A3.2 Spectroscopy of neodymium-doped silica

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A INTRODUCTION

Nd^{3+} ion lasers in glass and crystal form have been the workhorses of the laser industry for three decades. Silica exhibits excellent optical and mechanical properties thereby providing an excellent host for Nd^{3+} glass lasers and amplifiers. In a silica fibre environment the spectroscopic properties of Nd^{3+} doped silica enable the construction of highly efficient devices with low pump power requirements. In this Datareview the properties of Nd^{3+} doped silica are summarised with emphasis given to the impact of the spectroscopy on the realisation of practical devices.

B ABSORPTION AND FLUORESCENCE

Representative absorption and fluorescence spectra [1], taken from a germano-silicate Nd^{3+} doped silica fibre, are shown in FIGURE 1. They show strong absorption peaks centred at 590 nm, 750 nm, 805 nm and 890 nm, and fluorescence peaks around 950 nm, 1090 nm and 1320 nm. The three longest absorption wavelengths coincide with the operating wavelengths of semiconductor lasers. Therefore convenient low cost pump sources are available. The most commonly employed is the 805 nm wavelength due to the lower cost and production yield of AlGaAs lasers at that wavelength. While efficient lasing operation has been demonstrated at 940 nm [2,3] on the $^4F_{3/2}$-$^4I_{9/2}$ transition, and at 1088 nm on the $^4F_{3/2}$-$^4I_{11/2}$ transition [4,5], operation at the technologically important 1300 nm wavelength is frustrated by the presence of an excited state absorption band centred near the fluorescent peak from the $^4F_{3/2}$ metastable level to the $^2G_{9/2}$ and $^2G_{7/2}$ levels [6]. These levels correspond to ground

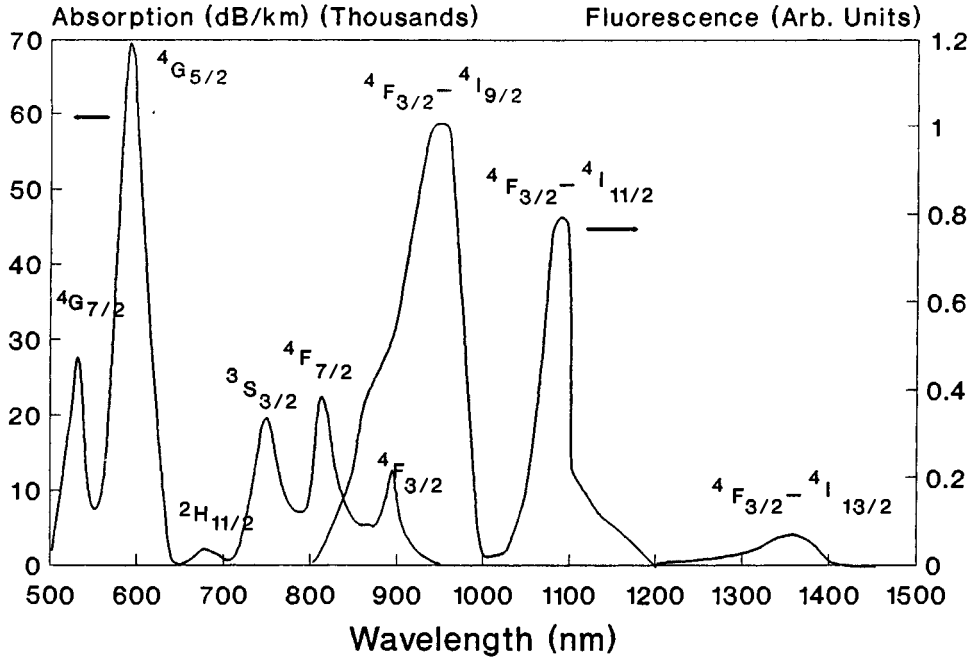

FIGURE 1 Absorption and emission spectra of Nd^{3+} doped germano-silicate fibre (after Townsend [1]).

state absorption in the 500 - 550 nm band. The excited state absorption comprises a near continuum extending from 1100 to 1380 nm. This shifts the oscillating wavelength away from the fluorescent peak towards 1400 nm and therefore eliminates Nd^{3+} doped silica as a host medium for a 1.3 μm amplifier for telecommunications.

C EFFECT OF GLASS COMPOSITION

It is important to consider the effect of glass composition on the properties of Nd^{3+} doped silica. Silica based glass fibre can be fabricated with a range of host compositions large enough to affect fluorescence and absorption peak positions, effective line shapes, line strengths and other spectroscopic properties. The composition is particularly important where a high concentration of Nd^{3+} ions is required, for example in a cladding pumped structure, or where a short cavity is required, for example in a q-switched or single-frequency device. High concentration can lead to devitrification, rare-earth microclustering and concentration quenching, all of which can severely affect the performance of an amplification device.

C1 Alumina and Phosphorus Co-Doping

It is well known from an extensive study of bulk silica by Arai et al [7] that co-doping with alumina and phosphorus has several beneficial effects on the properties of Nd^{3+} doped silica. These co-dopants can significantly increase the solubility of rare-earths in silica, inhibit devitrification and prevent clustering of rare-earth ions.

In a pure silica or germano-silicate host with high Nd^{3+} ion concentration a large proportion of the ions are unavailable for laser action because of microclustering and additionally the transition shows a lower fluorescence intensity than in virtually any other host. However the addition of a small amount of alumina or phosphorus virtually eliminates the fast decay component associated with the microclusters and increases the fluorescence intensity ratio of the $^4F_{3/2}$ to $^4I_{11/2}$ transition from 0.7 to 1.5. An optimised ratio of Al/Nd concentrations of between eight and ten has been determined. In the case of phosphorus doping a ratio of approximately 15:1 is appropriate.

As well as dispersing the microclusters these additional co-dopants also enable realisation of higher average Nd^{3+} concentrations without serious lifetime quenching occurring. This is illustrated in FIGURE 2 for the case of optimised Al co-doping.

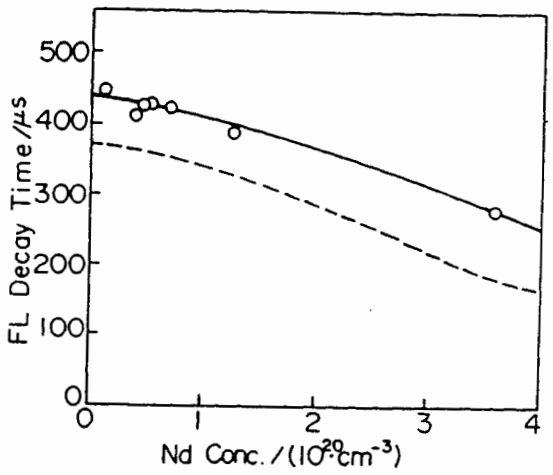

FIGURE 2 The decay time of the $^4F_{3/2}$-$^4I_{11/2}$ fluorescence in an alumino-silicate fibre as a function of Nd^{3+} concentration. The broken line represents that of an ED-2 silicate glass (after Arai et al [7]).

D PRACTICAL DEVICES

D1 $^4F_{3/2}$-$^4I_{11/2}$ Transition

The first demonstration of diode pumped single-mode operation of this transition was in 1985 [4]. The early publications reported lasing at 1088 nm in germano-silicate fibres. The efficiency of these devices was severely limited by microclustering. Currently however an alumino-silicate host is almost universally used for the reasons outlined in the previous section. Highly efficient laser sources pumped by high power diode bars have enabled the output power to be scaled to several tens of watts at 1060 nm [8,9].

D2 $^4F_{3/2}$-$^4I_{9/2}$ Transition

This is potentially a very important transition with applications in remote sensing, non-linear optics (e.g. frequency doubling to the blue) and as a high power pump for Yb or ErYb fibre lasers. The transition is three-level in nature and as such requires that the ground state absorption is bleached before gain can be achieved. Since the first demonstration of this lasing on this transition in 1987 [2], there has been little reported until 1997 when Dragic et al proposed such a source for remote sensing of water vapour. There have so far been no reports of power scaling this transition by cladding pumping. This is because under the weak pumping conditions inherent when double-clad fibre is employed, the strong gain at 1060 nm will lead to ASE self saturation thus suppressing the 940 nm transition irrespective of any wavelength selecting mirrors. However a recently proposed gain limiting structure with annular doping may permit future power scaling of this transition [10].

D3 $^4F_{3/2}$-$^2I_{11/2}$ Transition

As mentioned in Section B lasing on this transition is frustrated by signal excited state absorption. Nevertheless lasing at 1364 nm has been demonstrated in a phospho-silicate Nd^{3+} doped fibre [11]. The slope efficiency was very low however and the gain peak still at too long a wavelength for a practical telecoms amplifier. Further developments concerning this transition have therefore employed compound glasses such as fluorides and phosphates rather than silica based glasses.

E CONCLUSION

Nd^{3+} doped silica provides an excellent host for fibre lasers and amplifiers operating on the four level transition around 1060 nm. Very low threshold can be combined with the capability for power scaling to several tens of watts. The engineering of sources on the three level transition at 940 nm is hindered by competing amplified spontaneous emission at 1060 nm and on the four level transition around 1320 nm by a broadband excited state absorption between 1100 nm and 1380 nm. In most cases an alumino-silicate host is preferred because of improved rare-earth solubility and dispersion of microclusters.

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PART A: SILICA

CHAPTER A4

OPTICAL FIBRE MANUFACTURE

A4.1 Preparation and preform fabrication of silica optical fibres
A4.2 Drawing of silica optical fibres
A4.3 Loss of silica optical fibres
A4.4 Reliability of silica optical fibres

A4.1 Preparation and preform fabrication of silica optical fibres

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A INTRODUCTION

Silica optical fibres are in widespread use for optical telecommunications. These fibres result from volume production using chemical vapour deposition techniques (CVD) such as modified chemical vapour deposition (MCVD), vapour-phase axial deposition (VAD) or outside vapour deposition (OVD). Moreover, the function of optical amplification can be attached to an optical fibre, by doping rare-earth ions into its core glass. The rare-earth doped fibre then becomes an optical amplifier, or fibre laser. Among others, an erbium doped fibre (EDF) serves as a practical, optical amplifier at 1.55 μm. It is usually fabricated using a modification of the CVD technique.

Section B gives a brief explanation of the fabrication of glass and preform for silica optical fibres, and Section C describes rare-earth doped silica optical fibres.

B FABRICATION OF GLASS AND PREFORM FOR SILICA OPTICAL FIBRES

B1 Silica-Based Glass for Silica Optical Fibres

The silica optical fibre consists of a core made from silica-based glass, and a cladding made from pure silica glass or silica-based glass. The silica-based glass for the core includes a dopant for increasing the refractive index; the silica-based glass for the cladding includes a dopant for decreasing the refractive index. By way of illustration, FIGURE 1 presents dopant concentration versus refractive index [1]. Typical dopants used are GeO_2, TiO_2, Al_2O_3 and P_2O_5 for increasing the refractive index, and B_2O_3 and F for decreasing it. These dopants also allow adjustment of the thermal expansion and softening temperatures of glass. In reality, the limits of low-loss fibre fabrication have narrowed the options down to a core of GeO_2 doped silica and a cladding of F doped silica or pure silica-glass.

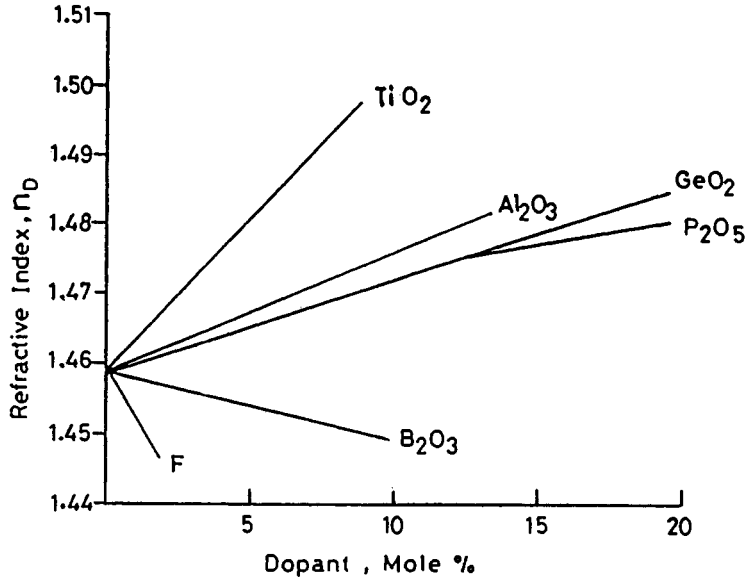

FIGURE 1 Refractive index of common dopants for silica (from [1]).

B2 Fabrication of Preform for Silica Optical Fibres

Fabrication of preforms can be accomplished using several techniques: MCVD [2], VAD [3], OVD [4] and plasma chemical vapour deposition (PCVD) [5], for example. Each method relies on CVD that produces glass from gaseous raw materials. By way of illustration, SiO_2-GeO_2 glass results from a gas mixture of $SiCl_4$-$GeCl_4$ under reaction according to EQNS (1), (2), (3) and (4). The gas constituents are varied to produce various glass compositions.

[oxidation]

$$SiCl_4 + O_2 = SiO_2 + 2Cl_2 \quad (1)$$

$$GeCl_4 + O_2 = GeO_2 + 2Cl_2 \quad (2)$$

[flame hydrolysis]

$$SiCl_4 + 2H_2O = SiO_2 + 4HCl \quad (3)$$

$$GeCl_4 + 2H_2O = GeO_2 + 4HCl \quad (4)$$

MCVD and PCVD are the processes that apply the oxidation reaction in a rotating silica-glass tube, on the inside wall of which fine glass particles of oxide (e.g. SiO_2, GeO_2) deposit in the form of soot. Subsequently, a traversing burner makes the soot layer consolidate into transparent glass. Glass layers, built up in large numbers, are subjected to intense heat so that the silica-glass tube collapses into a solid preform.

VAD and OVD apply the flame hydrolysis reaction and each includes two fabrication stages. The first stage of flame hydrolysis creates a porous body (called 'soot preform') and the second one makes the soot consolidate into a transparent glass preform. In the soot processes, OH^- impurities contained in the soot have to be removed. For this purpose, the soot is heat-treated in a chlorine or thionyl chloride-containing gas [6].

For the CVD, halides such as $SiCl_4$, $GeCl_4$ or $POCl_3$ are used as raw materials. Each halide has high vapour pressure as shown in FIGURE 2 [7]. The magnitude of each vapour pressure exceeds those of transition metal sources, a major cause of loss in an optical fibre. For example, taking $FeCl_3$, a source of Fe impurity ions, the pressure of any of the raw materials is as high as ten million to a billion times ($10^{8\text{-}10}$) that of $FeCl_3$. The significant differential vapour pressures ultimately eliminate transition metal impurities (Fe etc.) in the fabrication of glass by CVD. Thus, CVD is capable of creating high-purity optical fibres, and provides a route for removal of OH^- impurities. Consequently, CVD is judged to be ideally equipped for the fabrication of low-loss fibres.

B3 Novel Fabrication Processes for Silica Optical Fibres

Apart from the CVD-based methods, sol-gel techniques [8] and mechanically shaped preform (MSP) [9] have been proposed for fibre fabrication. However, these have yet to produce low-loss fibres. What have been recently proposed are hybridised processes: colloidal sol-gel processes [10,11], cold isostatic pressing processes (CIP) [12-16] and extrusion forming processes [17]. The hybridised process is one in which a preform is formed by overcladding a CVD-derived core rod with commercially available silica powder. On analysis, the CVD-derived core rod facilitates the production of low-loss fibres, while the commercial silica powder paves the way for less costly fibres.

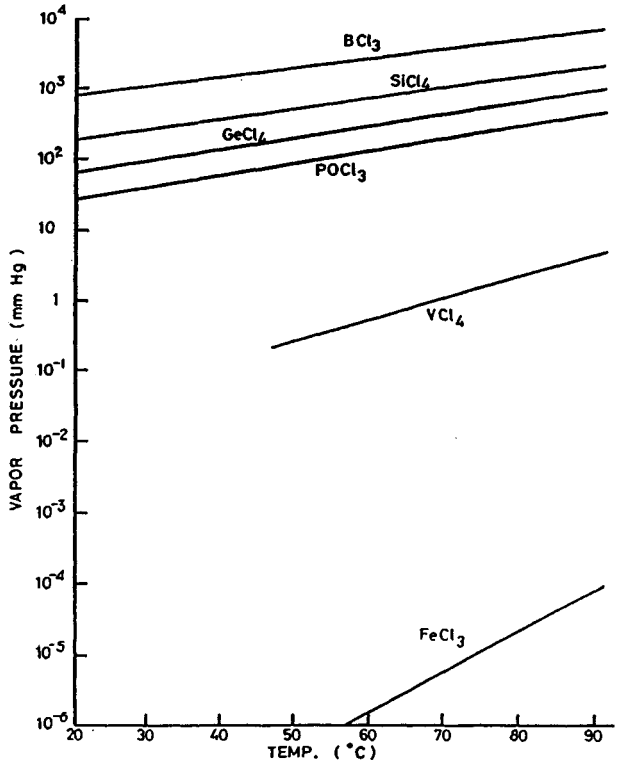

FIGURE 2 Relation of vapour pressures for metal halide additives and potential impurities (from [7]).

C FABRICATION OF GLASS AND PREFORM FOR RARE EARTH-DOPED SILICA OPTICAL FIBRES

C1 Silica-Based Glass for Rare Earth-Doped Silica Fibre

A rare-earth doped silica fibre consists of a core and cladding, with the core also doped with rare-earth ions (e.g. Er^{3+}, Nd^{3+}, Pr^{3+}). It has been found that higher concentrations of rare-earth ions incur clusters of rare-earth ions [18]. These clusters cause the characteristic degradation of optical amplification, called concentration quenching. Nevertheless, where there is some scheme to prevent the growth of clusters of rare-earth ions, high concentrations of rare-earth ions can be applied to provide amplification improvement. Arai et al [19] pointed out a host glass of Al_2O_3-SiO_2, P_2O_5-SiO_2, in which rare-earth ions are soluble enough to appreciably lessen clusters of rare-earth ions, and in turn high concentrations of rare-earth ions become possible. Much research into the host glass has since been carried out, and Miniscalco [20] gave a review of material-dependent properties influencing fibre amplifiers. In fact, it was reported that host glasses GeO_2-Al_2O_3-SiO_2 [21] and Al_2O_3-SiO_2 [22], used for erbium doped fibre (EDF), were proven to be receptive to high concentrations of Er^{3+}-doping and bring out high conversion efficiencies. Likewise it was reported that the Al_2O_3 content of Al_2O_3-SiO_2, which is easy to crystallise, could be made uncrystallisable by adding a small amount of P_2O_5, even with a high Al_2O_3 concentration [23]. Ainslie et al [24] elucidated the fact that Al_2O_3-P_2O_5-SiO_2, even if heavily doped with Er^{3+} or Nd^{3+}, would incur no growth of clusters of rare-earth ions. Thus, while the dopant GeO_2 can be used in silica optical fibres, P_2O_5 or/and Al_2O_3 is also frequently used as a dopant or a co-dopant with GeO_2 for heavy doping of rare-earth ions. Further details regarding the doping of silica fibres can be found in [25-35].

C2 Fabrication of Preform for Rare Earth-Doped Fibres

One of the simplest techniques for fabricating rare-earth doped fibre preforms refers to the rod-in-tube method, which has long been used because of its simplicity [36,37]. This method consists of two steps, to charge a silica-glass tube with core glass and to collapse the charged tube into a solid preform, and is still used as a technique to fabricate heavily Er^{3+}-doped fibres [38].

It would be preferable if rare-earth doped fibres could be fabricated by CVD, as silica optical fibres can be. Some organometallic compounds having a high vapour pressure were prepared [39], and were used for fibre fabrication [40,41], but they are not widely used. Research continues in this area.

Gas-phase doping processes can handle a solid rare-earth source itself. Poole et al [42] announced a modification of MCVD, followed by its alternative means, e.g. porous generator [43] and ampoule [44]. Meanwhile, a modification of the soot process was presented, i.e. sintering core soot subjected to vaporised compounds [45]. In the light of high doping concentrations, the flash-condensation technique was investigated [46].

The solution doping process soaks a porous body in a solution containing rare-earth compounds to dope rare-earth ions. This process was used to investigate the loss resulting from transition metal impurities [47-49], and recently has been the most commonly used method.

FIGURES 3 and 4 illustrate current general methods. FIGURE 3 starts off with a soot preform made in a VAD process. First, the soot is soaked in an alcoholic solution containing rare-earth halide, impregnated with the rare-earth compound, dried and consolidated into a transparent glass core rod. Finally, a cladding of glass is formed on the surface, where a rare-earth doped preform takes shape. FIGURE 4 refers to an MCVD-based method. The soot layer for the core is deposited at low temperature on the inside wall of a silica-glass tube, which is in turn soaked in an alcoholic solution of rare-earth halide, to make the soot layer impregnated with rare-earth material. After being dried, the silica tube is collapsed into a rare-earth doped preform. The CVD method is substantially capable of Al_2O_3 doping. However, the Al_2O_3 source for doping, aluminium chloride, being relatively low in vapour pressure, is often applied in the form of solution doping, in which aluminium nitrate can also be used.

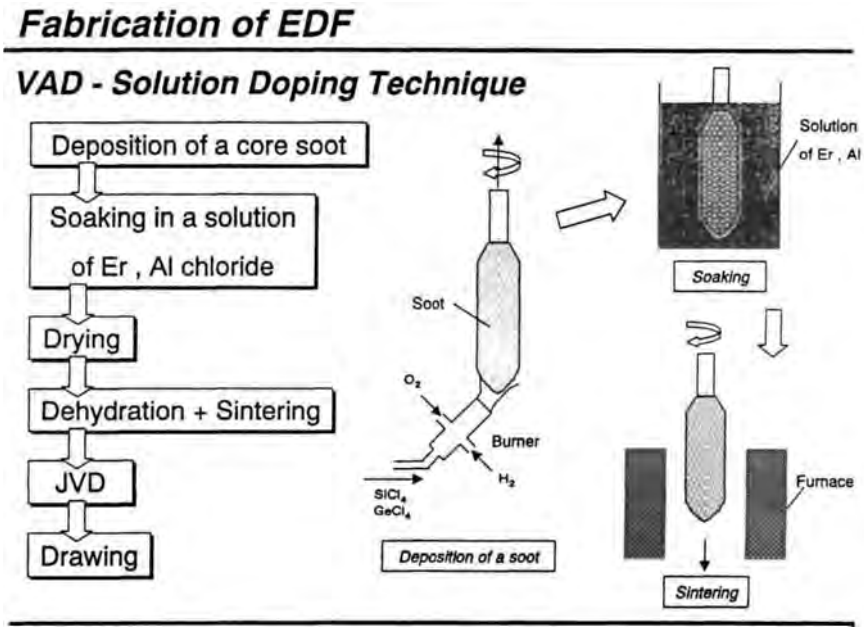

FIGURE 3 VAD-solution doping technique for fabrication of EDF preform.

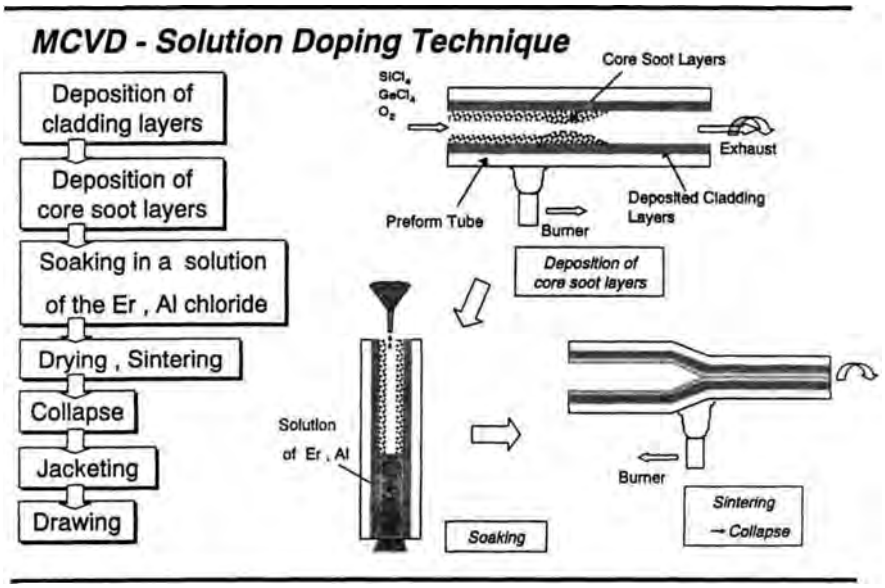

FIGURE 4 MCVD-solution doping technique for fabrication of EDF preform.

Meanwhile, a few more techniques have been proposed for solution doping, for example the aerosol delivery technique [50,51] using an aerosol as a carrier of the doping source to feed into the reaction zone, and the sol-gel technique [52] for forming a rare-earth glass layer on the inside wall of a silica-glass tube by dipcoating.

D CONCLUSION

Current silica optical fibres and rare-earth doped fibres have been reviewed regarding their technical progress, with a focus on the material and production technology.

Silica glass fibres are under volume-production, and their production method seems to have been finalised. However, less costly production means continue to be researched far and wide. For rare-earth doped fibres, the Er^{3+}-doped fibre has come into practical use and is fabricated by solution doping. Even more recently, research has continued the improvement of fibres from the aspects of fibre material and configuration. Also, several technical issues have yet to be resolved, namely practical high doping methods and cost reduction.

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