PROPERTIES, PREPARATION AND APPLICATIONS OF LOW-LOSS OPTICAL FIBRES CONTAINING RARE-EARTH IONS.

J.E. Townsend and W.A. Gambling Optoelectronics Research Centre, The University, Southampton, SO9 5NH, U.K.

I. INTRODUCTION

Traditionally little attention was paid to glasses doped with rare-earth ions because of their scarcity and lack of visible colouration. Some interest was stimulated by the demonstration of laser action in a neodymium-doped glass in 1961. Recently, however, the development of novel fabrication techniques for introducing high concentrations of rare-earth ions into low-loss optical fibres has aroused tremendous interest.

Several methods of preparation have been proposed but the solution-doping technique¹ developed at Southampton, is now employed world-wide. Subsequent application of these fibres has led to many new devices, both passive and active (e.g. temperature senors and lasers²).

II. FABRICATION

The widely-used Modified Chemical Vapour Deposition process (MCVD) for optical fibres is based on the high-temperature oxidation of reactant halides from the gas phase. The glass-forming reagents are obtained in the gas phase by bubbling carrier gases (e.g. O₂, N₂) through high vapour pressure precursor liquids (e.g. SiCl₄, GeCl₄). These materials, which determine the refractive index of the deposited glass, flow inside a rotating glass tube and are heated by an external burner traversing in the same direction as the gas flow. Oxidation in the hot zone results in formation of soot particles which are deposited further downstream. Subsequently, when the burner passes, the soot layer fuses to a clear glass. The tube is then collapsed into a solid preform for drawing into a fibre.

Many variants of the MCVD process have been adapted for the doping of fibres with rare-earth ions. For example, organometallic liquids of high vapour pressure have been employed as the dopant precursor³ in a conventional MCVD configuration. A wide range of dopant concentrations (up to several mole percent)

of high purity can be achieved but these materials are not commonly available, thus Poole4 adopted an alternative approach, employing rare-earth halide salts. A significant vapour pressure is generated by heating the sample above 1000°C in a dopant chamber placed upstream of the burner. A typical configuration is shown in Figure 1 with a schematic representation of the MCVD technique. Vapour-phase oxidation of the rare-earth salts allows homogeneous distribution of dopant, both axially and radially across the preform, but dopant concentrations in excess of a few hundred ppm are not readily achieved due to low precursor vapour pressure. In addition the precursor must be dried prior to use by heating in the presence of chlorine to remove OH-, which contributes excess absorption loss to the fibre. Further dehydration is employed during sintering to ensure low fibre losses. Nevertheless the technique is simple to implement and variants are widely used⁵.

For greater flexibility of fabrication, allowing a wider range of dopant materials, concentrations and co-doping of ions, Townsend1 developed a solution-doping technique. The temperature in conventional MCVD is greatly reduced to ~1000°C in order to deposit a porous oxide soot instead of a glass layer. Subsequently the frit is immersed in a dilute solution of the dopant ions, with any desired additional network-forming or modifying materials. Both aqueous and alcoholic solutions of halide or nitrate salts have been employed. Diffusion of cations through the soot occurs. Excess solvent is then removed and the soot dehydrated by heating in the presence of chlorine. doped, soot layer is then sintered into a clear glass and the subsequent MCVD processing steps are conventional. Both core and cladding may be doped. The highest rare-earth ion concentration cited⁶ is ~ 4 mol % in fibres co-doped with Yb^{3+} , Er^{3+} and Al_20_3 with each component incorporated in a predetermined ratio.

III. PROPERTIES AND APPLICATIONS

Rare-earth ions exhibit a wide range of features which are attractive for device applications. Here, each characteristic is discussed with reference to potential devices rather than presenting materials properties *per se*.

A. Absorption

Rare-earth ions in amorphous silica based fibres exhibit intense, narrow ($\Delta\lambda\approx20\text{nm}$) absorption bands characteristic of their electronic transitions whilst maintaining the low losses of telecommunication fibres in the pass bands. Consequently the absorption losses can change by several orders of magnitude over a narrow range, as shown in Figure 2 for a Yb³⁺ - Er³⁺ co-doped fibre 6.

1. Fibre Filter

These large variations in loss have been employed to construct a compact low-loss wavelength filter having an extremely high rejection ratio⁸. Transmission in the blocking band is 10%, whilst that in the pass band is 85%, with a wavelength separation of only 40nm. In this manner forward scattering of spontaneous Raman scattering was recorded for the first time.

2. Temperature Sensor

The distribution of ground state population is in accordance with Boltzmann's principle. Hence variations in the absorption spectrum with temperature are seen in those fibres with secondary energy levels close to the ground state, in particular Er³+, Nd³+, Sm³+ and Ho³+. The loss spectra of a GeO₂/SiO₂ fibre doped with 100ppm of Ho³+ for two temperatures, Figure 3, shows clearly that, over the wavelength range 665-685nm, the change in loss is greater than 2%/°C. The fibre configuration allows distributed temperature sensing by conventional backscatter techniques giving the spatial loss spectrum as shown in Figure 4. In this manner a temperature resolution, over long lengths of fibre, of better than 1°C, and a spatial resolution of 3.5m, over the range -200°C to 100°C has been demonstrated.

B. Emission

Rare-earth ions can emit characteristic fluorescence, if optically excited, from a number of transitions when contained in any host material. In amorphous silica the bands are inhomogeneously broadened, resulting in a broad linewidth of ~50nm as shown in Figure 5 for Er³⁺-doped, Al₂O₃/SiO₂ core fibre. Strong emission characteristics such as these have previously been employed in various bulk glass laser configurations. However, continuous-wave operation was difficult to obtain because of the

high excitation power required for transverse pumping and also the need for cooling to avoid fracture. The fibre configuration, however, provides excellent laser pumping for several reasons. Firstly, the nature of the guided-wave structure allows highlyefficient interaction between the excitation radiation and the dopant ions, partially overcoming the effects of small absorption Secondly, the small core area, typically 10-11m2, cross-section. provides high pump power densities even at the low powers available from semiconductor laser diodes. Compact, cheap and robust optical pumping sources can therefore be employed. Thirdly, thermal shocks are avoided, without the requirement for external cooling, owing to the small overall fibre cross-sectional $(10^{-8} \text{ m}^2).$

Consequently, laser operation has been demonstrated in silica, and fluoride-based, optical fibres over a wide range of wavelengths⁹, Figure 6, from 651nm to $2.7\mu m$. Included in this list are several new transitions, for example, laser action of Pr^{3+} in glass and, at 651nm, the first laser action of Sm^{3+} in any host. The erbium system is currently of greatest interest since the emission band at $1.535\mu m$ coincides with the lowest-loss, large bandwidth transmission window in silica fibre systems. High gain, up to >40dB, has been demonstrated¹⁰ and short lengths of optically-pumped erbium-doped fibre may now be employed in a communications network, instead of electronic repeaters, to amplify the transmitted signal.

Pulsed operation of fibre laser systems by Q-switching or mode locking the laser cavity generates short intense pulses with application for range finders, data storage systems, medical investigation and spectroscopic sources.

A number of more unusual applications for rare-earth-doped optical fibres are also under investigation. The high pump power densities can lead to multiple absorption of input radiation producing emission at higher energies. For example strong blue emission (475nm) is seen on excitation of ${\rm Tm}^{3+}$ at 670nm or $1.064\mu{\rm m}$. Research into such upconversion systems now extends world-wide.

Another potentially important device is the radiation dosimeter based on a fibre with a germanosilicate core doped with

neodymium^{11,12}, which is linear and rereadable even at high radiation doses and high temperatures.

More speculative ideas include the formation of integral longitudinal, gratings in the core of a fibre by using the photosensitive properties of Tm^{3+} . Alternatively, the paramagnetic ions Tb^{3+} , Dy^{3+} and Pr^{3+} exhibit large Verdet constants (20 times that of silica). Combined with the long interaction lengths possible in fibres these materials may produce highly sensitive magnetic field sensors.

IV. CONCLUSION

Fabrication techniques for the manufacture of low-loss optical fibres containing controlled amounts of rare-earth ions have been developed, the solution-doping technique being the most widely employed. Numerous devices have already emerged and some products are finding commercial application, but it is increasingly clear that many novel systems have yet to be fully realised.

V. REFERENCES

- J.E. Townsend, S.B. Poole and D.N. Payne,
 "Solution-doping technique for fabrication of rare-earth-doped optical fibres",
 Electronic Letters, 23, pp. 329-331, 1987.
- See for example, "Novel Optical Fibres" by W.A. Gambling and S.B. Poole in "Optical Fibres for Sensors", Artech House, 1988, pp. 249-276.
- 3. D.A. Thompson, P.L. Bocko and J.R. Gannon, "New source components for the fabrication of doped optical waveguide fibres", Proc. Society of Photo-Optical Instrumentation Engineers, 506, pp. 170-175, 1984.
- 4. S.B. Poole, D.N. Payne and M.E. Fermann, "Fabrication of low-loss optical fibres containing rare-earth ions", Electronic Letters, 21, pp. 737-738, 1985.

- 5. K. Arai, "Preparation of active ion-doped silica glasses and their properties", Bulletin Ceramic Society of Japan, 21, pp. 419-424, 1986.
- W.L. Barnes, S.B. Poole, J.E. Townsend, L. Reekie,
 D.J. Taylor and D.N. Payne,
 "Er³⁺-Yb³⁺ and Er³⁺-doped fibre lasers",
 J. Lightwave Technology, LT-7, pp. 1461-1465, 1989.
- 7. G.H. Dicke and H.M. Crosswhite,

 "The spectra of doubly and triply ionised rare-earths",

 Applied Optics, 2, pp. 675-686, 1963.
- 8. M.C. Farries, J.E. Townsend and S.B. Poole,
 "Very high-rejection optical fibre filters",
 Electronics Letters, 22, pp. 1126-1127, 1986.
- 9. D.N. Payne and L. Reekie, "Rare-earth fibre lasers and amplifiers", Invited Paper in Proc. ECOC '87, Helsinki, pp. 89-94, 1987.
- 10. R.I. Laming, V. Shah, L. Curtis, R.S. Vodhanel, F.J. Favire, W.L. Barnes, J.D. Minelly, D.P. Bour and E.J. Tarbox, "Highly efficient, 938nm diode pumped, erbium-doped, fibre amplifier with 24dB gain", Proc. IOOC '89, Paper PDA4, pp. 18-19, 1989.
- 11. A.E. Ellis, P.D. Moskowitz, J.E. Townsend and P.D. Townsend, "A novel rereadable radiation dosimeter with high temperature sensitivity", J. Phys. D., in the press, November 1989.
- 12. Y. Kirsh, J.E. Townsend and P.D. Townsend,

 "Kinetic analyses of the new sensitisation effect in the TL of silica optical fibres",

 Phys. Stat. Sol., 114, pp. 821-829, 1989.

V1. FIGURE CAPTIONS

- Figure 1. Schematic representation of the vapour phase deposition technique for the fabrication by the MCVD technique of rareearth lons with fibres.
- Figure 2. Spectral loss characteristics of a Yb³⁺/Er³⁺ co-doped fibre prepared by the solution doping technique.
- Figure 3. Spectral loss of a fibre with a Ho³⁺ doped core at 20°C and -196°C.
- Figure 4. Attenuation distribution along
 a Ho³⁺ doped fibre in regions
 of different temperatures
 determined by optical time
 domain reflectometry.
- Figure 5. Fluorescence spectrum of an aluminosilicate fibre having an Er³⁺ doped core.
- Figure 6. Laser emissions in silicate, and fluoride, glass optical fibres.

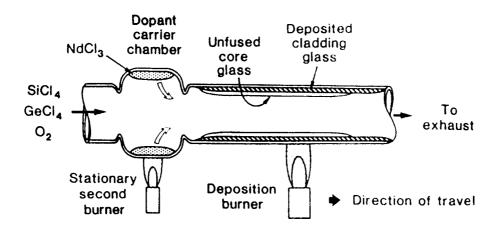


Figure 1. Schematic representation of the vapour phase deposition technique for the fabrication by the MCVD technique of rare-earth ions with fibres.

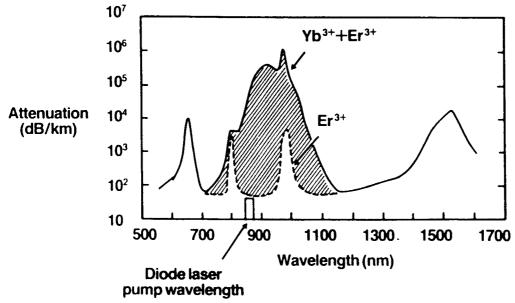


Figure 2. Spectral loss characteristics

of a Yb³⁺/Er³⁺ co-doped fibre

prepared by the solution doping technique.

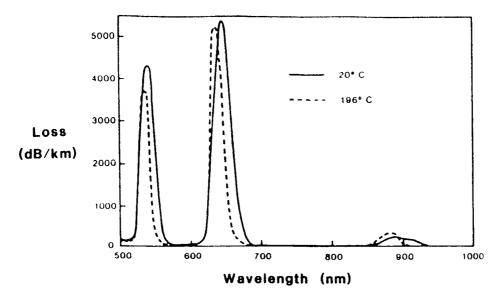


Figure 3. Spectral loss of a fibre with ${\rm a\ Ho^{3}}^{+}\ doped\ core\ at\ 20^{O}C\ and }$ ${\rm -196^{O}C}.$

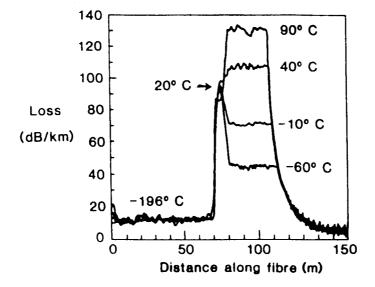


Figure 4. Attenuation distribution along

a Ho³⁺ doped fibre in regions

of different temperatures

determined by optical time

domain reflectometry.

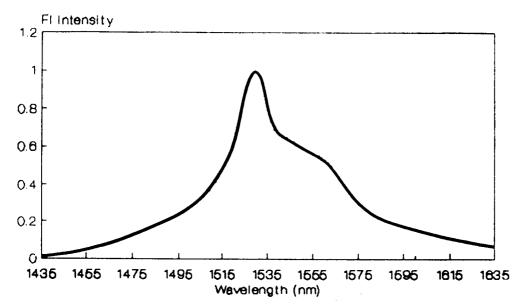


Figure 5. Fluorescence spectrum of an aluminosilicate fibre having an ${\sf Er}^{3\,+}\ \ {\sf doped}\ \ {\sf core}.$

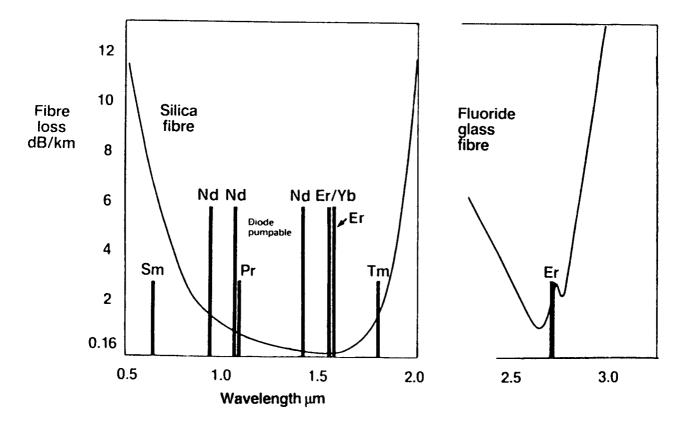


Figure 6. Laser emissions in silicate, and fluoride, glass optical fibres.