

## FABRICATION OF SPECIAL OPTICAL FIBRES

J.E.Townsend

The Optical Fibre Group, Dept. Electronics and Computer Science,  
University of Southampton, SO9 5NH, U.K.

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### *Introduction*

Techniques for the manufacture of silica based single or multi-moded optical fibres are well established and optical communications networks commonplace. Two fabrication methods are commonly employed, the modified chemical vapour deposition<sup>1</sup> (MCVD) and vapour axial deposition<sup>2</sup> (VAD) processes. However, interest in silica fibres for other applications, such as sensing and active fibre based devices requires novel production techniques.

Birefringent fibres of many types (for example, linear<sup>3</sup> and ultra low<sup>4</sup> birefringence as well as polarising<sup>5</sup> fibres) have been developed and are exploited commercially. In general, the fibre geometry is altered to optimise the polarisation characteristics. Many processes are employed, including the "Bow Tie"<sup>3</sup> and "PANDA"<sup>6</sup> configurations. Modification of the preform structure during its manufacture achieves the necessary geometry for the Bow Tie, whereas the PANDA fibre is formed from a composite preform by a "rod in tube" method. The process is also used to fabricate fibres with multiple or offset cores for sensor or switching applications.

Nonlinear optical effects, acousto-optic, magneto-optic and electro-optic effects are important for optimised fibre configurations. These coefficients are small in conventional, silica based fibres but greater effects are achieved by employing alternative hosts, including multicomponent glasses or even polymers. Although the fibre loss is compromised in such cases typically only a few metres of fibre are required since the nonlinear coefficient may be increased by several orders of magnitude. The "rod in tube" technique lends itself well to the fabrication of such fibres<sup>7</sup>.

Alternatively, considerable scope exists for modifying the properties of silica based fibres by adding suitable dopants, for example, transition or rare-earth metal ions, to increase the nonlinear coefficients. Such ions are not easily incorporated into a fibre preform by conventional techniques so novel processes have been developed. In the most widely used methods dopants are added either

from the vapour phase<sup>8</sup> or from solution<sup>9</sup>.

Fibres based on amorphous fluorides<sup>10</sup> were developed for telecommunications networks owing to the, theoretically, exceptionally low losses but difficulties in the fabrication of single mode fibres has prevented their exploitation. However, the materials properties of fluoride based glasses differ substantially from those of the oxides, owing to their low phonon energy. Thus, there is considerable interest in such fibres doped with rare-earth ions for upconversion lasers<sup>11</sup>. Appropriate fabrication methods have been developed, whereby the glass is prepared by fusing powdered raw materials and many types of ion may be incorporated.

It is clear that improved fabrication technology, often based on conventional manufacturing processes, is able to provide fibres having characteristics optimised for device applications and examples of such fabrication methods are described here.

#### *Conventional fibre manufacture*

Optical fibres are drawn, or pulled, from preforms, whose characteristics determine those of the fibre. Two processes are widely employed to prepare single mode or graded index preforms for telecommunications applications. The Modified Chemical Vapour Deposition process (MCVD) was proposed in the USA and England in the early 1970's and relies on gas phase oxidation of vapour phase reagents to form the glass<sup>1</sup>. The Vapour Axial Deposition method relies on hydrolysis of the same materials and was developed approximately 10 years ago by the Japanese<sup>2</sup>. Both techniques are described here.

#### *Modified Chemical Vapour Deposition*

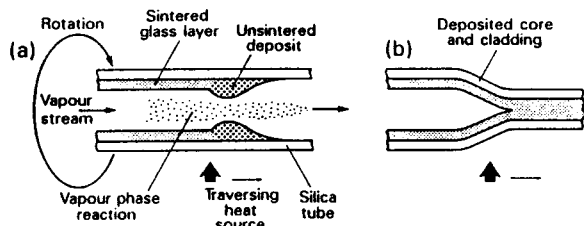


Figure 1. MCVD is based on the high temperature oxidation of reactant gases inside a rotating tube which is heated by an external source. A schematic of the process is shown in Figure 1. Controlled amounts of dopant are transported to the reaction zone by passing gases (e.g.  $O_2$ ) through the liquid precursors, usually halides, thereby avoiding contamination from transition metal ion impurities. Silicon tetrachloride ( $SiCl_4$ ) is used to form silica, the main component of

optical fibres.  $\text{SiO}_2$  is attractive due to the low loss and low dispersion<sup>12</sup> in the region 1300nm to 1550nm as well as its mechanical and chemical strength.  $\text{GeO}_2$  and  $\text{P}_2\text{O}_5$  are commonly added as fibre core material to raise the refractive index. Conversely,  $\text{B}_2\text{O}_3$  or fluorine reduce the refractive index of the deposited glass.

The reactant gasses flow, with  $\text{O}_2$ ,  $\text{N}_2$  and He, inside a rotating silica substrate tube mounted on a glass working lathe. The tube is heated by an external burner traversing in the same direction as the gas flow. Gas phase oxidation in the burner's hot zone (typically 1700°C) results in the formation of oxide soot particles, which are deposited further downstream. As the burner passes over the deposited layer, it is fused to a glass by viscous sintering. Deposition temperatures depend on glass composition and are sufficiently high to sinter the deposited material but not so high as to cause distortion of the tube (usually between 1300°C and 1650°C). Many layers of defined index are built up in this manner. After deposition of the required amount of material, the tube is collapsed to a solid rod by heating to approximately 2000°C, sufficient to soften the glass and allow collapse under surface tension.

### Vapour-phase Axial Deposition

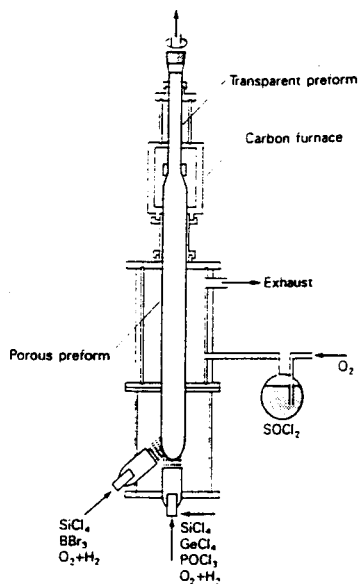
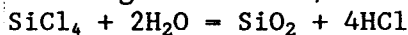


Figure 2.

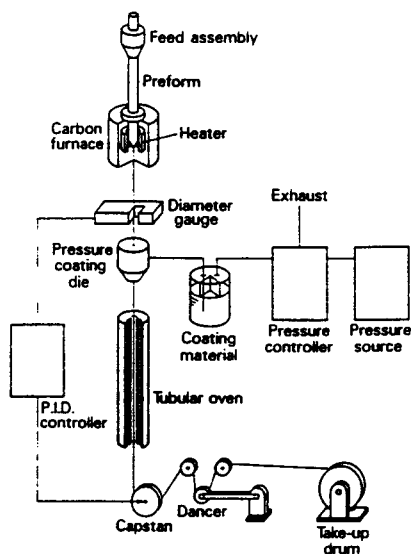
A schematic of the apparatus employed to prepare VAD preforms is shown in Figure 2. Halide precursor materials of the type used in the MCVD process are transported to the reaction zone where flame hydrolysis of the reagents occurs;



The oxide particles are deposited on the end of a rotating silica rod which is slowly withdrawn from the reaction zone as material is deposited. A porous preform is thus formed. This passes upwards into a furnace where it is first dried to remove residual water and then sintered to a transparent glassy rod. The VAD process suffers the drawback of being an inherently wet process with water incorporated into the porous material. If not removed the hydroxyl ions would induce considerable excess absorption loss around 1400nm in the fibre. However,

the dehydration process is highly effective<sup>13</sup> and fibre containing less than 0.8ppb can be prepared. The preform is dried in an atmosphere of chlorine whilst being heated to approximately 800°C, a temperature sufficient to remove both the physically and chemically adsorbed water. After dehydration, the preform is consolidated by heating to approximately 1700°C in an inert atmosphere of helium. Helium is used to facilitate outdiffusion of gases during sintering.

### Fibre drawing



Preforms prepared by either technique are drawn into fibre by the process is shown in Figure 3. The system comprises a preform support, furnace, diameter monitoring apparatus, coating applicator and furnace, capstan and take-up drum. The preform is fed vertically down into the furnace (at a constant speed) where it is heated to approximately 2000°C, under accurately controlled conditions. Fibre of predetermined diameter is drawn down from the region of softened glass. Under stable drawing conditions the rate at which the volume of glass is withdrawn from the furnace is equal to the rate at which the preform is fed into the hot zone. The fibre diameter (usually  $125\mu\text{m} \pm 0.25\%$ ) is controlled by changing the pulling speed of the

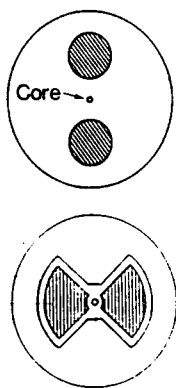
Figure 3. capstan based on information received from the diameter monitor directly under the furnace. A liquid coating is applied to the fibre and cured during the drawing process. This material is usually a ultra violet curable acrylate and serves the protect the fibre from dust which causes surface flaws and hence weakness.

### Novel fabrication processes

The production of fibre and preforms for devices can be separated into two categories, those which rely on geometric modification to the fibre configuration and those in which novel materials are employed. The design criteria for these two categories are discussed separately.

### Geometric Modifications

Both the polarising and polarisation maintaining characteristics of a fibre are determined by altering the guidance characteristics of the fibre across different axes. This is effected by introducing asymmetry of core diameter or refractive index. Form or shape birefringent fibres include those with elliptical cores, whose polarisation dependent guidance behaviour arises from differences in the size of the waveguide in two orthogonal directions. These preforms are prepared conventionally until the final collapse passes. At which point a partial vacuum is applied<sup>14</sup> inside the substrate tube, rather than the slight positive pressure normally used. This causes the core to collapse elliptically, whilst the low viscosity of the  $B_2O_3$  rich cladding glass allows the overall preform to remain circular. Control of core geometry is relatively poor, with this technique. Furthermore, the transmitted mode profile suffers the same ellipticity as the core, resulting in poor coupling with conventional, single mode fibres. It

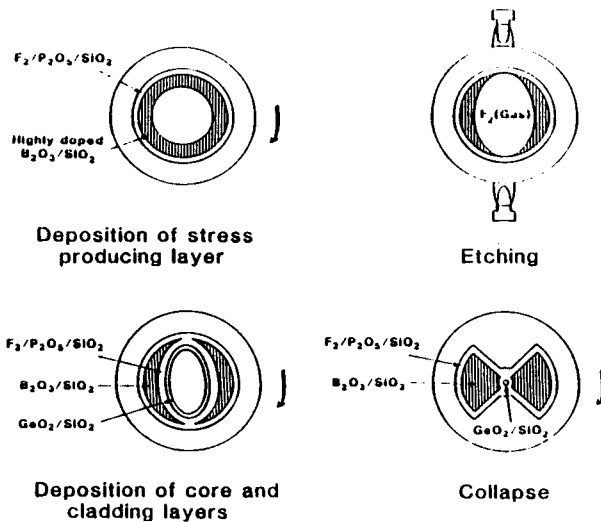


is preferable, therefore, to maintain a circular core and change the effective refractive index elasto-optically, by applying asymmetric stresses to the core. A number of techniques have been developed to manufacture stress birefringent fibres, including the elliptical cladding, PANDA and Bow Tie designs, of which the latter two as shown in Figure 4. Stress symmetry is achieved by placing more glass of high thermal expansion along one of the core axes. Borosilicate glass or alumina rods are usually employed. Elliptical cladding and PANDA fibres are pulled from preforms which are modified after conventional manufacture.

Figure 4. A standard preform, with low viscosity, high thermal expansivity borosilicate glass surrounding the core is used to form elliptical cladding birefringent fibre. The conventional circular preform is heated until slightly soft and then squashed by passing through steel rollers. The process deforms the substrate glass and the borosilicate cladding but the low viscosity of  $B_2O_3$ - $SiO_2$  results in this layer taking the strain and the core remains circular. A three to one ratio of fibre diameters along the major and minor axes is typical, allowing easy identification of the two birefringent axes.

PANDA and Bow Tie fibres are not 'keyed' in this manner, but higher stress asymmetry can be achieved, and hence increased birefringence. The rod in tube method is used to make PANDA fibres. Two

circular holes are drilled on opposite sides of the core of a standard preform, forming two tubes.  $\text{Al}_2\text{O}_3$  rods are then inserted, these have higher expansion coefficients than the silica substrate but can also withstand the temperatures encountered during fibre drawing. Fibre is drawn from the composite under high tension to avoid deformation of the core region and the preform geometry is maintained in the fibre form. PANDA fibres gives improved birefringence over the elliptical cladding design, however, theoretical analysis<sup>15</sup> shows that the optimum geometry consists of a "bow tie" of  $90^\circ$  sectors, with those sectors close to the core. A technique has been developed to manufacture this



design by modifying the MCVD process, and is shown schematically in Figure 5. Initially the process is conventional, with cladding material ( $\text{P}_2\text{O}_5/\text{F}_2/\text{SiO}_2$ ) being deposited, followed by deposition of the borosilicate glass which is to form the stress sectors. Rotation is then stopped and a narrow section of tube is heated, as shown. Fluorine rich gases e.g.  $\text{SF}_6$  flowing inside the tube react with the hot region and etch off some of the deposited borosilicate glass. This is repeated on opposite sides of the tube until all the

of the tube until all the  $\text{B}_2\text{O}_3\text{-SiO}_2$  has been removed. Fortunately, this is easily seen by eye; when hot the  $\text{B}_2\text{O}_3\text{-SiO}_2$  glass appears green whilst the cladding glass seems pink. Conventional deposition is then restarted, with the deposition of a silica buffer layer, followed by the core. With reference to the figure it may be seen that the bows develop during the collapse process, giving near optimum geometry.

In addition to the polarisation maintaining characteristics described, single mode fibre can be made to be actively polarising, where regardless of the launched polarisation, only one plane of polarisation propagates, with the others being lossy. Again the preform is modified after conventional manufacture. One side of the cladding

glass is removed by milling so part of the core is close to the surface. The modified preform may then be inserted into a tube, giving a D-shaped hollow inside the new composite preform. Liquid metal is injected into the hole and provides a loss mechanism by interacting with the evanescent field of one polarisation, the orthogonal one being unaffected. Extinction ratios of around 40dB have been demonstrated over a wide wavelength range<sup>16</sup>. The exposed D configuration (without surrounding tube) is of interest for environmental sensing, with the evanescent field interacting with the external medium.

An almost unlimited range of geometrically altered fibres can be prepared by using the rod in tube technique where rods may be inserted into standard preforms, as described for PANDA fibres. Alternatively, one or more preforms may be inserted into tubes to form fibres with offset cores, twin or multiple cores etc.. Such designs are primarily being tested for sensors or optical switches and the important design criteria are usually the relative separation of the core and the extent to which they are offset. These novel designs are drawn to fibres under high tension to prevent deformation by reducing the furnace temperature by approximately 100°C.

In addition it is possible to use the fibre drawing process to modify the fibre design, by spinning the preform during the drawing process. Standard step index preforms may be spun very quickly to null out any inhomogeneities in the core shape<sup>4</sup>. The core thus produced has a truly circular core and, therefore, negligible intrinsic birefringence, making any signal polarisation highly sensitive to environmental changes, such as pressure. Alternatively, the offset cores of composite preforms will precess when the preform is spun during the draw, resulting in a helical or quasi circular birefringent fibre<sup>17</sup>. Bow tie preforms have also been spun rapidly during the draw, at typical spin rates of 1800rpm. The net effect is to rotate the stress sectors rapidly about the core thereby producing a series of quarter wave plates. This rotates the preferred plane of linear polarisation, so that nearly circularly birefringent fibre is produced. The improved characteristics of this type of circularly birefringent fibre over the helical design have led to the development of high performance current monitors<sup>18</sup>, sensitive to around  $\text{lmA/Hz}^{1/2}$ .

In summary, a wide range of geometric modifications have been demonstrated, primarily with a view to achieving novel polarisation guidance behaviour. Many configurations are now commercially available and practical devices a reality. A selection of the manufacturing processes have been described to indicate the range of possible fibre

designs.

### *Materials modifications*

Silica was chosen for telecommunications grade optical fibres because of its low losses and both mechanical and chemical stability. Its nonlinear coefficients are correspondingly small. However, with increasing interest in fibres for sensors and devices other criteria become more important, thus novel glass structures are sought. Multicomponent glasses have been developed over many years to have highly nonlinear behaviour and optimal laser host characteristics. In addition, the low phonon energy (lattice vibration frequency) of fluoride glasses offers potential for modified dopant characteristics. However devices based on doped silica fibres have good compatibility with telecommunications fibres. Each of these categories of material is currently under investigation and the manufacturing processes are described here.

Multicomponent glasses cover a wide range of materials, from those based on oxides through to chalcogenide or semiconductor glasses. Many have highly nonlinear behaviour with resultant high losses, typically of order 1dB/m. These losses are not significant since short fibre devices are made. There are, however, considerable problems associated with preparing optical fibres from multicomponent glasses. In many cases the limits of glass formation are reached, with dopants being added to stabilise the structure and manufacture is often complicated, requiring controlled cooling and atmosphere to prevent cracking or devitrification. The rod in tube method is the preferred route to fibre manufacture from multicomponent glasses. Bulk samples are prepared by the normal methods then rods and tubes are drilled to form the composite preform. Due to the large expansion coefficients, wide range of melting parameters and chemical stability of many of these materials it is not sufficient to match only the refractive index parameters.

It has been determined<sup>7</sup> that differences in thermal expansion coefficients of around 15% may be tolerated. The range of acceptable fibre softening temperatures has also been assessed empirically. A large mismatch is acceptable if the core has the lower softening temperature. Indeed, it is feasible to draw fibre from a composite preform in which the core is molten.

The stringent cooling requirements needed for bulk glasses are not important in the drawing process since fibre is quenched (the glass is frozen in a disordered, liquid structure). In bulk systems a balance is required between rapid cooling, conducive to glass formation and



minimising the thermal gradient, which could cause cracking. During fibre pulling glass is quickly removed from the hot zone (many metres per minute) and the sample's cross sectional area is small ( $\sim 10^{-8}\text{m}^2$ ).

The third requirement for core and cladding compatibility is chemical stability. Some glasses become very reactive when molten and so will degrade the core/cladding interface. Also, multicomponent glasses contain a high concentration (many percent) of network modifying dopant ions which become mobile at high temperature. These ions may diffuse across the core cladding interface so changing the composite structure and refractive index of the two components.

To minimise these problems nearly identical glasses are preferred for core and cladding glasses, the core glass having slightly higher concentrations of dopant to increase the refractive index and nonlinear characteristics. Considerable research is still required in this area but useful fibre devices have already been demonstrated<sup>7</sup>.

### *Fluoride glasses*

Optical fibres made from heavy metal fluorides have been under investigation for many years, with a view to developing ultra low loss ( $<0.01\text{dB/km}$ ) fibres operating in the range 2-10 $\mu\text{m}$ . Such losses have not been achieved in long lengths of single mode fibre due to scattering losses between core and cladding and phase separation of the glass. The theoretically loss losses arise from the low phonon energy of lattice vibration frequency of the glass structure which also has potential benefits for active fibres, in particular upconversion lasers. Here, several pump photons are absorbed by the dopant, exciting the ions to progressively higher energy levels before emission. The resultant emission frequency is higher than that of the pump, making this an attractive route for generating blue emission from readily available red or infrared laser diodes.

Fluoride fibres are based on ZBLAN glass,  $\text{ZrF}_4\text{-BaF}_2\text{-LaF}_3\text{-AlF}_3\text{-NaF}$  and lanthanum has similar chemical performance to the rare-earth laser ions so it is relatively straight forward to replace La during the standard fabrication procedure. High purity vapour phase preparation has not been developed due to lack of precursors, although simple fluoride glasses based on organometallics have been demonstrated. These materials are highly toxic and considerable care is required during manufacturing. Alternative techniques have therefore been developed. Metal fluorides are prepared in the liquid phase by fusing together powdered raw materials<sup>10</sup>. These purified, water-free, starting materials are generated by standard methods. A preform is cast from the liquid

glass. A tube of cladding glass is formed first; A heated mould is partly filled with cladding glass then tipped to the horizontal position and spun rapidly to generate a tube of uniform thickness and the glass is rapidly quenched to avoid crystallisation. The core glass is then poured into the tube of cladding material to form the preform.

The fibre drawing process is similar to that used for silica based fibres but the drawing temperature is around 300°C and an inert, water-free atmosphere is essential to prevent surface crystallisation.

### *Silica based glasses*

The nonlinear coefficients of silica are small and many dopant ions, including the transition metals and rare-earths are not highly soluble in silica but the loss losses achievable in such fibres make them attractive in long devices or distributed sensors. The main advantages of silica based doped fibres over other categories is the excellent compatibility with telecommunications systems and a widely established manufacturing technology. The rare-earth ions have been studied most extensively to date and several methods for incorporating these ions have been developed, based both on VAD and MCVD.

High vapour pressure organometallic precursors have been employed in the conventional MCVD process, with the rare-earth ion being transported in the vapour phase to the reaction zone in the same manner as the glass forming material. The technique is simple to implement but the precursors are not widely available, are toxic and very expensive.

An alternative, widely used method, exploits the high purity achievable via the vapour phase, without needing unusual reagents<sup>8</sup>.

Solid, low vapour pressure rare-earth halide precursors are used and

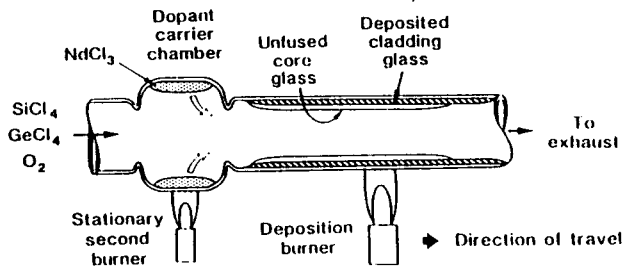


Figure 6.

incorporated in the fabrication process as shown schematically in Figure 6. A dopant chamber is located upstream of the substrate tube and the precursor is fused to the chamber walls before fabrication commences. During conventional deposition of the core glass the dopant chamber is heated to approximately 1000°C to achieve a significant vapour pressure of rare-earth halide. Accurate temperature control is essential for control of precursor vapour pressure and hence dopant

incorporated in the fabrication process as shown schematically in Figure 6. A dopant chamber is located upstream of the substrate tube and the precursor is fused to the chamber walls before fabrication commences. During conventional deposition of the core glass the dopant

concentration. This vapour is transported to the reaction zone with the glass forming reagents where it is oxidised and deposited with the silica. Collapse and fibre drawing are conventional. Fibres containing a few hundred parts per  $10^6$  are readily prepared in this manner. Losses approach those of telecommunications grade fibres, in the regions away from the dopant absorption bands, as shown in Figure 7, for a fibre doped with  $\text{Er}^{3+}$ .

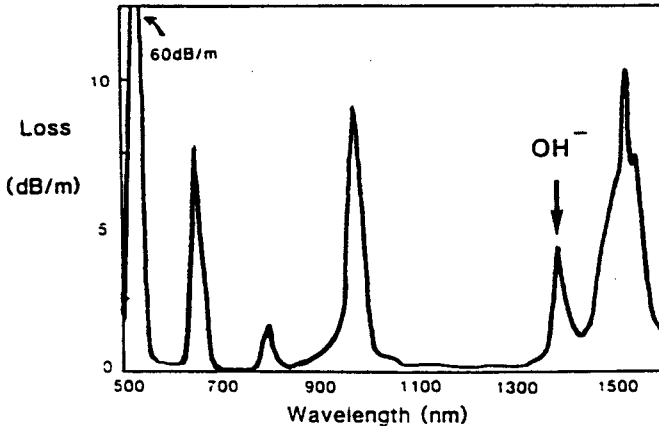


Figure 7.

whereby the ions are added from solution<sup>9</sup>, overcomes these difficulties. Since this is not a vapour phase process there is an increased risk of impurity contamination but, by careful preparation, high quality fibres with high dopant concentrations (many mole percent) and baseline losses of order 5dB/km are produced. Very simple modifications to the MCVD process allow the incorporation of dopant ions from solution. The conventional fabrication process is followed until the core layer is deposited. A reduction in burner temperature of around 500°C allows the formation and deposition of the oxide soot without subsequent fusion of this porous layer. The frit is then immersed in a dilute solution of the dopant ions. Additional glass forming of glass modifying reagent can also be added at this stage, for example alumina can be included from  $\text{AlCl}_3$  dissolved in the solvent. Water or alcohols<sup>21</sup> are most commonly used as the solvent since the halide precursors are soluble in both and they are safe to handle. After soaking, excess solution is removed, the tube replaced in the lathe and the porous layer dried. Dehydration is based on that used in the VAD method, as described earlier. Chlorine is passed along the inside of the tube which is heated to approximately 1000°C, so

Back scatter measurements confirm that the dopant is incorporated evenly in the glass. Unfortunately this method is not very versatile since it is difficult to add high concentrations of dopant or to incorporate several different types of ions simultaneously in predetermined ratios.

An alternative method,

evaporating residual solvent. The dopant ions are not volatile at this temperature and remained trapped in the frit. Fusion of the soot layer occurs at the conventional temperature and collapse of the tube into the preform is also standard. Normal fibre drawing methods can be

applied to these doped preforms. Fibres containing around 4mol% of rare-earth dopant have been produced in this manner and codoped fibres are readily fabricated. The spectral characteristics of a fibre containing around 800ppm  $\text{Er}^{3+}$  and 17,000ppm  $\text{Yb}^{3+}$  in an  $\text{Al}_2\text{O}_3$ - $\text{SiO}_2$  core fibre are shown in Figure 8, with erbium, ytterbium and aluminium all added from solution.

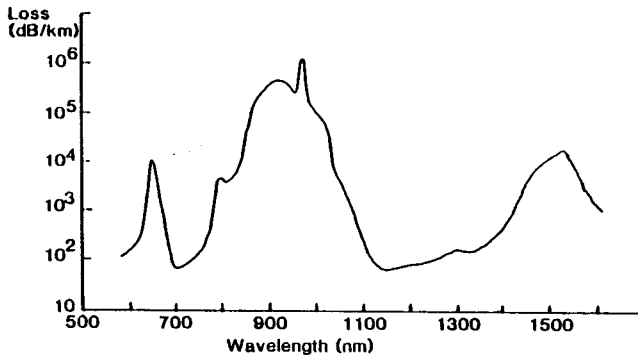


Figure 8.

### Conclusions

In summary a wide range of techniques have been developed to produce fibres of novel geometric or materials characteristics of which the most notable have been described here. Some processes are now employed commercially whilst others are still being developed. Many interesting devices have already been demonstrated and further research in fabrication will lead to an even greater range of practical fibre based sensors and devices.

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