

# FABRICATION OF $\text{Al}_2\text{O}_3$ CO-DOPED OPTICAL FIBRES BY A SOLUTION-DOPING TECHNIQUE

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## Abstract

A new technique for fabricating optical fibres containing  $\text{Al}_2\text{O}_3$  is described. Application of the technique to single-mode and rare-earth-doped fibres is discussed.

## Introduction

Many materials have been proposed<sup>1</sup> as alternatives to  $\text{GeO}_2$  in the core of silica-based fibres. Of these,  $\text{Al}_2\text{O}_3$ <sup>2,3</sup> is one of the most promising owing to its relative cheapness and its reduced volatility which leads to a reduced index dip in the fibre core. Widespread adoption of  $\text{Al}_2\text{O}_3$  has, however, been limited by the difficulties in incorporating the dopant due to the low vapour-pressures of the aluminium precursors (normally halides) which require the use of a heated deposition system. The importance of  $\text{Al}_2\text{O}_3$  as a co-dopant has recently re-emerged in rare-earth-doped fibres where its use has been shown to greatly reduce clustering in the rare-earth ions<sup>4</sup> and hence allow the incorporation of far higher levels of rare-earths than previously thought possible<sup>5</sup>. These high dopant levels are likely to prove very important in the development of compact all-fibre devices.

The solution-doping technique for the fabrication of rare-earth-doped fibres<sup>6,7</sup> provides a way of extending the MCVD fabrication technique to permit the introduction of additional, matrix-modifying co-dopants, including  $\text{Al}_2\text{O}_3$ . Although previous work with the solution-doping technique has concentrated on incorporating small quantities of rare-earth elements we show here that it may be easily extended to the incorporation of higher concentrations of other core constituents by simply increasing the solution strength.

In this paper an extension of the solution-doping technique is described by which up to 8.5%  $\text{Al}_2\text{O}_3$  has been incorporated as a dopant in silica-based optical fibres. Results obtained show that low-loss fibres may be fabricated and that the fibres may be co-doped with rare-earth elements in concentrations of up to 2% with no evidence of clustering.

## Experiment

In the solution-doping technique a conventional cladding is first deposited (typically a  $\text{SiO}_2/\text{P}_2\text{O}_5/\text{F}$  glass) following which the core layers are deposited at a reduced temperature to form a partially-sintered, porous soot. An additional dopant is then introduced by removing the tube from the

lathe and soaking it in an aqueous solution of the required material to ensure saturation of the porous frit. The tube is then replaced in the lathe, dried, fused and collapsed. To date this technique has been used only for the introduction of relatively low (<3000ppm) concentrations of rare-earth ions, but we have found that to increase the concentration of a component in the fibre it is only necessary to increase the strength of the solution used.

To incorporate  $\text{Al}_2\text{O}_3$ , a starting aluminium salt is required which is obtainable in a highly pure form and is easily soluble in water.  $\text{AlCl}_3$  is a suitable material, being readily available in 99.9995% pure form and being highly soluble. Preforms were fabricated using solutions of strengths up to 3 molar  $\text{AlCl}_3$  together with various concentrations of rare-earth ions. To prevent phase separation of the alumino-silicate glass,  $\text{P}_2\text{O}_5$  was incorporated in the unfused core layer for the higher aluminium concentrations<sup>3</sup>. It was found that owing to the high solubility of  $\text{AlCl}_3$  the acetone rinse employed previously to remove excess water<sup>6</sup> also removed the  $\text{AlCl}_3$  and thus the preform had to be dried in an  $\text{O}_2/\text{Cl}_2$  flow to remove as much adsorbed water as possible before the normal gas-phase drying procedure.

## Results

A number of preforms have been fabricated containing  $\text{Al}_2\text{O}_3$ -dopant levels up to 8.5 molar %. At the highest dopant concentrations, some phase-separation was observed, particularly at the centre of the deposited core. It is suggested that this is due to volatilisation of  $\text{P}_2\text{O}_5$  or  $\text{SiO}_2$  from the inner deposited layer during the collapse process. Figure 1 shows the refractive index profile of a typical preform. The profile is a good approximation to a step profile and indicates the consistency of incorporation of the  $\text{Al}_2\text{O}_3$  across the core.

The main application of the technique is expected to be in the fabrication of rare-earth-doped fibres, to which the majority of work has been directed. Fibres containing up to 2% of rare-earth ions have been fabricated with no evidence of dopant clustering, suggesting that the rare-earth ions are being incorporated into the host glass matrix rather than interstitially, as in  $\text{GeO}_2$ -doped silica<sup>4</sup>.

A typical loss spectrum of an  $\text{Er}^{3+}$ -doped fibre (125 m O.D, cut-off = 1250nm) containing about 300ppm  $\text{Er}^{3+}$  is shown in Fig 2. Note the extremely low loss of around 7dB/km away from the absorption bands, despite the high dopant level. Superimposed on the same graph for comparison is the loss spectrum of a  $\text{GeO}_2/\text{SiO}_2$  core  $\text{Er}^{3+}$ -doped fibre containing 200ppm  $\text{Er}^{3+}$  which shows the shifts in the relative intensities of the absorption spectra caused by the presence of the alumina. The associated fluorescence spectra are shown in Fig 3 and it should be noted that there is very little shift in the peak fluorescence wavelength, compared to the large shift observed in, for instance, the 4-level  $^4\text{F}_{3/2} - ^4\text{I}_{11/2}$  transition in  $\text{Nd}^{3+}$ . The excited-state absorption of an Al-containing  $\text{Nd}^{3+}$ -doped fibre has also been measured<sup>8</sup> and is shown to be considerably reduced compared to that in a  $\text{SiO}_2/\text{GeO}_2$  fibre.

## Discussion

The ability to incorporate high dopant levels without clustering will lead to many new application for active fibre devices. For instance, in a Q-

switched laser, the short cavity lengths possible using higher dopant levels means that short pulses may be more easily obtained. For instance, it is possible to obtain 15nSec fwhm pulses with peak powers of 110W, when pumping a fibre containing 0.8% Nd<sup>3+</sup>, with a single-stripe semiconductor diode laser<sup>9</sup>. In addition, these highly-doped fibres are expected to be particularly useful in the development of co-doped or sensitized laser systems (eg Yb/Er) where high dopant concentrations (up to 17 mole % ) are required<sup>5</sup>.

During the development of the technique, the level of rare-earth incorporation was considerably higher than would be expected from previous results, for solutions of the same strength<sup>6</sup>. This is possibly due to complexing of the rare-earth-ions and the aluminium ions in solution leading to preferential incorporation of the rare-earth ions. Further work is required to characterize this effect and to exploit any advantages which may incur from being able to control the ligand field environment of the rare-earth ion in this way.

A further possible application of the technique is to introduce some of the alternative dopants such as Nb, Zr and Ta proposed in reference 1. The ligand fields of these ions may lead to improvements in the performance of active fibre devices or alter the non-linear performance of the fibres and lead to new or improved non-linear fibre devices. Improvements to the drying techniques used (and hence the fibre loss) may lead to its application in the fabrication of telecommunications-type fibres, particularly where GeO<sub>2</sub> is unsuitable as a dopant. For fibre lasers, however, generally short lengths are used and the residual water content (which is still only a few ppm) is unlikely to cause problems.

### Conclusions

Fibres co-doped with up to 8.5 mole % of Al<sub>2</sub>O<sub>3</sub> have been fabricated by the solution-doping technique. The method is particularly suitable for producing highly-doped rare-earth-doped fibres as it introduces all the additional dopants in the same step. The possible application of the technique to telecommunications-type fibres has been discussed and it may also provide a simple method for the addition of new dopant materials such as Nb, Zr, Ta.

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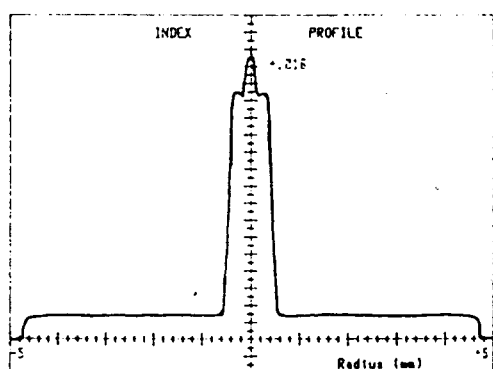


Fig 1 Refractive-index-profile of  $\text{Al}_2\text{O}_3$ -doped fibre

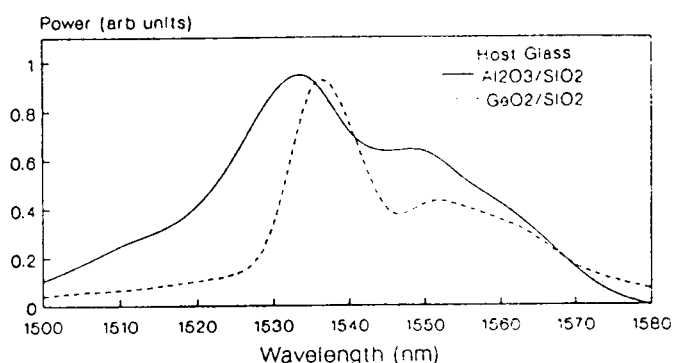


Fig 3 Fluorescence spectra of  $\text{Al}_2\text{O}_3$ - and  $\text{GeO}_2$ -doped fibres co-doped with  $\text{Er}^{3+}$ .

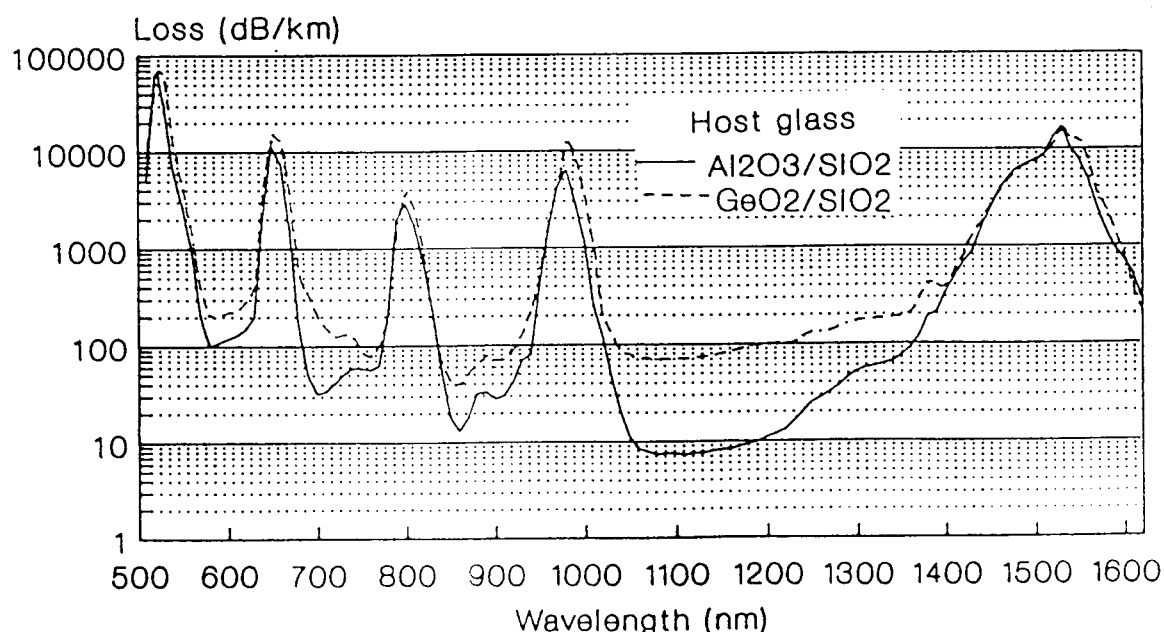


Fig 2 Loss spectra of  $\text{Al}_2\text{O}_3$  doped fibre co-doped with  $\text{Er}^{3+}$ . A similar  $\text{GeO}_2$ -doped fibre is shown for comparison.