decay rate is the sum of multiphonon decay, concentration quenching and impurity quenching, and therefore can be substantially increased by contributions

of the latter two processes. Concentration quenching takes place as a result of cross-relaxations among dopant ions, and is largely determined by their interaction range and spatial distribution<sup>[11]</sup>. Impurity quenching can be due either to higher-energy phonons originating in impurities, or to resonant energy transfer from excited ions to impurities (so-called "killer sites") which provide fast non-radiative decay paths. Impurity quenching depends on the nature and strength of interactions between dopant ions and impurities, on the presence of resonant energy levels in impurity ions, and on the concentration and distribution of impurities<sup>[11]</sup>. Whilst the multiphonon rate is an intrinsic parameter of the ion-host combination, both concentration and impurity quenching may be reduced by careful preparation and processing of the glass.

We have investigated concentration quenching in cadmium halide glass. Figure 2 shows the measured total  ${}^1G_4$  lifetime plotted against  $Pr^{3+}$  concentration. As can be seen, the quality of the data will not allow to differentiate between various possible models of the concentration quenching process. Nevertheless, these results confirm that concentration quenching in  $Pr^{3+}$ -doped glass is negligible at concentration levels below ~600 ppm. As a consequence, when fabricating preforms, the doping level of the core was consistently maintained in the 500-600 ppm range.

We have also extensively investigated the extent to which impurities contribute to increased non-radiative decay rate of the  ${}^1G_4$  level. High purity glass samples were prepared from high purity starting materials and processed in oxygen- and water-free atmosphere. Some of the samples were doped with controlled amounts of impurities. Figure 3 displays the measured total decay rate of the  ${}^1G_4$  level in cadmium halide and gallium fluoride glasses as a function of OH<sup>-</sup> concentration, defined as the integrated absorption of the 3500  $cm^{-1}$  water peak. The data graphically demonstrates the detrimental effect that the OH<sup>-</sup> impurity has on the lifetime of the  ${}^1G_4$  metastable level of  $Pr^{3+}$  ion. Although once again it is not possible to speculate on the exact mechanism of the impurity quenching process, it can be estimated by extrapolating to zero impurity level that with a further reduction in the level of impurities the

observed lifetime may be extended to 350-370  $\mu\text{s}$  in cadmium halide glass and to 210-220  $\mu\text{s}$  in gallium fluoride glass.

The lifetime of the  ${}^1G_4$  metastable level determines the quantum efficiency of the amplifier and sets the upper bound on its realisable gain. However, the actual net gain delivered by a fibre amplifier is also strongly affected by fibre loss<sup>[12]</sup>.

$$QE = \frac{\tau_{total}}{\tau_{radiative}}$$

$$g_0 = \frac{\sigma_{emission} \tau_{total}}{h\nu_{pump}} \frac{P_{abs}}{A_{core}} \mathcal{F} \quad (2)$$

$$g_l = g_0 \alpha l$$

where  $g_0$  and  $g_l$  are respectively the small-signal gain of the amplifier and the net gain per length  $l$  of the fibre,  $\sigma_{emission}$  is the emission cross-section,  $P_{abs}$  and  $A_{core}$  are the pump power absorbed by the core and its cross-sectional area,  $\mathcal{F}$  is the pump-signal overlap integral, and  $\alpha$  is the total absorption coefficient at the signal wavelength.

The intrinsic component of the total fibre loss consists of contributions from Rayleigh scattering and infrared absorption edge. Figure 4 shows the calculated intrinsic loss in cadmium halide glass. The Rayleigh scattering loss was estimated from the available ZBLAN data, with adjustments for the refractive index and glass transition temperature, according to the method described elsewhere<sup>[13]</sup>. The IR absorption edge was measured using standard techniques<sup>[14]</sup>, and the data fitted to the loss equation. The intrinsic losses in cadmium halide glass were found to be  $-0.1$  dB/km at 1  $\mu\text{m}$ .

Due to the difficulties in fibre fabrication, in practice losses in fluoride glass fibres are dominated by extrinsic factors, such as scattering from microcrystals and impurity absorption<sup>[8]</sup>. Figure 5 shows the total measured loss in gallium fluoride multimode fibre. The losses at signal and pump wavelengths are respectively 5 dB/m and 7 dB/m. The corresponding values for a cadmium halide fibre are 7 dB/m and 10 dB/m. These losses exceed the intrinsic loss values by

a factor of  $10^4$ , indicating that much work remains to be done in improving fibre quality. For an efficient, high gain PDFA to become feasible, the losses in fibre must be reduced by at least an order of magnitude.

Based on the above measurements, it was possible to carry out extensive amplifier modelling with a view to evaluating the projected performance of PDFA based on cadmium halide and gallium fluoride glasses. The expected gain curve of the 1.3  $\mu\text{m}$  amplifying transition of  $\text{Pr}^{3+}$  in cadmium halide glass was calculated from the measured absorption and emission spectra using methods described elsewhere<sup>[15]</sup>. This calculation also took into account the effect of signal excited state absorption (ESA) from the  $^1\text{G}_4$  level to the above  $^1\text{D}_2$  level, the net gain curve being the difference between the emission and ESA. The calculated gain curve is almost symmetric (see Figure 6), and has a peak cross-section of  $3.5 \times 10^{-25} \text{m}^2$  at 1305 nm and a FWHM of 70 nm. Since amplifier gain is proportional to the net emission cross-section (see equation 2), this is a crucial parameter, as is the width of the curve and its position well within the 1280-1320 nm transmission window.

In order to establish the amount of pump required to obtain a desirable level of gain, the gain versus pump curves were calculated for a PDFA based on cadmium halide and gallium fluoride glasses; these are shown in Figure 7. A similar curve for a state-of-the-art ZBLAN-based device is also included in Figure 7 for comparison. The model assumes either counter- or bi-directional pumping scheme. The assumed background loss is in all cases 0.05 dB/m, corresponding to the state-of-the-art ZBLAN fibre. The calculated slope efficiencies for cadmium halide, gallium fluoride and ZBLAN are respectively 0.55, 0.35 and 0.21 dB/m, the increase in efficiency being directly related to the extended  $^1\text{G}_4$  lifetime in these glasses.

Since amplifier gain is inversely dependent on background losses, and since fabricating novel glasses into low-loss fibres is a major technological challenge, it was important to establish what level of losses can be comfortably tolerated while maintaining acceptable gain. Accordingly, gain versus loss curves were calculated for PDFA based on cadmium halide and



gallium fluoride glasses; these are shown in Figure 8. A bi-directional pumping scheme was assumed, with a pump power of 100 mW. The results indicate that a gain of 20 dB, which is the minimum required of a commercial device, can be achieved with a background loss of 1 dB/m for cadmium halide glass and 0.5 dB/m for gallium fluoride. These values are an order of magnitude greater than typical losses in a state-of-the-art ZBLAN fibre. Further increases in gain can be achieved by using a double-pass pump configuration<sup>[4]</sup>.

In order to optimize the fibre geometry of PDFA based on new cadmium halide and gallium fluoride glasses, modelling related to the numerical aperture (NA) of the fibre has been carried out. Amplifier gain increases sharply with increasing NA, owing to the better confinement and overlap of pump and signal<sup>[16]</sup>. Simultaneously, as the NA rises, the core diameter must be reduced so as to maintain a constant cut-off wavelength<sup>[17]</sup> (~900 nm). This leads to increased losses, as well as incurring greater coupling losses. The optimum NA is therefore a trade-off between higher gain and losses. Gain versus NA curves for cadmium halide and gallium fluoride glasses are shown in Figure 9a. NA versus core curves for the same glasses are given in Figure 9b. These calculations indicate that the optimum NA is ~0.4 for cadmium halide fibre and somewhat higher for gallium fluoride fibre. Indeed, gallium fluoride glass fibre with NA as high as 0.52 has been successfully fabricated.

#### 4. Conclusion

Two new glasses based on cadmium halide and gallium fluoride have been developed and fabricated into Pr<sup>3+</sup>-doped fibre. A variety of measurements have been carried out aimed at investigating the potential of these glasses as hosts for 1.3  $\mu\text{m}$  PDFA. Amplifier modelling of the projected device characteristics was performed. The results indicate that the new glasses promise higher gain and quantum efficiency, with consequent lower pump requirements, than the state-of-the-art ZBLAN-based PDFA device. Both glasses are therefore excellent candidates for a second generation of PDFA devices.

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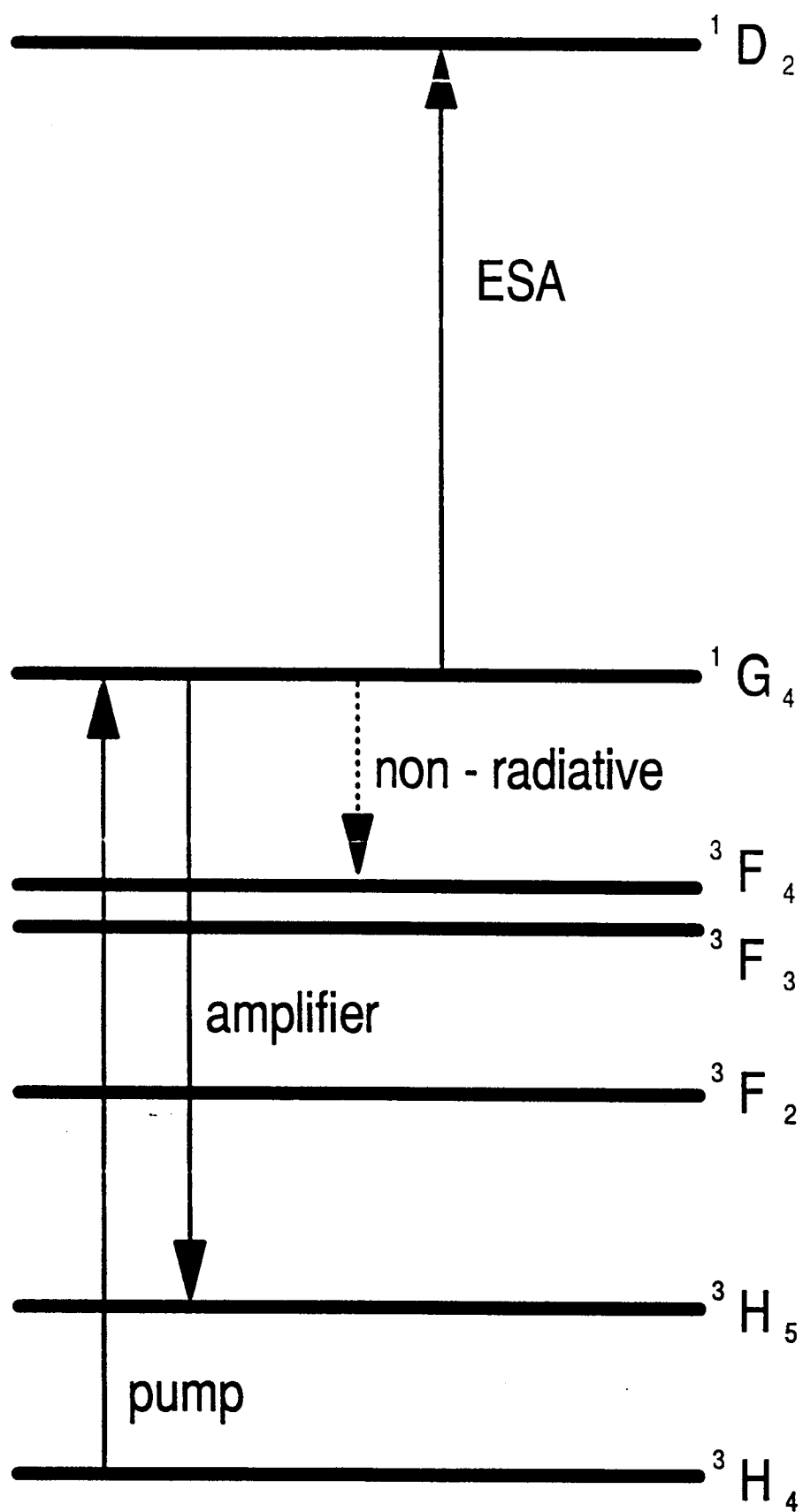
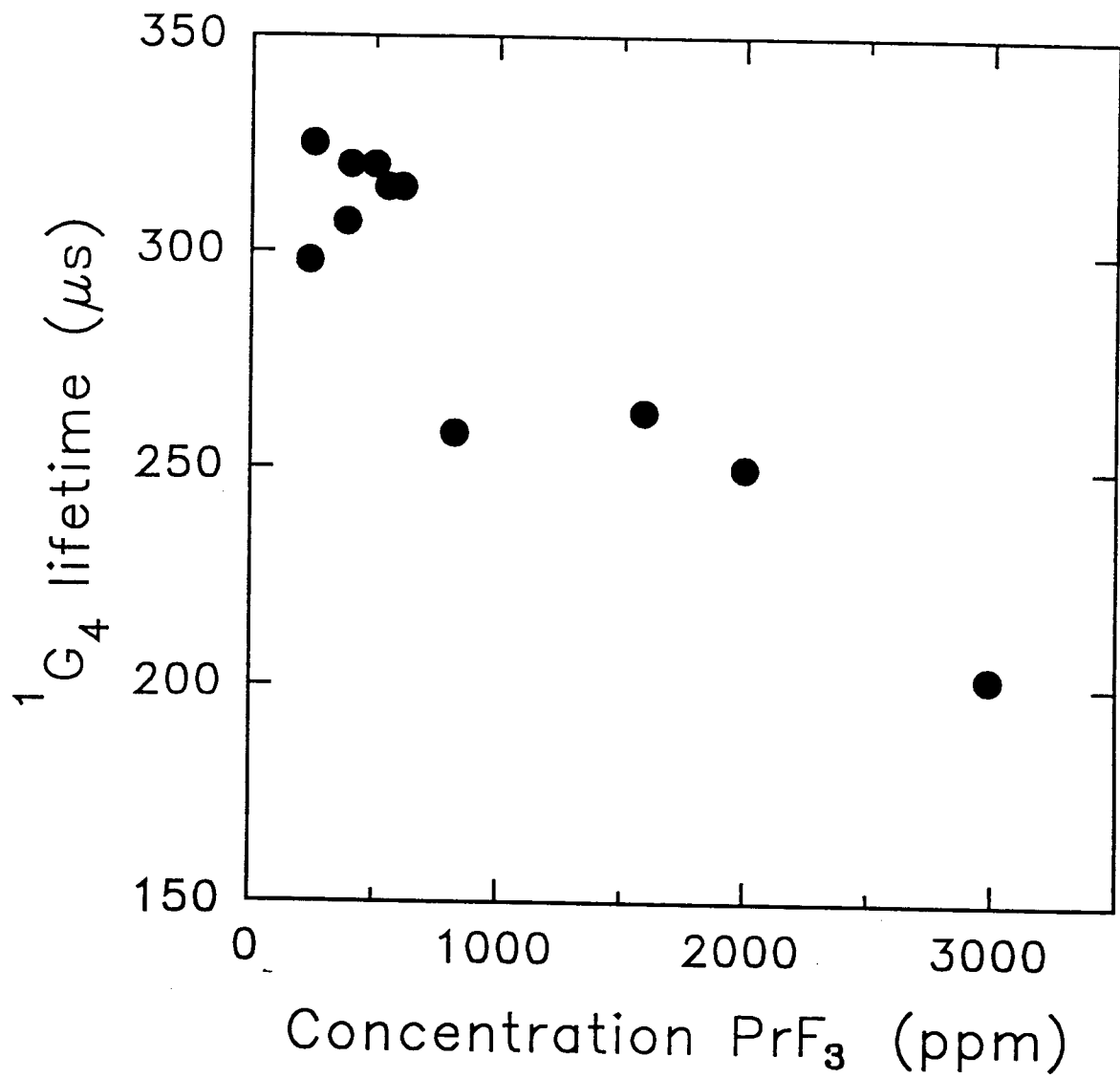
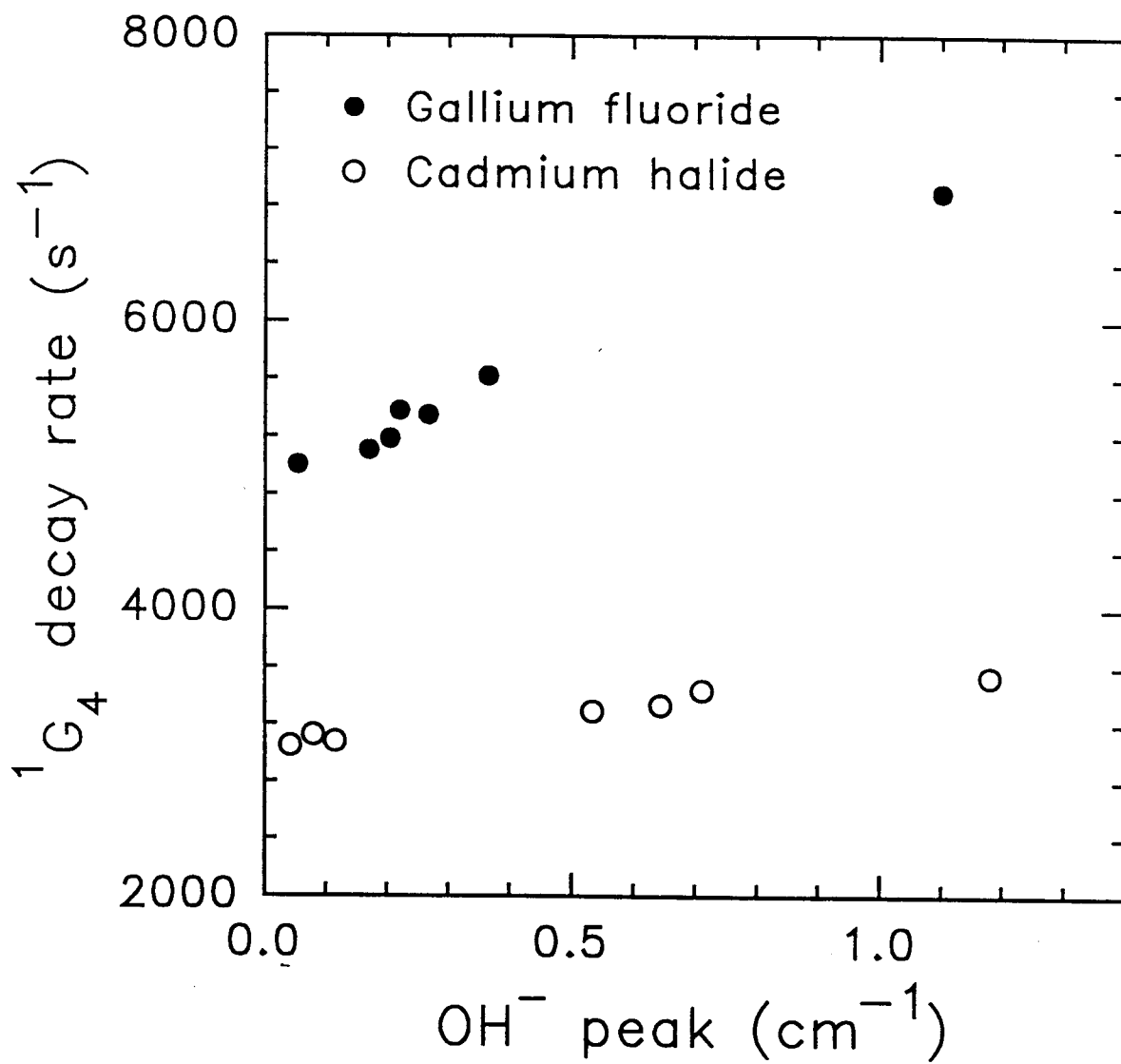


FIG 1





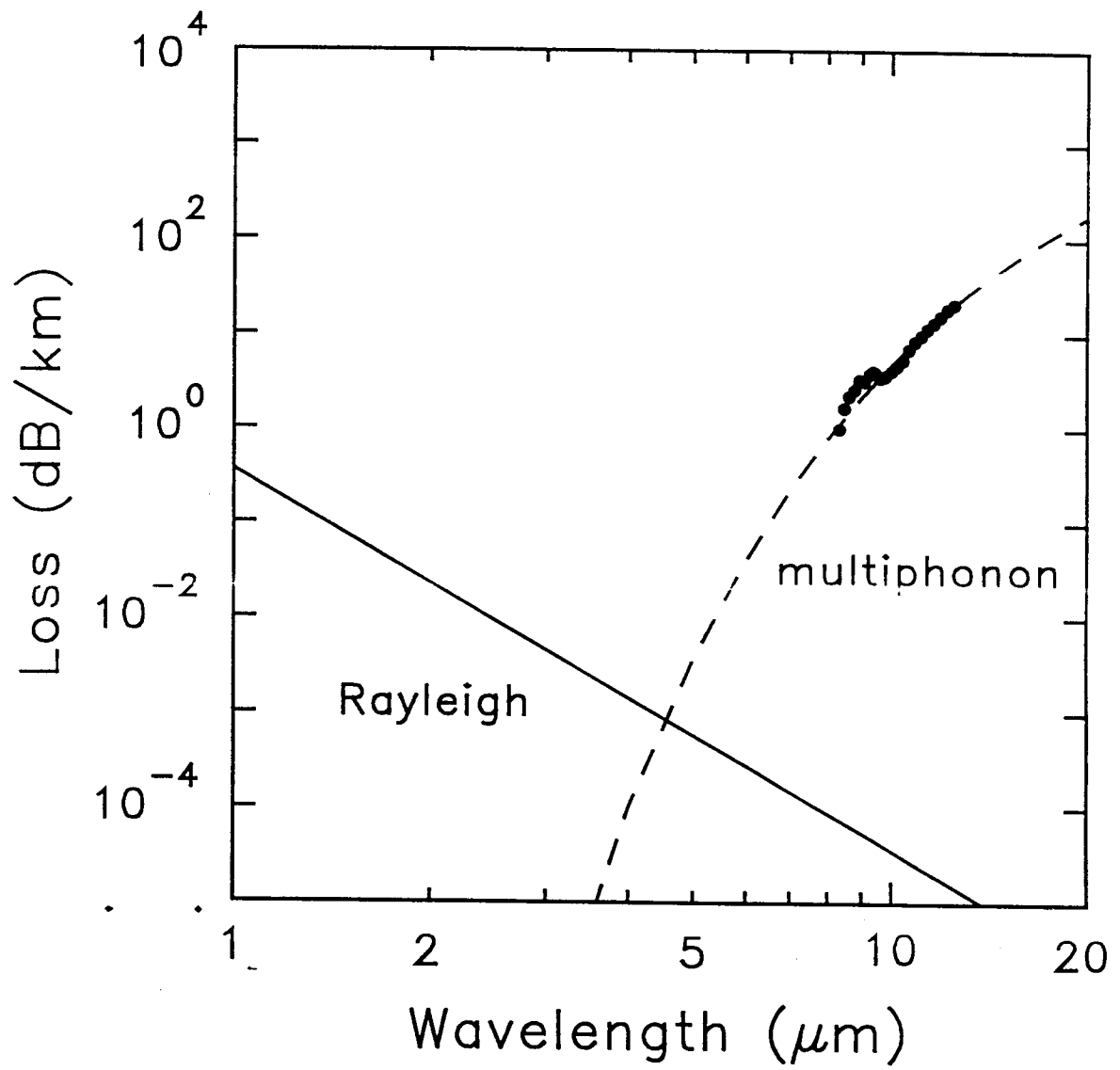


FIG 4

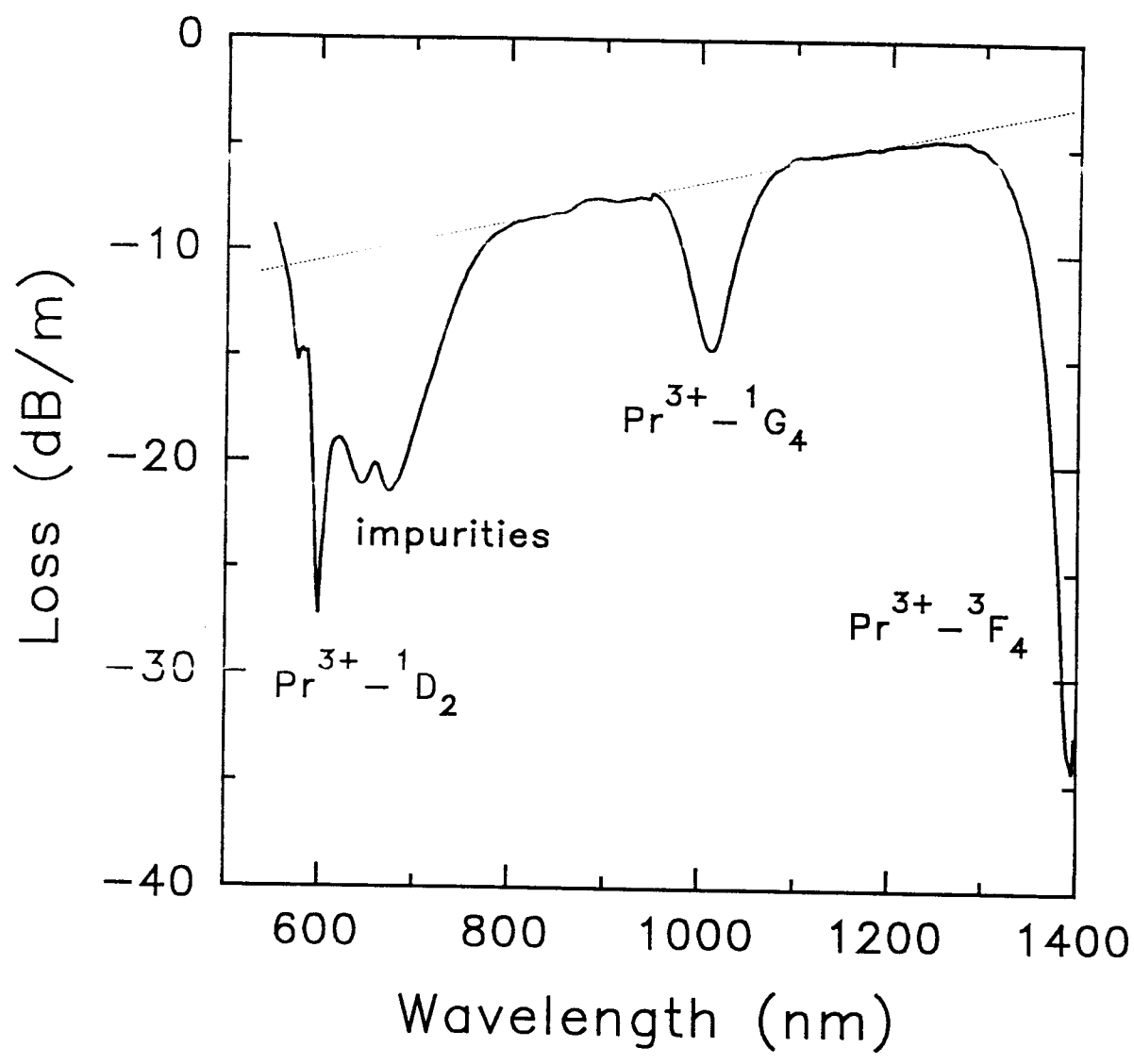


FIG 5



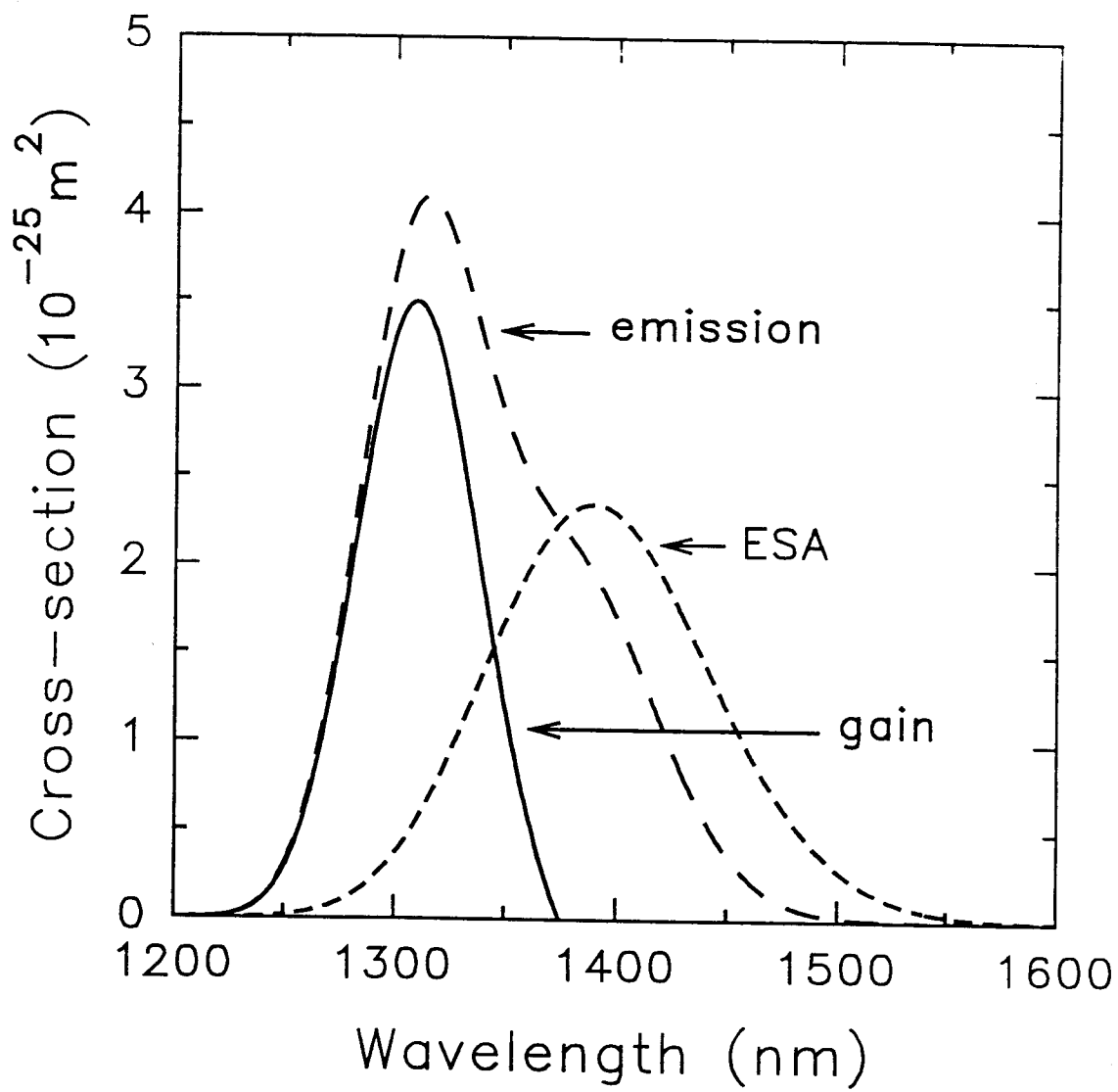


FIG 6

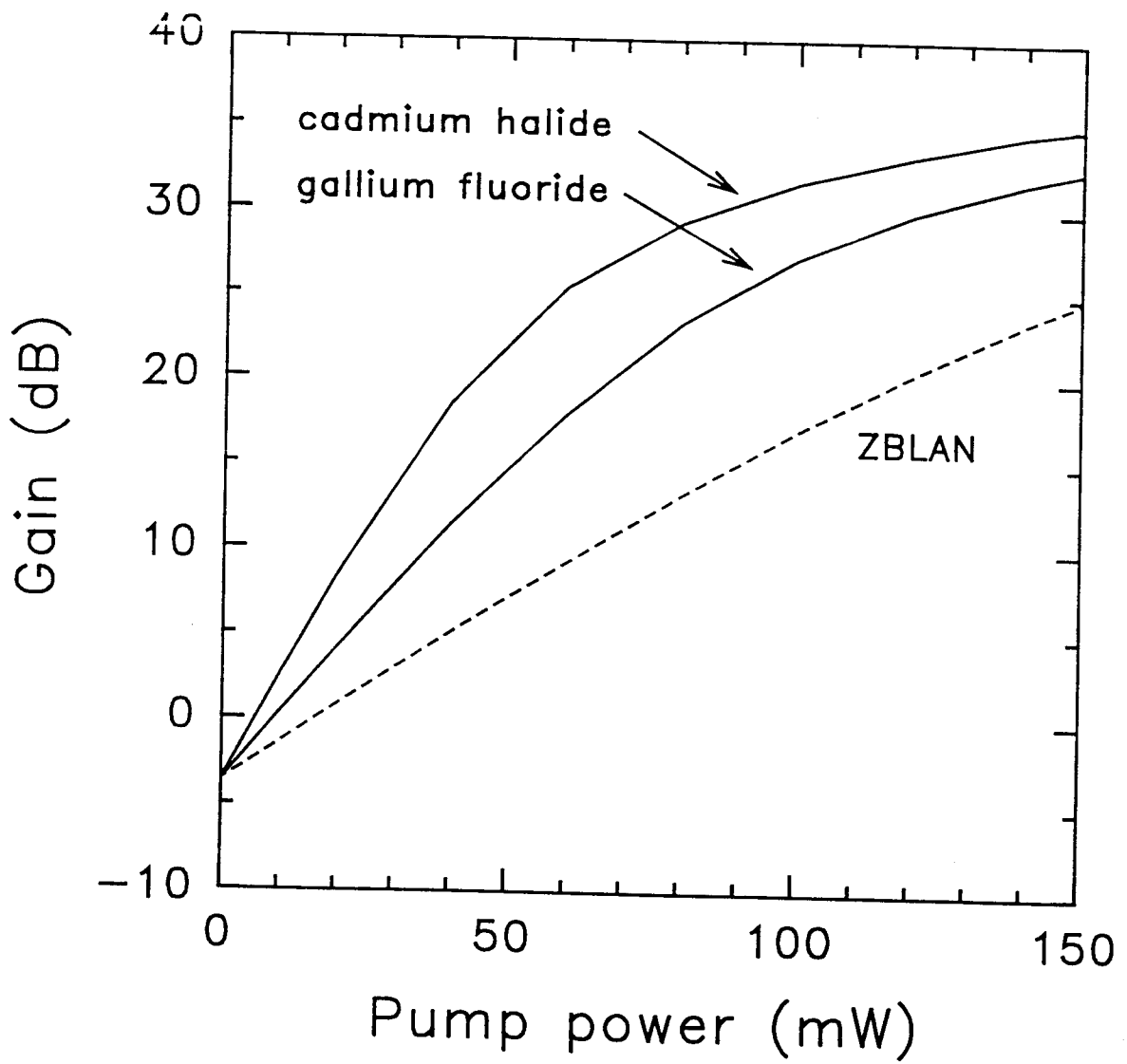


FIG 7

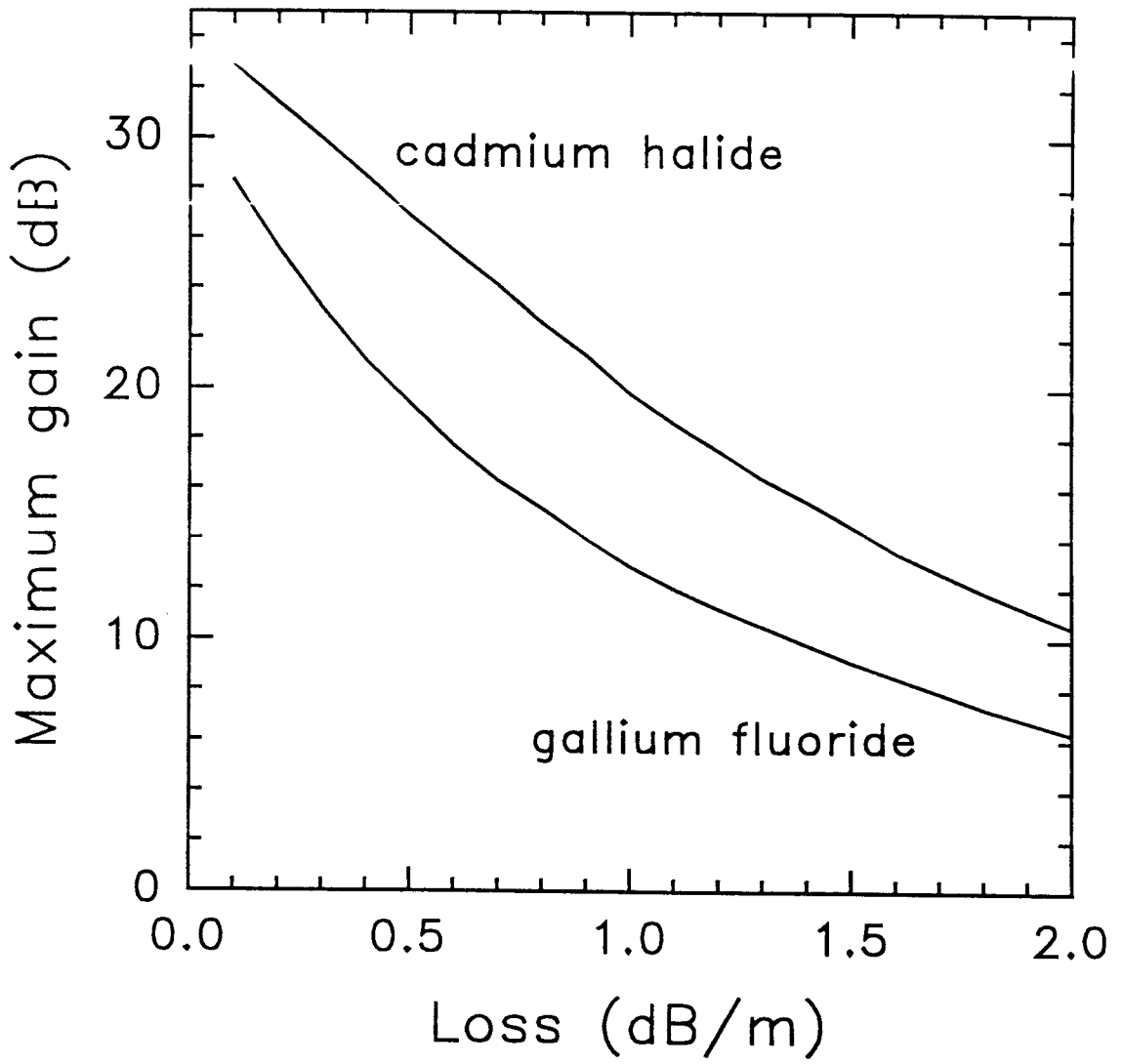


FIG 8

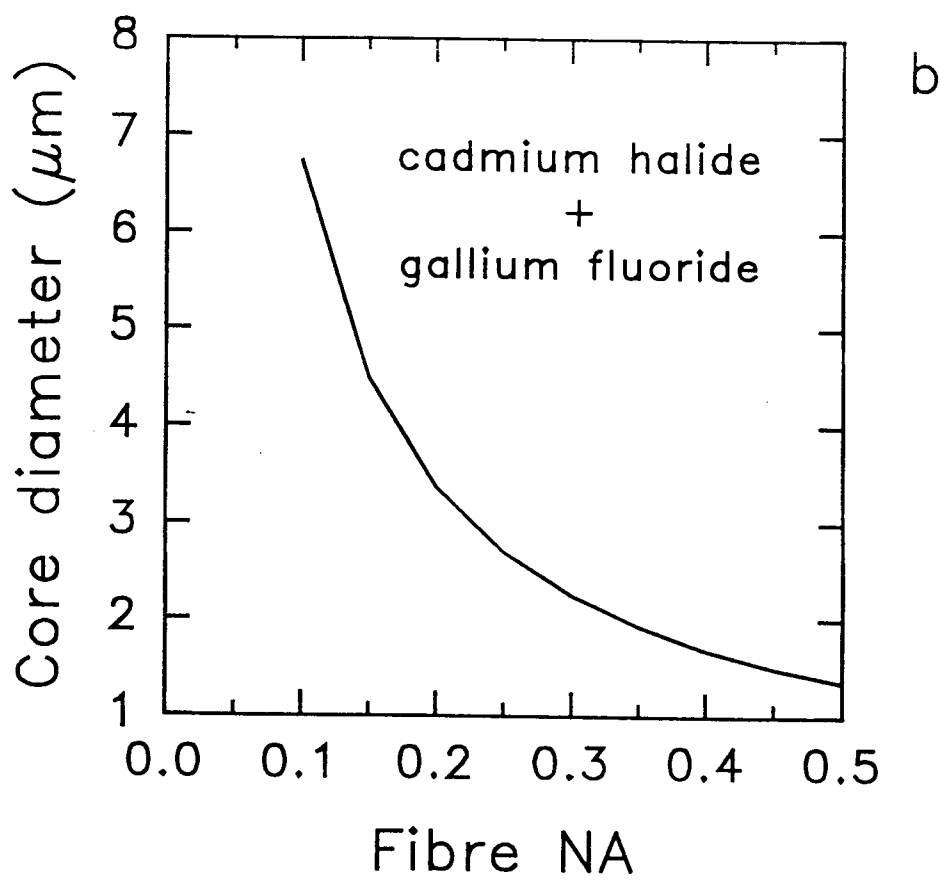
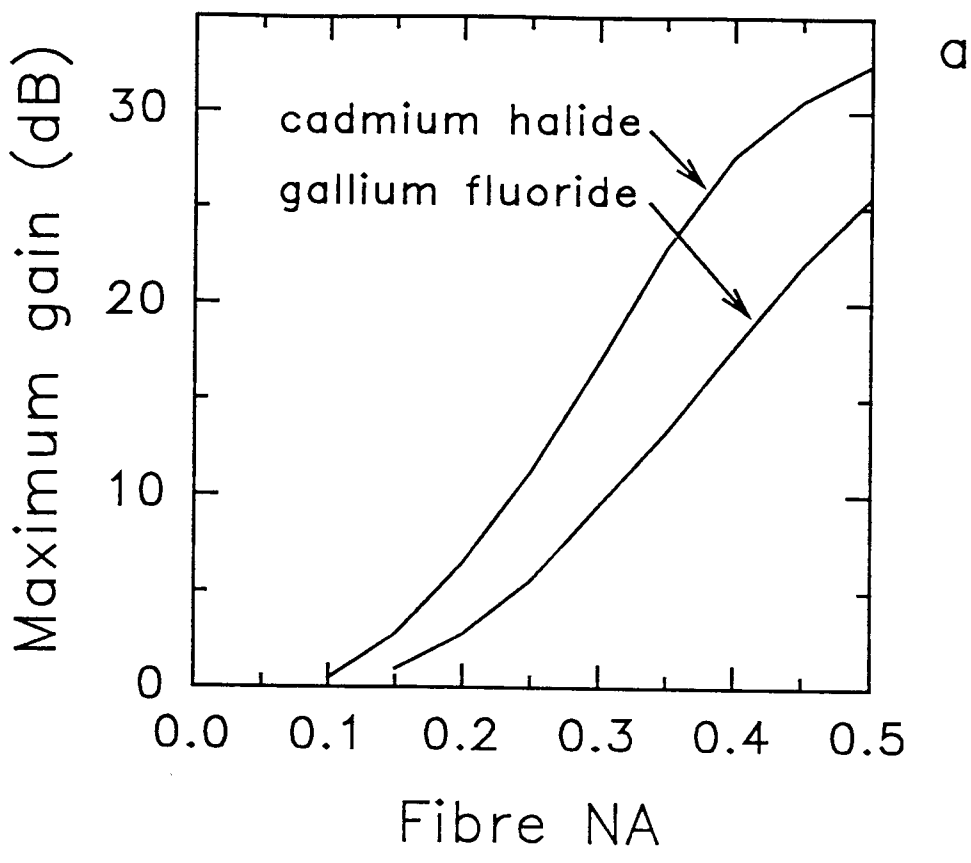


FIG 9