SOLUTION-DOPING TECHNIQUE FOR FABRICATION OF RARE-EARTH-DOPED OPTICAL FIBRES

A solution-doping technique is reported for the reproducible fabrication of low-loss optical fibres containing up to 4000 parts in 10^6 of rare-earth ions. The method produces excellent dopant uniformity and is sufficiently versatile to allow codoping with different ions.

Fig. 1 *Absorption spectrum of fibre containing ~ 3800 parts in 10^6 Nd^{3+}*

Introduction: Rare-earth-doped optical fibres are of great interest for a variety of applications including fibre lasers[1] and sensors.[2] We have previously reported[3] a gas-phase doping technique which allows the fabrication of mono- and multimode fibre containing small quantities of rare-earth dopant in the core. However, with this method it has proved difficult to achieve more than 900 molar parts in 10^6 of dopant in the core region as required, for example, to construct short, highly doped fibre lasers. In addition, very precise temperature control is required to maintain dopant concentration, and there is only limited potential for the introduction of co-dopants (e.g. Yb^{3+} with Er^{3+}).

We report here an adaptation of earlier work[4] which overcomes these disadvantages. Using solution doping, accurate control of dopant levels is easily achieved and higher dopant levels than were previously possible have been obtained. Results are presented for single and codoped fibres. In addition, the technique used to remove OH^- impurities during preform fabrication has been investigated in detail.

Fibre fabrication: The fibre preform is fabricated by a modification to the MCVD technique which allows the addition of dopant ions from solution, rather than from the vapour phase as described previously.[3]

A conventional cladding is first deposited (typically an $SiO_2/P_2O_5/F$ glass) after which the core layers are deposited at a reduced temperature so as to form an unsintered porous soot. The tube is subsequently removed from the lathe and immersed for periods of up to 1 h in an aqueous solution of the required dopant precursor (typical concentration 0·1 M). Any soluble form of the required dopant ion may be employed as the solute, although, for experimental convenience, work has concentrated on the rare-earth halides. Following immersion, the tube is rinsed with acetone to remove excess water and replaced in the lathe.

Owing to the aqueous nature of the solution-doping technique, an effective dehydration process is essential to produce low-loss fibres. The core drying and fusion procedure previously reported[5] was used. The preform is heated to around 600°C by a traversing burner, and chlorine gas is flowed through the tube. The core layer is subsequently sintered and the preform collapsed in the usual manner. Fibre drawing is conventional.

Results and discussion:

(a) Single dopant ions: A number of fibres containing various rare-earth ions (Nd^{3+}, Ho^{3+}, Eu^{3+}, Er^{3+}, Yb^{3+}, Dy^{3+}) have been fabricated with dopant concentrations between 1 and 4300 molar parts in 10^6. The technique has been used to fabricate both mono- and multimode fibres and also highly birefringent 'bow-tie' fibres. The absorption spectrum of a single-mode fibre containing 3800 parts in 10^6 of Nd^{3+} is shown in Fig. 1. The curve was obtained using a multiple cutback technique[5] with fibre lengths of between 20 cm and 250 m to obtain accurate measurements both within the absorption bands and the low-loss regions. Despite the high doping level the loss is less than 70 dB/km in the window between 1 μm and 1·5 μm and is probably largely due to the presence of transition metal impurities. Losses of 30 dB/km have been seen in other similarly doped fibres. Since only 20 cm of the fibre is required for a fibre laser, the losses at the lasing wavelength (1·09 μm) are negligible. The OH^- absorption peak of only 13 dB/km at 1390 nm confirms the effectiveness of the dehydration process.

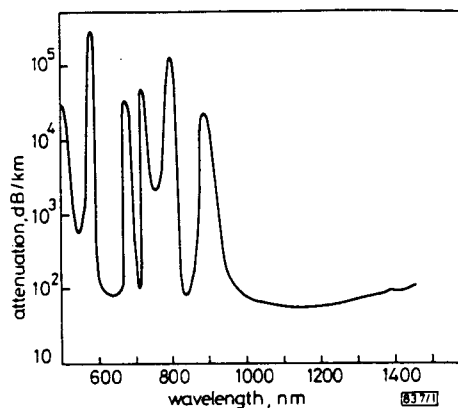*(b) Dehydration:* Three factors affect the core soot dehydration: (i) the drying gases, (ii) the temperature and (iii) the duration of drying. Previous work[6,7] has found that a chlorine-rich atmosphere at 1000°C gives the optimum drying conditions. However, this temperature could not be used here as the more volatile rare-earth dopants would evaporate. Moreover, the optimum chlorine/oxygen ratio is prohibited, since excess oxygen is required to prevent dopant loss by halogenation. These limitations lead to relatively long dehydration times to produce acceptable results. Nevertheless, Fig. 2 shows that there is an exponential dependence of OH^- concentration with time, even for a nonoptimal drying temperature of 600°C and a Cl_2/O_2 ratio of 5 : 2. Thus fibres containing less than 1 part in 10^6 OH^- are readily obtained, provided the drying time exceeds 30 min.

(c) Control of impurity concentration: A wide variation in the dopant concentration may be achieved by altering either the duration of immersion of the preform tube or, more controllably, the solution strength. The dependence of dopant concentration in the fibre on solution strength for a fixed immersion time of 1 h is shown in Fig. 3. From the Figure it is clear that even dilute aqueous solutions of around 0·1 molarity result in much higher rare-earth content (~ 2500 parts in 10^6) than was previously produced.[5] Typically, dopant concentrations of around 0·4% can be obtained after immersion for only 1 h in
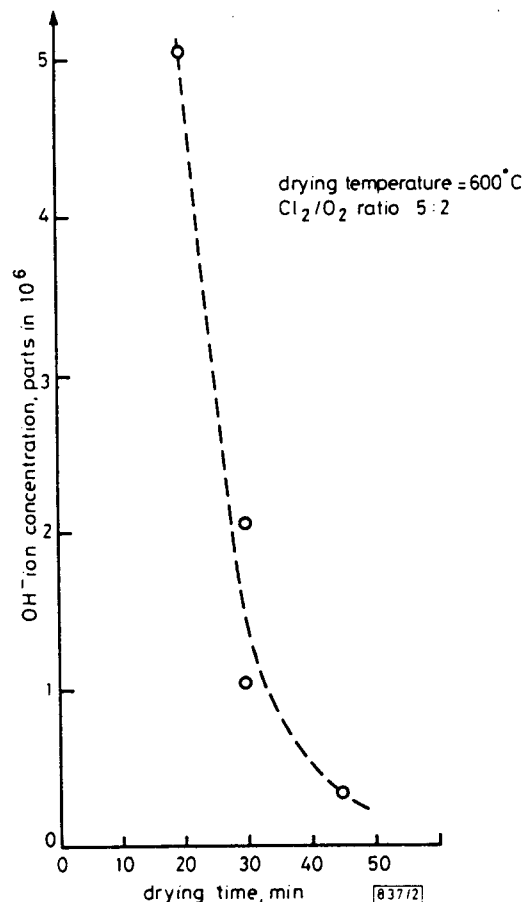

Fig. 2 *OH^- ion concentration dependence on drying time*
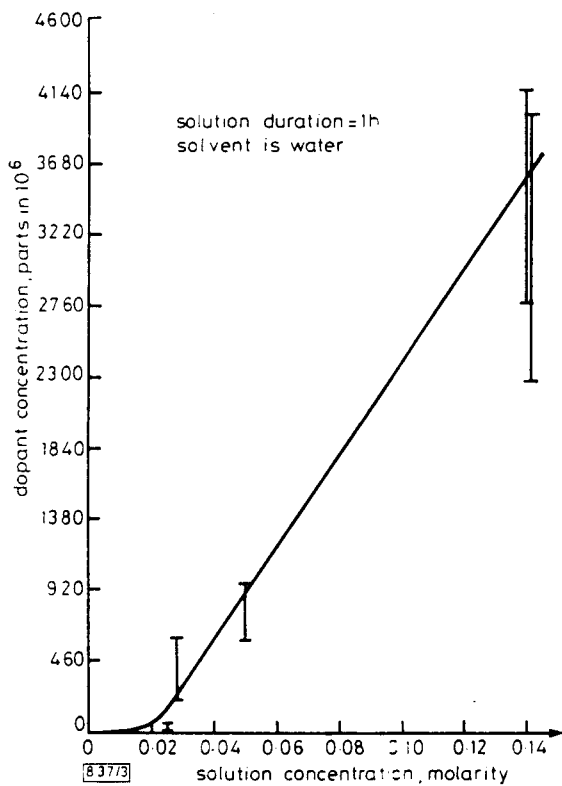
Fig. 3 *Dopant concentration dependence on solution concentration*

Fig. 4 *Absorption spectrum of fibre containing ~1200 parts in 10^6 Er^{3-} and in ~4300 parts in 10^6 Yb^{3-}*

a 0·14 M solution. Higher concentrations (a few per cent) have also been achieved, but usually result in devitrification of the core.

(d) *Codoping:* Codoping is a useful technique to provide pump absorption bands in the fibre lasers. To demonstrate the versatility of solution doping, a preform containing erbium codoped with ytterbium was fabricated. The doping solution contained $ErCl_3$ and $YbCl_3$ in the ratio 1 : 4, and the soaking and drying conditions were similar to those described above. The absorption spectrum of a fibre pulled from this preform is shown in Fig. 4, with the Yb^{3+} absorption band clearly visible at the diode laser wavelength of 850 nm. We calculate the concentrations of Er^{3+} and Yb^{3+} as approximately 1200 and 4300 parts in 10^6, respectively. This calculation is complicated by the proximity of the Yb^{3+} absorption bands at 860 nm and 970 nm to the Er^{3-} absorption band at 980 nm. Nevertheless, the relative concentrations in the fibre are similar to those in the initial solution. Again, despite the very large absorptions present, losses of less than 60 dB/km are obtained.

Conclusions: A simple, versatile and reproducible technique has been developed for uniform addition of rare-earth ions into the core of optical fibres. High dopant concentrations are
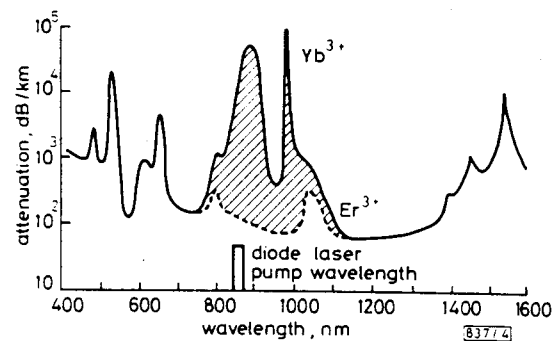
possible without significantly compromising the fibre losses. The method is particularly suited for controllably codoping the fibre and will find application in the development of distributed fibre sensors and active fibres for optical communication systems.

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