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Microscopy characterization of doped fibers

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Abstract

The use of the Soft X-ray Contact Microscopy technique is discussed as a possible new tool to get information on dopant distribution in the core of single-mode optical fibers with 50 nm spatial resolution.

Keywords: Dopant constration; Optical fibers; X-ray microscopy

1. INTRODUCTION

In all the applications of doped fibers, the concentration and distribution of erbium ions play a crucial role in determining the performances and characteristics of the devices. Actually, these ions act as the laser active centers and their distribution inside the matrix is fundamental in dictating the properties of the overall system, a broad tunable laser source emitting around 1550 nm. The control of the dopant distribution is fundamental (Othonos *et al.*, 1995; Garcia *et al.*, 2003) since the active fiber works like an amplifier, in absence of the smoothing effect on the electromagnetic field due to the optical feedback from cavity mirrors, as it happens in lasers. In this paper, we propose an experimental method for the measurement of dopant distribution in the core of single-mode optical fibers (with 50 nm spatial resolution), based on the Soft X-ray Contact Microscopy (SXCM) technique (Batani *et al.*, 2000, 2002; Desai *et al.*, 2003). The technique should provide quantitative results on fiber doping. Its drawback is that it will be relatively slow because of the requirement of slicing of the fiber and the use of photoresist.

The achievable resolution of this technique ($\sim$ 50 nm) is not as high as electron microscopy, but better than optical microscopy (many factors contribute to limit the resolution, that is: (i) Fresnel diffraction; (ii) "penumbral blurring"; and (iii) shot noise). However, the most important feature of the technique are that:

1. actual fibers, and not only preforms, can be investigated. This is important since the process of drawing the fiber from the preform can indeed cause some redistribution in the concentration of dopants, and
2. by suitably choosing X-ray photon energy, chemical element discrimination is possible.

Moreover, a single X-ray shot allows the simultaneous analysis of many fiber slices, irradiated under the same conditions.

2. THE EXPERIMENT

We propose a new technique for measurement of dopant distribution in the core of single-mode optical fibers, which is based on SXCM. The overall picture of the proposed technique is hereafter reported, together with its basic elements: the source, the target and its relevant preparation problems, the fundamentals of the interaction between the probe and the sample, and the diagnostics.

2.1. The source

A laser-plasma source produces X-rays in a window that is selected by choosing a suitable target and filters. In particular, here we will refer to two different sources, which are available within the frame of the E.U. Large Facility Program: the one at Rutherford Appleton Laboratory (RAL), at Abingdon, United Kingdom and Prague Asterix Laser System (PALS) in Prague (Czech Republic).

The laser-plasma X-ray source at RAL (Turcu *et al.*, 1994) delivers an X-ray average power of $\approx$ 1 watt at

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1 nm wavelength ($h\nu \approx 1$ keV) into 2π steradians, from a point source ($\approx$ 10 μm diameter). The source is operated in ambient Helium gas at atmospheric pressure, which results in contamination free delivery of X-rays to the exposure cell. The plasma source is generated by a picosecond excimer laser system operated at 248 nm wavelength and delivering a diffraction limited beam of trains of 16 pulses, each 5 ps long and separated by 2 ns. The lasing energy is 350 mJ/pulse-train at a repetition rate up to 100 Hz. The RAL laser-plasma X-ray source is the first source fully scheduled for application experiments. It complements Synchrotron Radiation Sources (SRS) in: (a) source geometry: 10 micron point source; (b) temporal characteristic: pulsed ps source at 1–100 Hz; and (c) wavelength range 0.5–50 nm.

The single beam PALS high power iodine laser (Jungwirth *et al.*, 2001) is a photolytically pumped gas laser with an emission wavelength at 1.315 micron. The PALS laser actually derives from the Asterix IV laser system which was previously located in Garching. The PALS system is set in the classical Master Oscillator Parametric Amplifier (MOPA) configuration. The pulse to be amplified is delivered by an acousto-optically mode locked oscillator, generating sub-ns pulses. The amplifier chain consists of 6 amplifiers of increasing diameters and length. The final amplifier has a clear aperture of 29 cm. The PALS laser delivers a maximum output power of 4 TW at a pulse length of 0.3 ns, corresponding to an energy of 1.2 kJ at ω and about 450 J at 3ω.

A preliminary step is the evaluation of the performances of the two X-ray sources showing that the microscopy experiment can be performed in both cases.

In the case of PALS, many sample holders can be positioned into the irradiation chamber, allowing a significant statistical analysis of the results. Actually, using a large source with a large X-ray flux, there is the possibility to put the microscopes (i.e., the holders for samples and photoresist) very far from the source, typically 10 cm, as already done in SXCM experiments of living biological cells (Batani *et al.*, 2002). Even with a large focal spot (400 microns, even if in reality the PALS spot size can be reduced down to 50 microns) this reduces the penumbral blurring much below 50 nm. Since this is the limit of the technique, imposed by diffraction effects and shot noise, it means that penumbral blurring is practically negligible on PALS.

In the case of the RAL source, the experiment will rely on a small laser source; a low X-ray flux is involved so that the sample holders can be positioned closer to the source. However, optical fibers (unlike living biological cells) do not suffer a composition change when exposed to X-rays for long exposure times, it will be possible to irradiate the samples for times up to several minutes. If one integrates the X-ray dose over a longer period of time, say 100 seconds, the 100 Hz repetition rate RAL source would possibly deliver the same X-ray dose as the single shot 1.2 kJ pulsed laser. This implies that this kind of experiment can possibly be done at other facilities too.

2.2. Target preparation

A "radiography" of the sample will be recorded onto a X-ray photoresist and analyzed, after development, by means of an Atomic Force Microscope. The geometrical properties of the beam and the physics of the interaction of X-rays with the sample coupled to the photoresist pose strict conditions on the geometry of the samples themselves. The spectral window is controlled in order to enhance the contrast between the matrix and the dopant in the fiber.

Inter-linked parameters, such as the variation of dopant distribution and thickness, are critical to assess X-ray absorbed doses. The interesting region in the fiber, where relevant changes of the dopant distribution are present and should be controlled, is the one near the core, having a diameter of about 10 microns (see Fig. 1). The resolution ability of the proposed technique is around 50 nm, the distribution of the relevant ions can then be mapped and the ion concentration estimated.

To estimate the different absorption properties of relevant materials in optical fiber systems, Figures 2a and b show, for the sake of example, the transmission of a layer of the same thickness of Er and SiO_2 in the region around 200 and 1400 eV. These are the interesting regions in which to perform the experiment being close to absorption edges in Erbium, and hence allowing for a large difference between absorption coefficients of Er and SiO_2. Also the higher energy region ($h\nu > 10$ keV) is interesting: here the large mass difference produces strong differences in X-ray absorption.

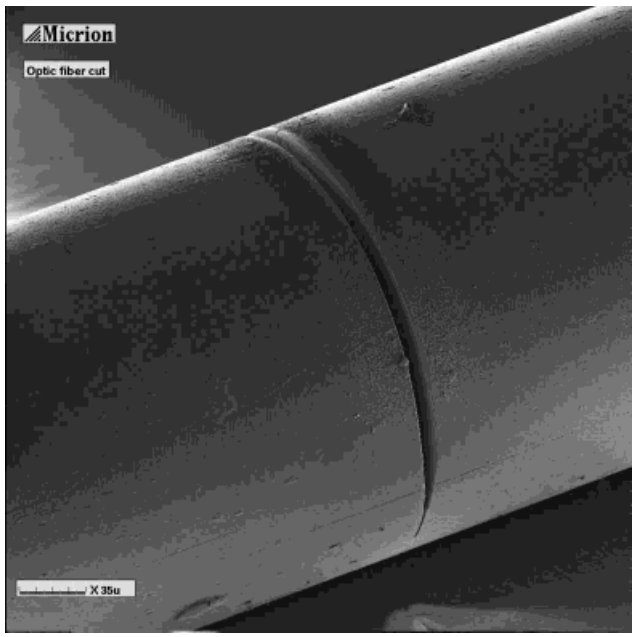

Fig. 1. Image of an Er doped fiber obtained with the Focused Ion Beam technique (Milani *et al.*, 2004) having a core diameter of about 10 microns and working at wavelengths around 1550 nm. The fiber has been both imaged and cut (in the middle) using FIB.

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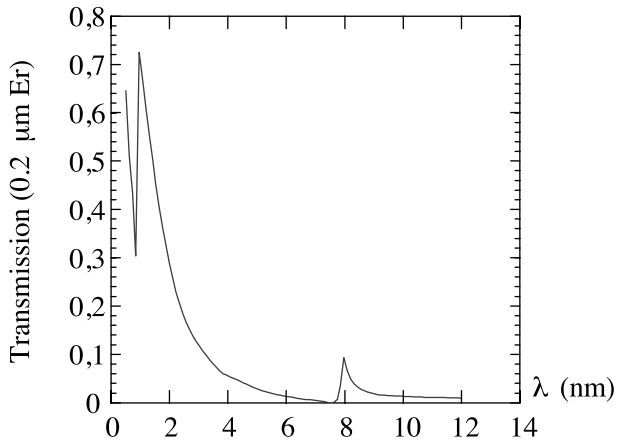

Fig. 2a. Transmission of a 0.2 μm thick layer of Er in the region between 100 eV and 3 keV. Notice the X-ray absorption edges of Er around 140 and 1450 eV.

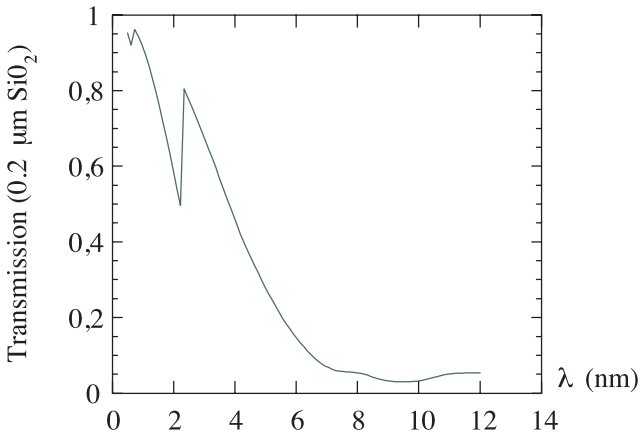

Fig. 2b. Transmission of a 0.2 μm thick layer of SiO_2 in the region between 100 eV and 3 keV.

Multiple targets can be simultaneously irradiated provided that a reasonable energy is supplied of the order of Joules in hundredths of ps on surfaces of 100 square microns. Critical parameters for the technique are the sample thickness that rules the energy density to the photoresist and the X-ray energy selected range to enhance, that is the contrast between the different fiber elemental components.

2.3. The atomic force microscope

Using soft X-rays with energy around the Er absorption edge, a natural contrast emerges in samples between core and cladding and inside the core among regions with different dopant concentration. The sample, or the samples (since the technique allows the simultaneous investigation of many

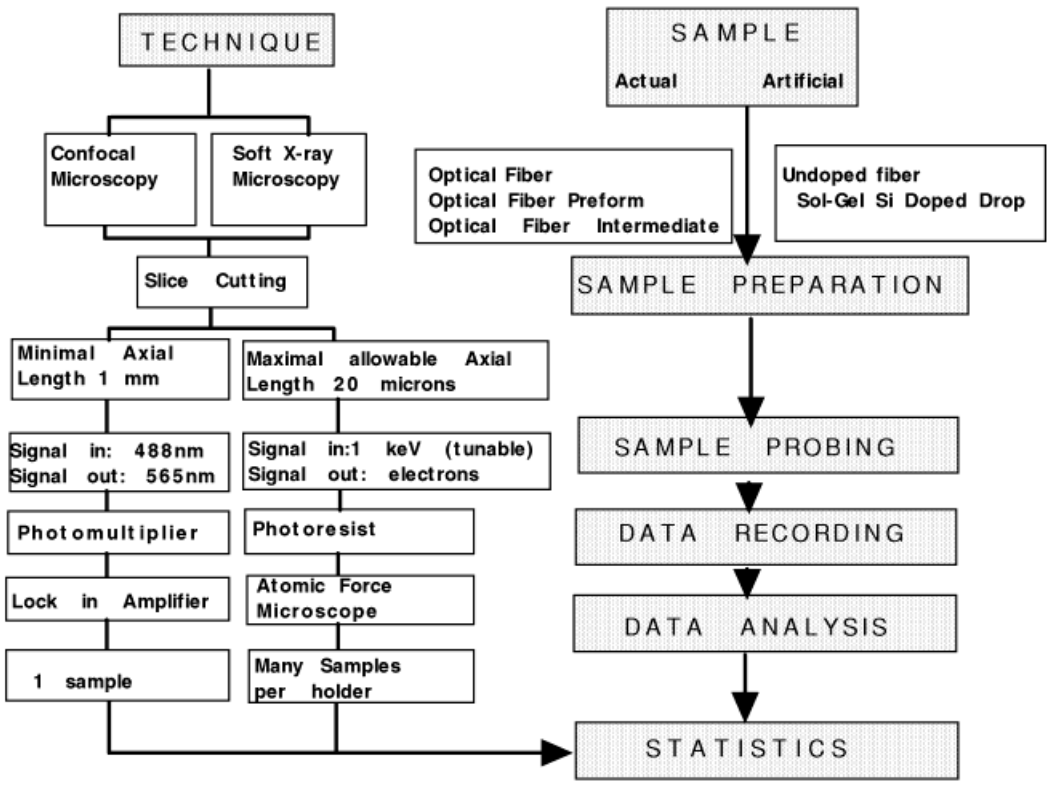

Fig. 3. A flow chart for the comparison of SXCM and Confocal Microscopy techniques

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samples) is put in a holder directly above the X-ray photoresist. The specimen will have the shape of a cylinder with height as small as possible. A microradiography of the samples will be recorded on the photoresist which can be later analyzed using a scanning Electron Microscope, or an Atomic Force Microscope (AFM).

In particular, with AFM it is possible to envisage a "step-by-step" development technique, in order to achieve the best possible result. This is important because the correct development time for the resist can be vary because of fluctuations in the X-ray flux and in the sample height and characteristics.

3. POSSIBLE RESULTS

Apart from cutting in thin slices, the proposed technique allows the imaging of the sample without any need of preparation stage, which can change fiber material parameters. It can be compared with standard techniques that do not guarantee that the ion distribution remains accurate when the fiber is drawn from the preform and with recently reported phase-sensitive confocal microscopy where the spatial profile of fluorescence is detected (Othonos *et al.*, 1995). Figure 3 reports a comparison between the proposed technique and an optical one. The first necessary step in order to make the proposed approach feasible is to prepare samples in the desired shapes and sizes. This can be achieved by preparing 20 micron thick slices from the actual fibers.

4. DISCUSSION AND CONCLUSIONS

One serious problem of the proposed SXCM technique is the possibility of avoiding X-ray emission outside the desired spectral window, which can be probably obtained by the insertion of suitable filters. A further critical element is represented by the structure of the sample holder and the location of fiber slices. The experience acquired in handling biological specimens, for SXCM experiments in particular (Batani *et al.*, 2002; Masini *et al.*, 1999; Milani *et al.*, 1999), turns out to be very useful in solving this problem. The proposed technique appears to be free from some bias such as the dependence on the supplier for calibration, fluorescence signal saturation, and lack of ability in discriminating between dopant ions that are optically active or not.

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