Determination of Erbium Distribution in Optical Fibers Using Confocal Optical Microscopy

D. Uttamchandani, A. Othonos, A. T. Alavie, Member, *ZEEE,* and M. Hubert, Member, *ZEEE*

***Abstract*— Confocal fluorescence microscopy has been used to detect the 565 nm wavelength fluorescence generated from Er^{+3} ions present in erbium doped optical fibers, thereby enabling the concentration profile of these ions in the fiber core to be determined. The fluorescence intensity distribution also enables the total Er^{+3} ion concentration in the fibers to be estimated, and the estimated values are shown to be in good agreement with data supplied by the fiber manufacturers.**

I. Introduction

OPTICAL fibers doped with rare-earth elements, particularly erbium, are now being used extensively in fiber lasers and fiber amplifiers [1]. The design and manufacture of such fibers, along with modelling and performance of optical devices based on these fibers, will be greatly assisted if practical measurements are available on the concentration profile of the rare earth ions present in the fiber core and surrounding cladding. Most techniques used to date, such as secondary ion mass spectroscopy combined with electron probe microanalysis and X-ray fluorescence, involve measurements being made on the fiber preform [2]. There is, however, no guarantee that such measurements will remain accurate when the fiber is subsequently drawn from the preform. It is expected that some redistribution in the concentration of the rare-earth elements will occur during the fiber drawing process. This re-distribution is normally disregarded because of the difficulty in implementing techniques capable of directly measuring the rare earth concentration profile in the fiber core, due to its small (typically less than 5 μm) diameter. Techniques which have recently been investigated for the measurement of erbium in fibers include near-field microscopy [3], and a differential mode launching technique [4].

In this Letter we report an alternative method which allows direct determination of erbium profile and concentration in the fiber core. Use has been made, for the first time, of a confocal optical microscope [5] (model Biorad MRC 600) operating in the fluorescence detection mode to undertake high resolution fluorescence microscopy of the end face of an optical fiber. The objective of the experiment was to detect and map the fluorescence produced by erbium ions in the core of a number of fibers. In our preliminary work, we investigated the possibility of utilizing fluorescence microscopy to determine the erbium concentration in the doped fiber using an argon ion laser. In this experimental work we determined that the Er^{+3} ions can be excited with 488 nm laser light in the doped fiber resulting in fluorescence with a peak wavelength at 565 nm. The choice of a confocal microscope for the detection of the fluorescence was based on its 0.33 μm spatial resolution (for a 40 times objective lens with 0.55 numerical aperture) and planar discrimination (i.e. the depth of sensitivity is estimated to be less than 1 μm). The high spatial resolution along with the short depth of field for detection and the high sensitivity of the photon counting makes the confocal microscope an ideal system for studying the fluorescence from the erbium doped fibers.

An air cooled argon ion laser was used as the optical source in the confocal microscope. The 488 nm laser beam from this source was focused onto the cleaved end face of erbium doped fibers using a 40 times objective lens with a numerical aperture of 0.55. Fluorescence generated from the cleaved end face was collected and separated from the laser light using a high pass optical filter whose low wavelength cut-off was at 515 nm. A THORN EM1 type 9828B head-on photomultiplier operated in the single photon counting mode was used for detection of the fluorescence at 565 nm. The combination of high pass filter and spectral response of the photomultiplier provided a sensitivity window between 515–600 nm allowing the detection of the Er^{+3} fluorescence. Observation of the video image produced on the display monitor of the confocal microscope system shows a spatial variation in fluorescence intensity emitted from short sections of fiber (approximately 1 mm in length). The upper right hand corner of Fig. 1 shows a video monitor display of a fluorescence image obtained from an experimental AT&T erbium doped fiber with a core diameter of 3.1μm and an erbium ion concentration of 450 ppm [6].

Fig. 1 also shows the fluorescence intensity variation detected when the laser beam of the confocal microscope was scanned along the centre line of the cleaved face of the same experimental AT&T fiber. This result corresponds to a line scan extending over 10 μm, beginning from the cladding region near one side of the core, then progressing across the core diameter and terminating in the cladding on the opposite side of the starting point. For comparison, a Gaussian function $f(r) = \exp(-r^2/\sigma^2)$ is also plotted in Figure 1, where r is the distance from the centre of the fiber core, and σ the Gaussian half-width where f(r) equals l/e of its maximum value. It can be seen that the fluorescence profile from the experimental

Manuscript received September 22, 1993; revised December 23, 1993.

D. Uttamchandani is with the Department of Electronics & Electrical Engineering, University of Strathclyde, 204 George Street, Glasgow G11XW, Scotland (U.K.).

A. Othonos and M. Hubert are with The Ontario Laser & Lightwave Research Centre, University of Toronto, McLennan Physical Laboratories, 60 St. George Street, Suite 331, Toronto M5S1A7, Canada.

A. T. Alavie is with the The Institute for Aerospace Studies, University of Toronto, Downsview, Ontario M3H-5T6 Canada.

IEEE Log Number 92 1658 1.

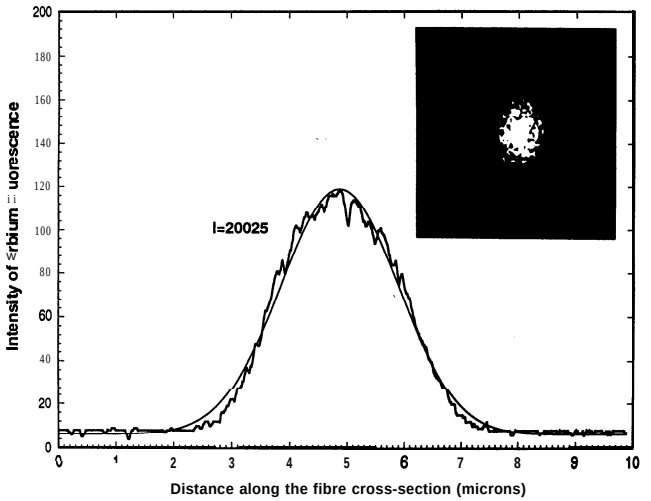

Fig. 1. Fluorescence distribution at the centre of the experimental AT&T fiber. For comparison, a Gaussian curve with $\sigma = 1.44\ \mu$m is fitted on the trace. A two dimensional video image showing the fluorescence obtained from the confocal microscope is shown in the upper right hand comer.

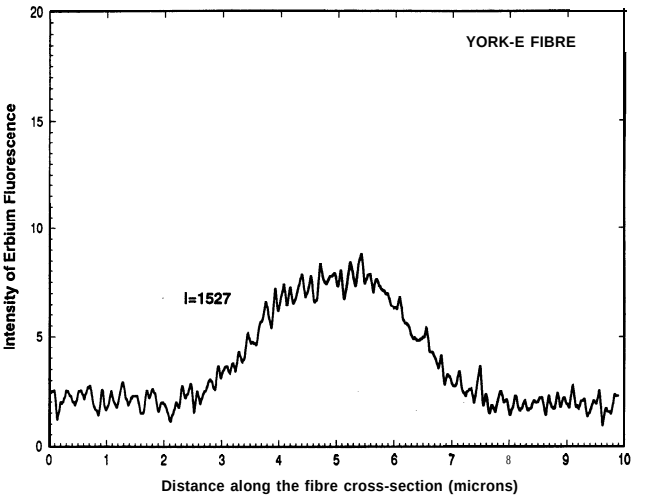

Fig. 2. Fluorescence distribution at the centre of the York-A fiber.

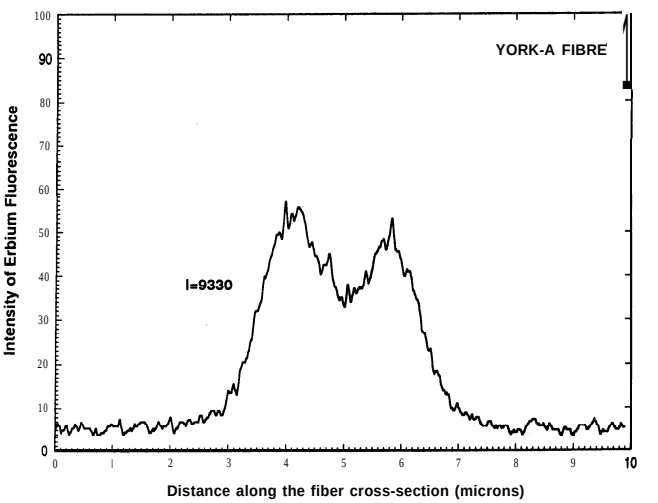

Fig. 3. Fluorescence distribution a tthe centre of the York-E fiber.

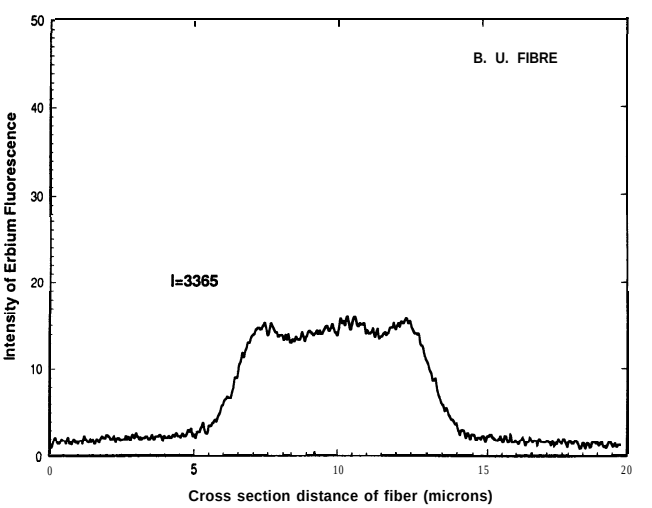

Fig. 4. Fluorescence distribution at the centre of the Brown University fiber.

AT&T fiber closely matches the Gaussian distribution when $\sigma = 1.44\ \mu$m. In addition to the intensity distribution, the total fluorescence intensity was evaluated by integrating the area under the fluorescence distribution curve. For the experimental AT&T fiber used the integral under the fluorescence curve, denoted as "I," had a value of 20,025 arbitrary units (a.u.). The confocal microscopy technique was then used to analyze an optical fiber not containing erbium ions. No fluorescence was observed from the undoped fiber, confirming that the fluorescence observed from the experimental AT&T fiber is indeed a feature of the presence of erbium ions.

Three other erbium doped fibers were tested in the same way, using the same experimental conditions used to produce the results of Fig. 1. Figs. 2 and 3 show, respectively, the fluorescence intensity distributions obtained from type DF1500A and DF1500E amplifier fibers, both supplied by York Fibers Ltd. These fibers have an erbium ion concentration of 200-240 ppm and 40–45 ppm respectively [7]. The corresponding values of the integral of "I" for these fibers are 9330 and 1527 a.u. The DF1500A fiber exhibits a ring shaped fluorescence intensity distribution with a pronounced dip in intensity near the centre of the fiber core. The fluorescence intensity distribution is not constant around the circumference of the ring, implying an uneven erbium ion distribution in this fiber sample due to "burn off'. This experiment was repeated over several sections of the same fiber and similar observations were obtained. The DF1500E fiber exhibited a weaker fluorescent signal compared to the first two fibers investigated, but with no obvious dip in the fluorescence intensity.

Fig. 4 shows the fluorescence distribution obtained from a fiber produced at Brown University [8] with an erbium concentration of 70-80 ppm. The fiber exhibited a W shaped fluorescence profile which is probably similar to the refractive index variation across its centre. The value of "I" calculated for this fiber was 3365. In order to convert this value into an equivalent erbium ion concentration we have used the numerical constant which converts between the value of "I" and the actual erbium concentration of the experimental AT&T fiber, which was taken as the reference. The same constant

TABLE I
ESTIMATE OF Er^{3+} CONCENTRATION IN DIFFERENT FIBERS USING VALUE OF INTEGRATED INTENSITY.

Fiber Type	Integrated intensity under fluorescence curve (a.u)	Actual Er^{3+} concentration (ppm)	Estimated Er^{3+} concentration (ppm)
AT&T	20,025	450	given
YORK-A	9330	200-240	210
YORK-E	1527	40-45	34
BROWN UNIVERSITY	3365	70-80	76

has been used to estimate the erbium concentration in the two York fibers, for comparison purposes. Table I shows the results.

The estimated erbium concentration for the YORK-E fiber is seen to be around 14% smaller than the average value of the actual erbium concentration in the fiber. This discrepancy is probably due to the weak fluorescence signal produced from this fiber giving rise to a noisy intensity profile trace and therefore greater measurement inaccuracy (this is by no means a limitation of the technique rather it is a limitation of our photomultiplier detection system). The results obtained from the other three fibers show much better agreement between the experimental results and the manufacturers data.

In these experiments we considered one of the doped fibers as the standard for calibration, however it is possible to use a section of preform of known erbium concentration to calibrate the detection system.

Finally, due to the guiding nature of the optical fiber under test which could potentially give rise to erroneous readings with respect to the concentration profile, we undertook two simple experiments to resolve these concerns. First, surface illumination of the fiber with the 488 nm line of the Argon ion laser gave no indication of an interference pattern which is usually associated with operating a single mode fiber above its cut off. Second, a 1 mm length of a test fiber was evaluated using the present technique and then cut in half and reevaluated. Comparison of the results showed no significant change (less than 5% change) between the fluorescence from the two samples indicating no or at least a very small dependence on the guiding properties of the fiber. While this technique presents perhaps a simple alternative for the measurement of erbium concentration and profile in the fiber, further modifications and investigations will be necessary to fully realize the potential use of this approach.

In summary, for the first time a confocal fluorescence microscope has been used to estimate the erbium ion distribution and concentration in four different types of erbium doped fiber. The estimated results are in good agreement with data provided by the fiber manufacturers.

ACKNOWLEDGMENT

The authors wish to thank D. J. DiGiovanni and AT&T, York Fiber Ltd. and T. Morse of Brown University for providing the erbium doped fiber used in these experiments. This work has been supported by the Ontario Laser and Lightwave Research Center.

REFERENCES

[1] Special issue on Optical Amplifiers, *IEEE J. Lightwave Technol.*, vol. 9, pp. 145-296, 1991.
[2] D. W. Oblas, F. Pink, M. P. Singh, J. Connolly, D. Dugger and T. Wei, *Digest of Materials Research Society Annual Meeting 1992,* (MRS Pittsburgh, Pa), pp. 56.3.
[3] J. K. Trautman, E. Betzig, J. S. Weiner, D. J. DiGiovanni, T. D. Harris, F. Hellman and E. M. Gyorgy, "Image contrast in near field optics," *J. Appl. Phys.,* vol. 71, pp. 4659-4663, 1992.
[4] A. M. Vengsarkar, D. J. DiGiovanni, W. A. Reed, K. W. Quoi and K. L. Walker, *Optics Letters,* vol. *17,* page 1277-1279, 1992.
[5] T. Wilson (ed), *Confocal Microscopy,* Academic Press, New York, N.Y., 1990.
[6] Data and fiber provided by AT&T.
[7] Data and fiber provided by York Fibers Ltd.
[8] Data and fiber provided by Brown University.