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Gradual-Time Solution Doping for the Fabrication of Longitudinally Varying Optical Fibres

Jean-François Lupi^{ID}, Manuel Vermillac, Stanislaw Trzesien, Michèle Ude, Wilfried Blanc^{ID},
and Bernard Dussardier^{ID}

Abstract—Standard MCVD process and solution doping are commonly used for processing rare-earth doped optical fibers. Since the 1970s, this technique is mainly employed to fabricate homogeneous preforms in the longitudinal direction. A variant is proposed here, by using two doping solutions and by applying a gradual time doping. To demonstrate the validity of this approach, three preforms were prepared with longitudinal gradient of La, Ce, and Al. Preforms were characterized by refractive index profile measurements and concentrations in optical fibers were characterized by EPMA. These results will allow to perform rapid material development in optical-fiber domain. This process is also expected to offer a new way to prepare optical fibers with varying longitudinal characteristics.

Index Terms—Materials processing, optical fibers.

I. INTRODUCTION

NOWADAYS, optical fiber technology is the cornerstone of many applications such as telecommunications, material processing or sensors [1], [2]. To fulfill the characteristics required for these applications, many processes have been developed to prepare silica-based optical fibers: MCVD (Modified Chemical Vapor Deposition), OVD (Outside Vapor Deposition), VAD (Vapor Axial Deposition), DND (Direct Nanoparticle Deposition), Rod-in-tube and powder in tube [3]–[10]. Thanks to its versatility, MCVD is one of the most common process used to prepare specialty optical fibers. In this case, the composition of the core can be chosen by applying one of the following techniques: impregnation of the porous core layer through the solution doping [9], [11]–[14], chelates delivery [15], [16], nanoparticles [17], [18], flash vaporization [19], REPUSIL (Reactive Powder Sintering) [20], [21] or sol-gel [22].

All these processes aim at preparing homogeneous preforms in the longitudinal direction in order to prepare many kilometers of fiber with constant opto-geometrical characteristics. While this approach is of interest from an industrial point of view, there

is also an interest to prepare optical fibers with a longitudinal variation of its characteristics such as the core size, the numerical aperture or the concentration of rare-earth ions. For instance, to suppress the Brillouin scattering, it has been proposed to change the numerical aperture all along the fiber [23]–[26]. A meter-scale gradual doping technique would also be a versatile alternative to the so-called thermally expanded core technique which is developed for materials containing elements with specific diffusion behavior [27], [28]. Such fiber-based devices are less sensitive to alignment [29], [30] and allow applications such as frequency comb filtering [31]. An approach to develop fiber with longitudinally varying characteristics is also of interest in the context of the development of new fiber lasers or amplifiers. Indeed, their performances (output power, mitigation of photodarkening, chromatic or intermodal dispersion...) depend strongly on the composition and opto-geometrical features. In order to systematically screen the effects of these parameters, there is an interest to prepare a single preform with longitudinally varying composition, instead of series of preforms, leading to several pieces of fiber with approximately constant characteristics over a typical 10-m length necessary of spectroscopic studies and/or fiber laser or amplifier tests.

In this context, we propose in this paper a process allowing to prepare a longitudinally varying fiber based on the MCVD process and a modified solution doping technique.

II. GRADUAL TIME DOPING TECHNIQUE

The standard process used to prepare silica-based optical fibers is based on the preparation of a preform fabricated by the conventional MCVD process and the so-called solution doping technique applied to incorporate elements such as aluminum (Al) or rare-earth ions. For the standard solution doping technique, a solution containing dissolved chlorides salts soaked a porous silica layer deposited inside substrate silica tube by MCVD. After soaking of the layer, the doping solution is drained off and the core layer is dried and sintered down to a dense glass layer. Then the tube is collapsed into a solid rod, referred to as a preform, at an elevated temperature higher than 1800 °C. The preform is stretched into a 125 μm -fiber using a drawing tower at temperature higher than 2000 °C under usual conditions.

Through the solution doping technique, the composition of the core depends on many parameters such as the porosity of the silica layer, the concentration of chlorides salts, the solvent (water, ethanol, etc), and the soaking time. Typical time to reach the stationary concentration is reported to be about one hour [9],

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The authors are with the Université Côte d'Azur, CNRS, Institut de Physique de Nice, UMR7010, Nice 06108, France (e-mail: jean-francois.lupi@unice.fr; manuel.vermillac@unice.fr; stanislaw.trzesien@unice.fr; michele.ude@unice.fr; wilfried.blanc@unice.fr; bernard.dussardier@unice.fr).

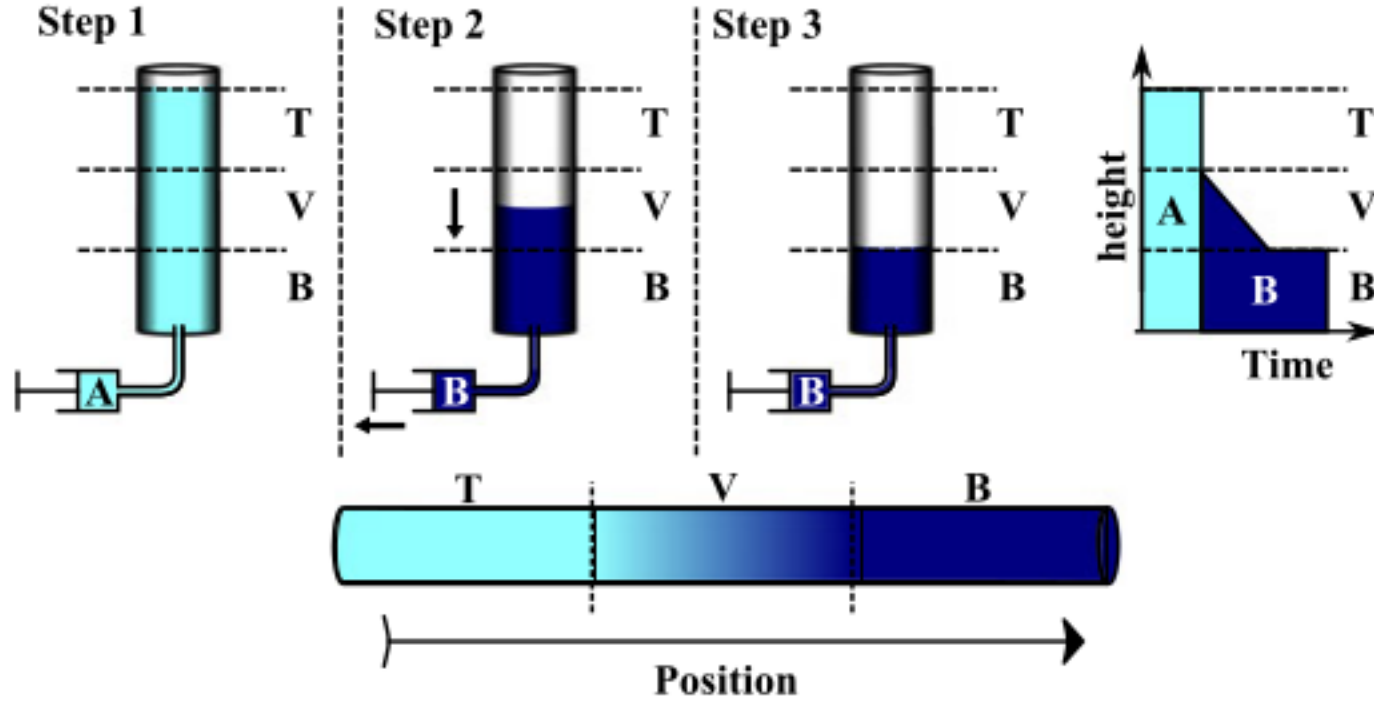


Fig. 1. Schematic representation of the time gradual doping technique.

[11]–[14]. In our process, we propose to vary the soaking time to modify the composition of the core.

To demonstrate the ability of this approach, we have followed the procedure sketched in Fig. 1 where the tube is divided in three parts labeled T (Top), V (Variant), B (Bottom) corresponding to the upper, intermediate and lower part of the tube in vertical position, respectively. Two doping solutions (named A and B) are used. Both solutions contain the same elements, with the same concentration for the invariant elements (constant concentration all along the preform) and with a higher concentration in solution B for the variant elements (longitudinally-varying element concentration). The process can be described as follow:

Step 1: the entire preform (up to the top of the T part) is soaked with the solution A for more than one hour to reach the stationary concentration.

Step 2: solution A is removed and the solution B is injected up to the top of the V part of the preform. The rising time is around one minute. Before introducing the solution B, there is no oxidation step nor rinsing even nor drying of the porous layer. The solution B is slowly drained until the bottom of the V part (the top of the B part) of the preform. The draining time is about 20–30 minutes. During this second soaking, the doping solution and the porous layer concentrations are at the equilibrium for the invariant elements in the V part. However, this is not the case for the variant elements. Then, in this part of the preform (V part), due to the varying soaking time, it is expected that the composition changes as the concentration of variant elements increases.

Step 3: the lower part of the preform (B part) is soaked for more than one hour with the solution B in order to reach the complete equilibrium in this part. Finally, the solution B is removed and the MCVD process is completed.

III. PREFORMS AND FIBERS PREPARATION

To test the validity of this protocol, several samples were prepared. All the preforms were prepared with the same recipe. A single pure silica porous layer was deposited inside a silica tube (outer and inner diameters were 20 and 17 mm, respectively) with a SiCl_4 flow rate of 50 cc/min (bubbler temperature: 35 °C) at a temperature deposition of 1650 °C and a burner's speed of 20 cm/min.

TABLE I
COMPOSITIONS OF THE DOPING SOLUTIONS

Name	Solution A (mmol/l)	Solution B (mmol/l)	L_v/T_v
Grad-La	Al:700 Tm:20 La:30	Al:700 Tm:20 La:150	10cm/30min
Grad-Al	Al:700 Tm:20	Al:2000 Tm:20	10cm/20min
Grad-Ce	Al:700 Tm:20 Ce:0	Al:700 Tm:20 Ce:80	10cm/20min
Cst-Al	Al:700	Al:700	N.A.
Cst-Al2	Al:93	Al:187	N.A.

L_v and T_v correspond to the length and the draining time of the V part, respectively. The variant elements are bolded.

Four compositions of doping solution were used and are reported in the Table I. Three solutions, containing aluminum (Al), thulium (Tm), lanthanum (La) and cerium (Ce) chlorides were prepared using water as solvent. The preforms *Grad-Al*, *Grad-La* and *Grad-Ce* were prepared with aluminum, lanthanum and cerium as variant elements, respectively. The length and the draining time of the V part are reported also in the Table I. Taking the same “0 cm” reference position for all the preforms, the T, V and B parts correspond to the 0–13, 13–23 and 23–40 cm positions, respectively. Two preforms (*Cst-Al* and *Cst-Al2*) were prepared without V part and the limit between the B and T parts corresponds to the middle of the tube. The preform *Cst-Al* was prepared to test the effect of the double solution-soaking on the final concentration. A single doping solution was used, containing 0.7 mol/l of aluminum chlorides dissolved in ethanol. For the preform *Cst-Al2*, the water-based solutions A and B contained 0.093 and 0.187 mol/l of aluminum chlorides, respectively.

All the preforms were drawn into 125 μm fibers with the same conditions. As the preform diameter is 9.5 mm, the correspondence between the longitudinal position of the preform and the fiber is determined assuming a conversion factor of 5800 (1 cm of preform corresponds to 58 m of fiber).

IV. RESULTS

A. Homogeneity of the Porous Layer

As already mentioned, except the concentrations of the doping solution and the doping time, the porosity can influence also the final composition. A silica porous layer was deposited with the same conditions as previously indicated and analyzed all along the preform through the acquisition of Scanning Electron Microscope (SEM, JSM 7000F JEOL, Ltd, Tokyo, Japan) images (Fig. 2). For all these pictures, the magnification and the accelerating voltage were $\times 10.000$ and 5 keV, respectively. The porosity appears almost constant all along the V zone (between 13 and 23 cm), with large pore size (typically in the μm scale). Therefore, we assume that the variations of concentrations observed in the following are not attributed to a change of the porosity.

B. Effect of the Double Solution-Soaking

In the gradual time doping technique, the B part of the preform is soaked twice. The *Cst-Al* preform was prepared to determine the effect of the double solution-soaking on the final concentration of the invariant element in the B part. For this preform,

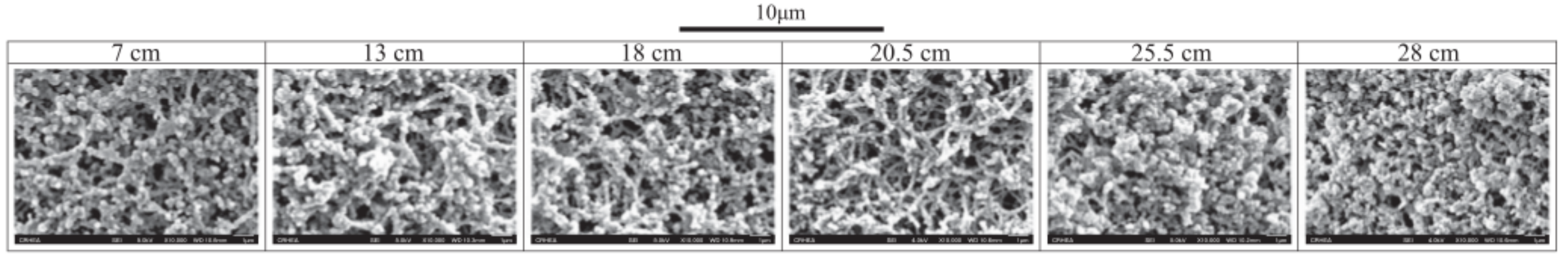


Fig. 2. Images of the as-deposited porous layer snapped at different positions all along the tube (from 7 cm to 28 cm) before the solution doping step. All images have the same scale. The variant (V) part is between 13 and 23 cm.

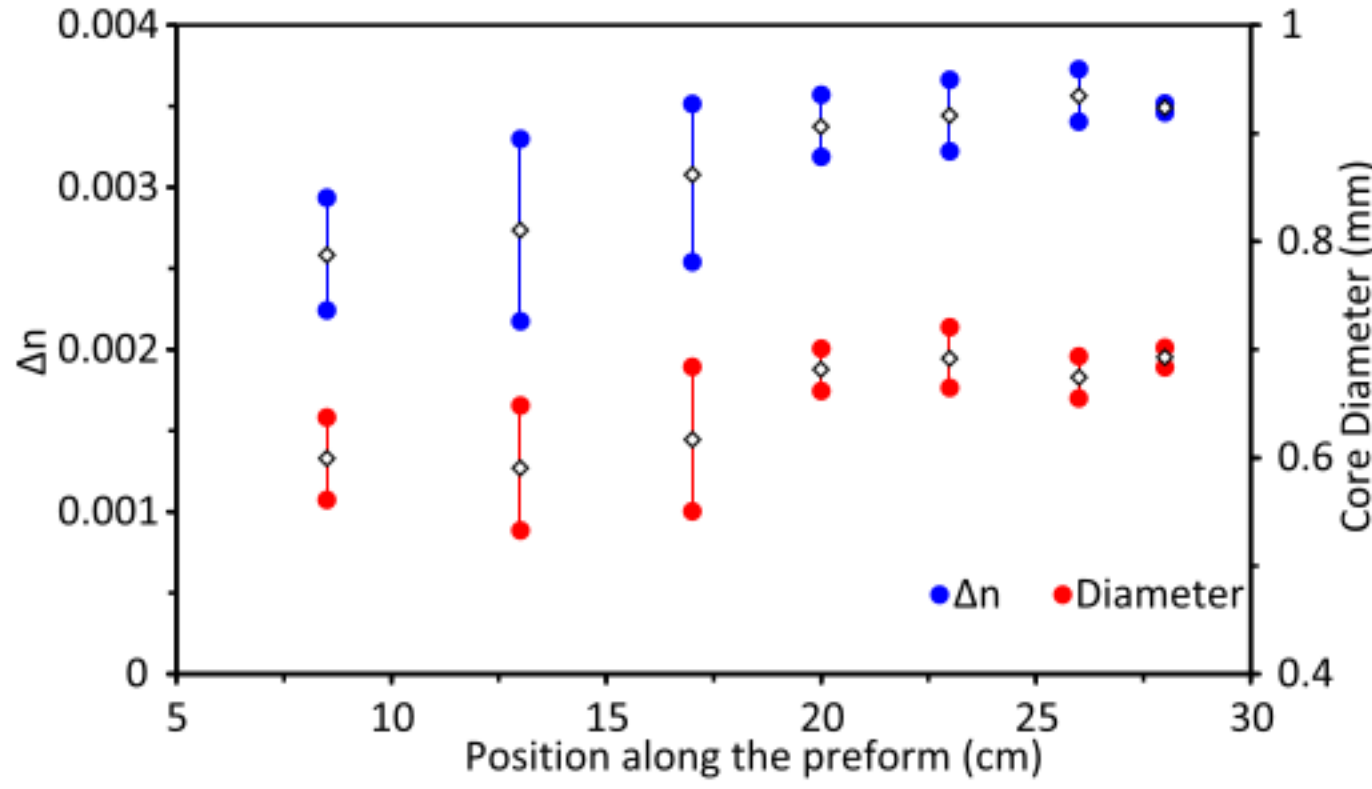


Fig. 3. Refractive index differences and core diameters measured along the *Cst-Al* preform. The preform was soaked one and two times for the positions below and higher than 17 cm, respectively.

there is no V part. The higher and lower parts of the preform (the separation is at 17 cm) were soaked one and two times, respectively. The soaking time is one hour for each steps. The porous layer was not oxidized nor rinsed between both soaking. The refractive index profiles of the preforms were measured with a York Technology Preform Analyzer Model P101 at two azimuthal angles separated by 90° . If the core is slightly elliptical, the refractive index contrast (Δn) that is calculated from a beam deflection measurement, may suffer large variations between 0° and 90° angles. The real Δn value is approximately the average of both measurements. The refractive index differences of the singly and double solution-soaking parts are $2.5-3 \times 10^{-3}$ and $3-3.5 \times 10^{-3}$, respectively (Fig. 3). The core diameter slightly increases also from 0.6 to 0.67 mm (Fig. 3). The large deviations in both the refractive index difference and the core diameters are due to the ellipticity of the core. As a conclusion, when the same solution is used to soak the porous layer twice without performing any oxidation step in between, one shall expect only a limited increase of the concentration of invariant element (aluminum in this case). Instead, if oxidation steps are performed between soakings, much stronger increase is expected, as demonstrated by F. Tang *et al.* [13], [14].

C. The Case of a Two-Composition Preform Without V Part

The *Cst-Al2* preform was prepared with a two solution soaking and without V part (as for *Cst-Al*). The variation of the refractive index along the preform *Cst-Al2* is reported in the Fig. 4. In the case of this two-composition preform (without V part), the refractive index change between both parts is not sharp.

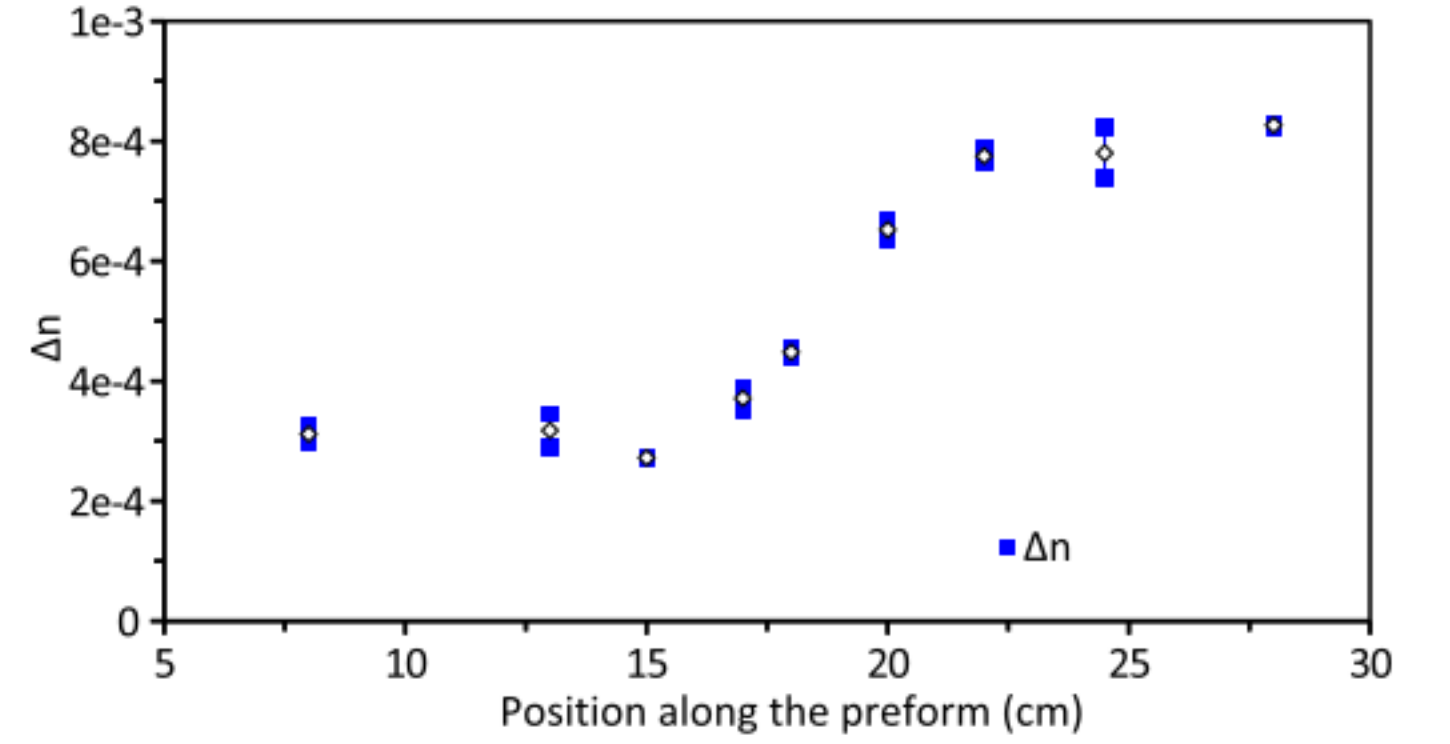


Fig. 4. Refractive index differences measured in the preform *Cst-Al2*.

The refractive index between both parts changes over a length of 5 cm (~ 290 m of fibers). This variation could be explained by the capillarity diffusion occurring during the second soaking or gradient concentration induced element diffusion during the second soaking or the collapsing pass.

D. Varying Aluminum Content in the Sintered Core Layer

A tube with the same porous layer as discussed in the previous section was soaked according to the *Grad-Al* recipe, then sintered. The longitudinal concentration of aluminum of this sintered layer (prior to the collapsing step) was estimated with EDX (Energy Dispersive X-Ray analyses) measurements on a JSM 7000F JEOL (Ltd, Tokyo, Japan) microscope. The accelerating voltage, acquisition time and magnification were 20 kV, 70 s and $\times 500$, respectively. The thulium concentration is too low to be accurately determined and it is not discussed here. The results are presented in the Fig. 5. Al_2O_3 concentrations in the T and B parts are 6.7 and 19 wt% (4 and 12 mol%), respectively. The concentration ratio in the sintered layer between both parts is very close to the one of the doping solutions (0.7 and 2 mol/l). In the V part (between 13 and 23 cm), Al_2O_3 concentration increases almost linearly. Then, in this case, one can estimate that the time to reach the equilibrium of the concentration is about 20 minutes. One can observe some variation of the refractive index in the B part, this can be attributed to the inhomogeneity of the porous layer in this area.

E. Varying Preforms and Fibers

In this section, we are interested in the variations of composition measured in the preforms and the fibers. The refractive

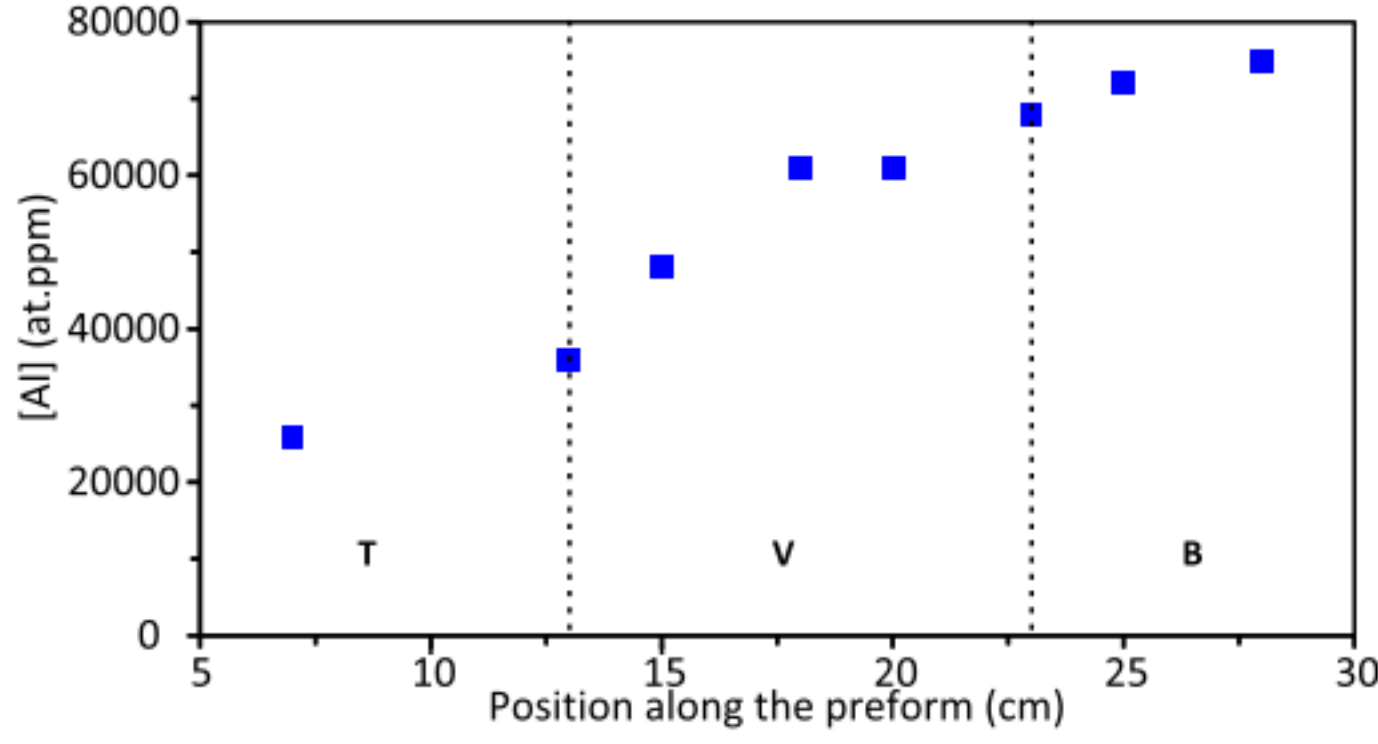


Fig. 5. Aluminum concentration along the sintered layer (before collapsing) prepared according to the *Grad-Al*.

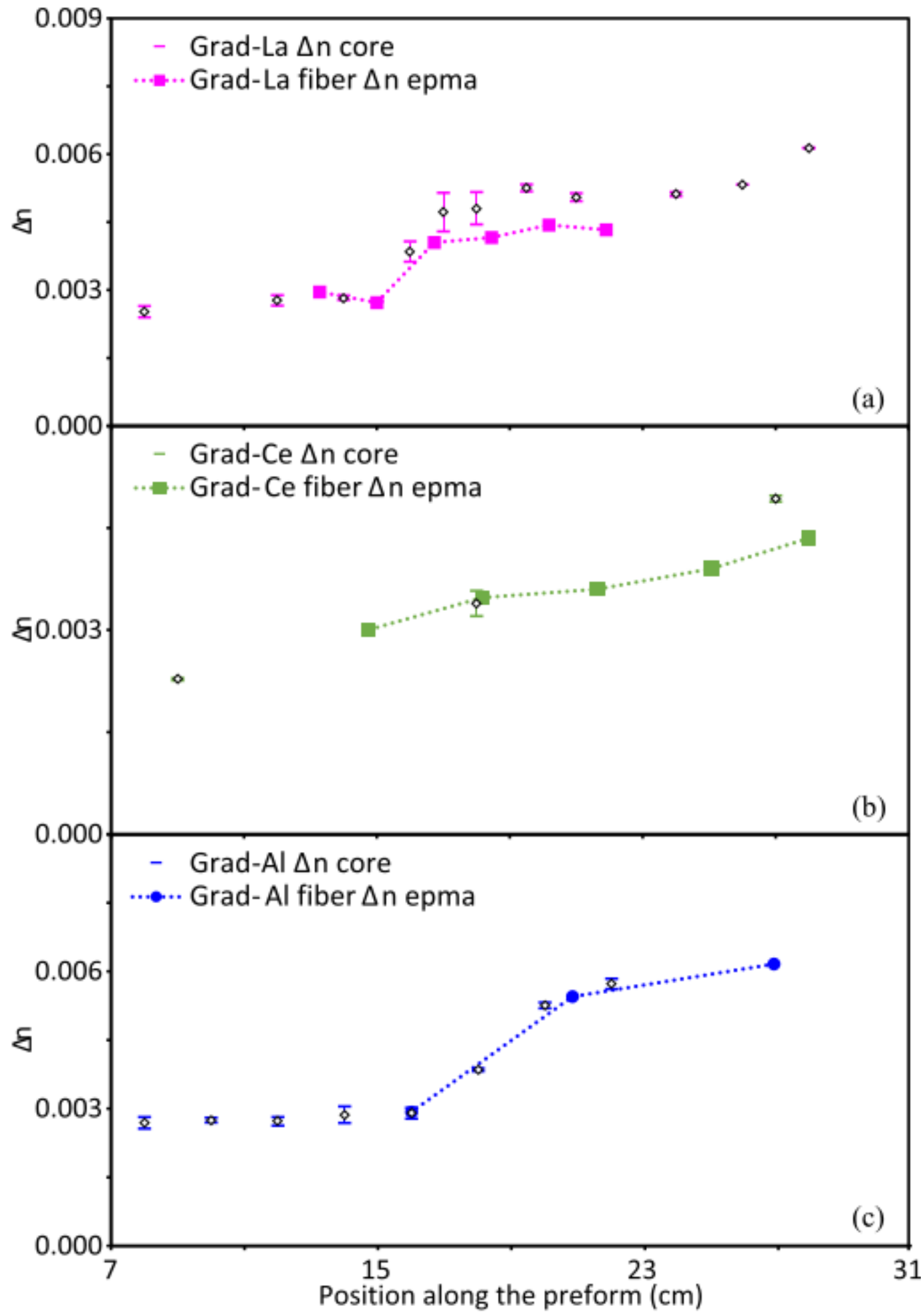


Fig. 6. Refractive index differences measured in the preforms (dot) and estimated by EPMA measurements in fibers (dotted-dashed line) for the samples *Grad-La* (a), *Grad-Ce* (b) and *Grad-Al* (c).

index differences and the core diameters of *Grad-Al*, *Grad-Ce* and *Grad-La* preforms are reported in Figs. 6 and 7, respectively. The refractive index difference varies as expected due to the doping process: lower and higher values in the T and B parts, respectively. In the V part, the refractive index increases almost linearly. The length of the V part depends on the preforms. It is 8, 12 and 11 cm for *Grad-La*, *Grad-Ce* and *Grad-Al*, respectively. These lengths are close to the 10 cm length of the V part

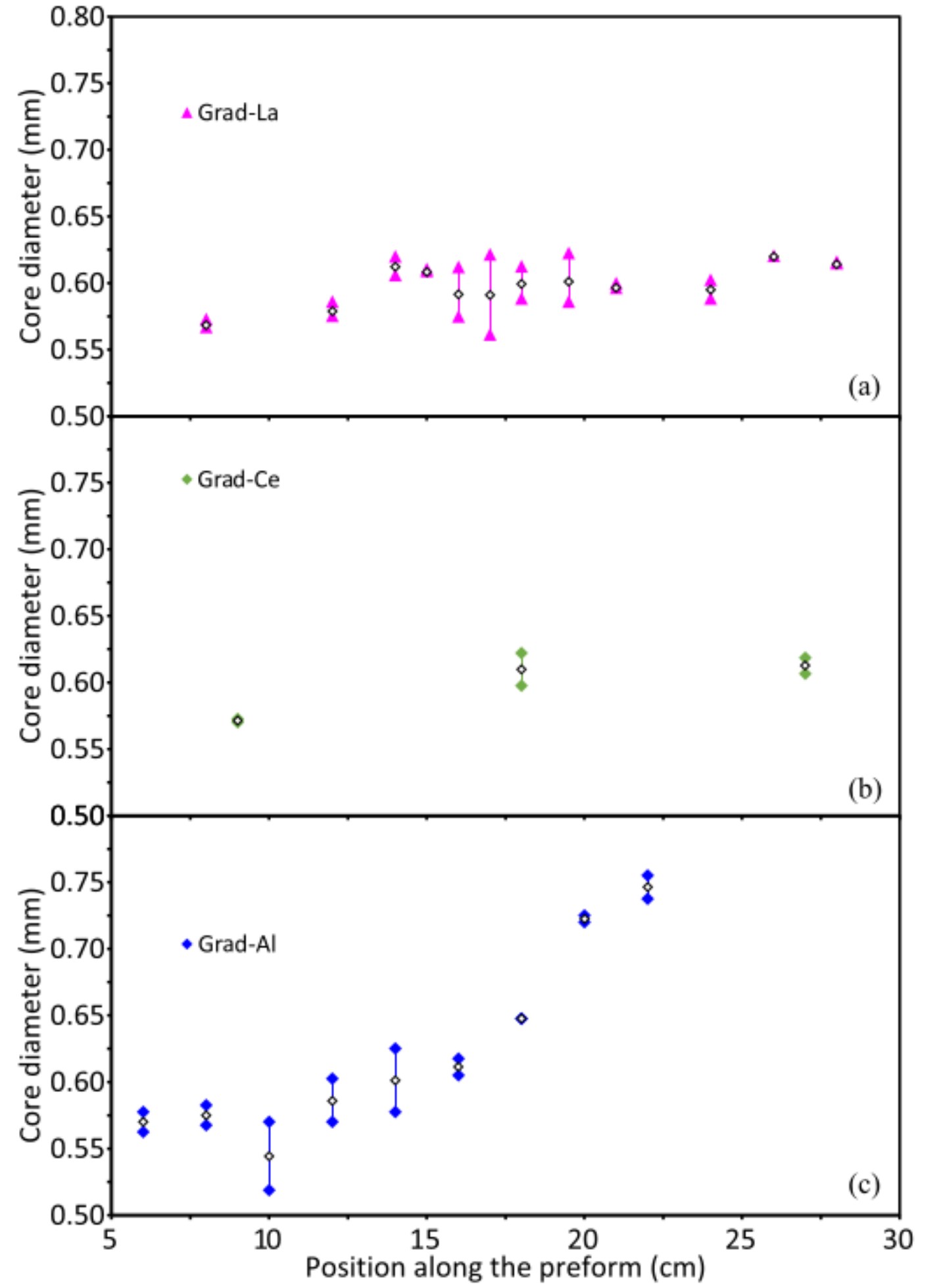


Fig. 7. Core diameters estimated from the refractive index profiles of the preforms *Grad-La* (a), *Grad-Ce* (b) and *Grad-Al* (c).

targeted during the doping process. The sensible variation of the core diameter is related to the increase of the refractive index in the *Grad-Al* preform. This is explained by the diffusion of aluminum in the cladding as already observed by [32] due to the high temperature occurring during the collapsing step. For preforms *Grad-Ce* and *Grad-La* (aluminum is an invariant element), the core diameters slightly increases from the T to the B part, from 0.57 to 0.6 mm. This increase is small and is similar to the one observed for the *Cst-Al* preform (Fig. 3). Moreover, a higher content of rare-earth ions in the B part may also induce a larger diameter.

Quantitative electron probe micro-analyzer (EPMA) analyses were made on cleaved fibers with a CAMECA (France) SX-FIVE electron microprobe using a 20 kV acceleration voltage. Aluminum, thulium, cerium and lanthanum concentrations along the fibers are reported in the Fig. 8. In all these preforms, thulium ions are invariant elements (same concentration in solution A and B), and its concentration is almost constant all along the fibers. The same occurs for aluminum in the *Grad-La* and *Grad-Ce* fibers where thulium is an invariant element also. These measurements confirm the reproducibility of the process because the same concentration of the invariant element in both solutions leads to the same concentration in the final fibers. In-

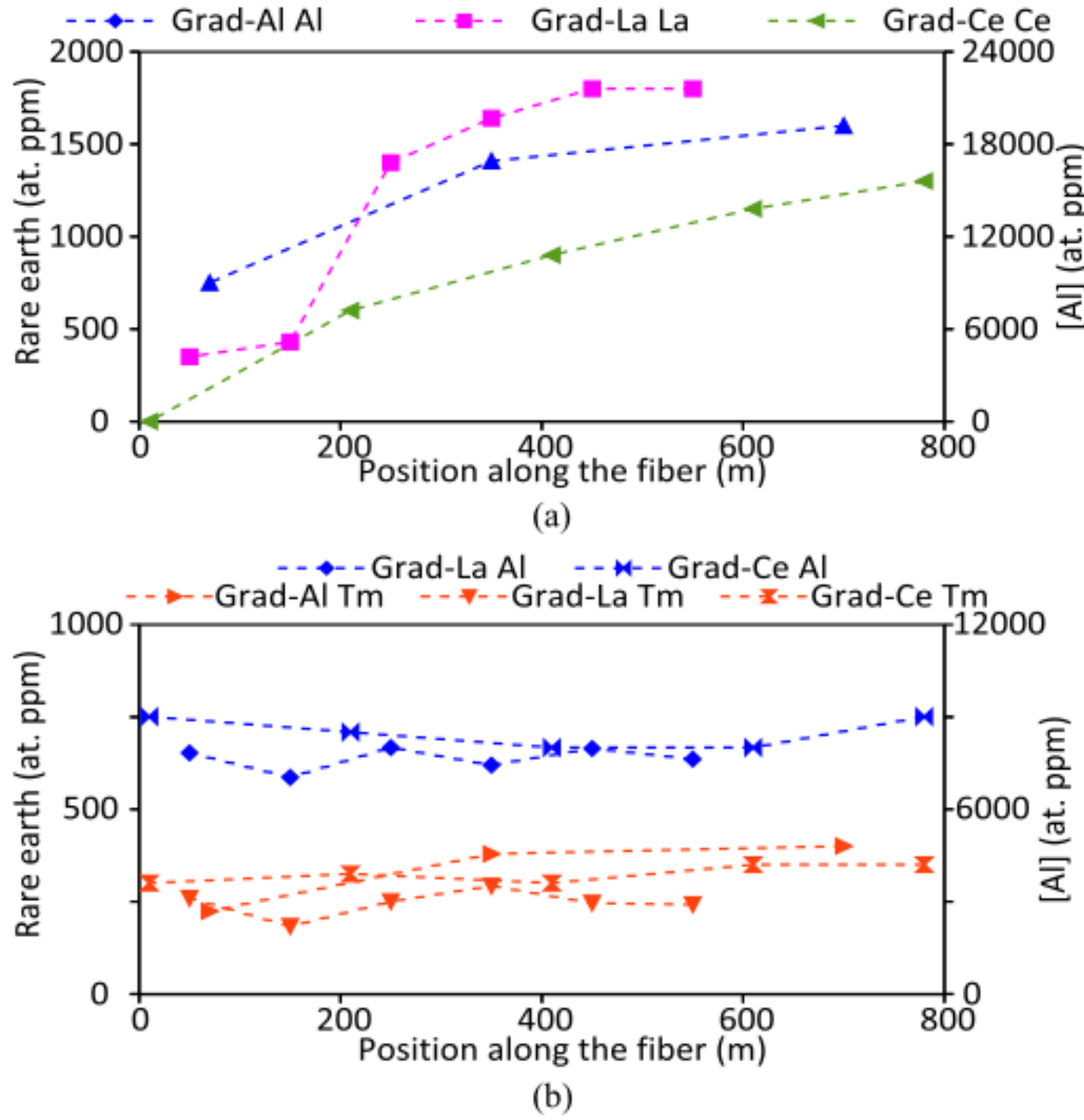


Fig. 8. Al, La, Ce and Tm concentrations measured by EPMA as a function of the position in fibres drawn from the preforms *Grad-La*, *Grad-Ce* and *Grad-Al*. All values are in atomic ppm. Variant and invariant elements are reported in (a) and (b), respectively.

deed, thulium concentration is about 300 ppm.at for the three preforms, and aluminum concentration is about 8000 ppm.at in *Grad-La* and *Grad-Ce*.

For the variant elements, there is a clear variation of its concentrations along the fibers, as expected with the process. The variation of the concentration in the fibers are directly related to the variation of the refractive indices measured in the preforms. These concentrations were used to estimate the refractive index at each position. The calculated refractive indices are reported in the Fig. 6. To reach a satisfying match between both measurement series, we used these conversion factors for the refractive index increment: $+0.3 \times 10^{-3}/1000 \text{ ppm.at } Al^{3+}$ and $+1 \times 10^{-3}/1000 \text{ ppm.at } RE^{3+}$ ($RE^{3+} = La^{3+}, Ce^{3+}$ or Tm^{3+}). It has been reported previously that the refractive index of alumino-silicate glasses decreases when the ionic radius of the rare-earth ions decreases [33]. But this variation is typically 10% only. Therefore, in our calculation, we assumed the same refractive index increment for all rare-earth ions. These increments are in agreement with those reported by [32].

V. DISCUSSION

Thanks to the gradual time doping solution, we were able to prepare fibers with longitudinally varying refractive index differences and core diameters. Regarding the values reported in this article, a 50% increase of the refractive index can be reached over 100 m (*Grad-La*) or 500 m (*Grad-Al*). Variation over such length is of interest in the frame of the systematic compositional screening approach of the composition as the fiber composition for typical characterisations can be assumed

homogeneous over 10 meters. For the fibers requiring a variation over a shorter length, several approaches are possible. One route would be to remove the V part as in the case of the *Cst-Al2* preform. In this case, a short varying length was obtained and could be engineered by changing the porosity of the core layer for instance. Moreover, as this varying length depends on the ratio as the squares of the diameters of the preform and the fiber, the varying zone can be shortened in the fiber by reducing the diameter of the preform or increasing the diameter of the fiber.

VI. CONCLUSION

A variant of the standard solution doping technique has been proposed to prepare fibers with longitudinally varying characteristics (concentration, refractive index, core diameter) along the fiber. This method is based on the variation of the soaking time and the use of two doping solutions with variant and invariant elements. While the concentration of the invariants elements is constant all along the fiber, the concentration of the variant element depends on the soaking time. These results were reported for aluminum and rare-earth ions which are common doping elements for fibers. Thanks to this process, we have reported a variation of the fiber characteristics over 100 to 500 meters. Such variations are of interest to screen the relation composition-properties. It is also discussed on the opportunity to shorten this varying length. All these results highlight the efficiency, the simplicity and the low cost of our variant technique to perform rapid material development in optical-fiber domain.

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Jean-François Lupi received the Ph.D. degree in physics from the University of Nice Sophia Antipolis, Nice, France, in 2016. After his Ph.D. on photo-darkening, he is currently involved in the field of mode instability in power fiber laser with the Danmarks Tekniske Universitet, Lyngby, Denmark.

Manuel Vermillac received the Master's degree in physics, science of materials, from the University of Nice Sophia Antipolis, Nice, France, in 2014. He is currently working toward the Ph.D. degree in the field of glass science, nanoparticles, and optical fibers in the Institute of Physics of Nice, Valbonne, France.

Stanislaw Trzesien received the advanced chemistry technician Diploma in 2003. Since 2014, he is currently working toward the chemical and process engineering degree at CNAM, Paris, France. He joined the LPMC (now Institut de Physique de Nice) laboratory in 2005 as a material development technician on optical fibers manufacturing platform, in parallel he became Safety and Security Assistant in 2009.

Michèle Ude received the Technical degree in physical measurements in 1987. She joined IRSTEA, Antony, France, as an Engineer Assistant for design, realization, and use of experimental models. Since 2004, as an Engineer in scientific instrumentation and experimental techniques, she has been in charge of management, improvement, use and service of the MCVD Preforms Manufacturing Centre and the associated equipment (drawing tower, characterization equipment) in the Laboratoire de Physique de la Matière Condensée, Nice, France.

Wilfried Blanc was born in Paris, France, in 1973. He received the Ph.D. degree in physics in 2000 from the University Claude Bernard, Lyon, France. From 2000 to 2002, he was a Postdoc with the ICMCB (Institut de Chimie de la Matière Condensée, Bordeaux, France), funded by Rhodia. In 2002, he joined the Laboratoire de Physique de la Matière Condensée (now Institut de Physique de Nice), Centre National de la Recherche Scientifique (CNRS), where his main interests are the design, realization, and characterization of rare-earth-doped silica optical fibers, which are made by using a modified chemical vapor deposition technique. He has authored and coauthored 55 research papers in international journals.

Bernard Dussardier was born in Athens, Greece, in 1966. He received the Graduate degree in optical physics and engineering from the Institut d'Optique Graduate School, Orsay, France, in 1989, and the Ph.D. degree from the University of Nice Sophia-Antipolis (UNS), Nice, France, in 1992. From 1992 to 1995, he was a Research Fellow with the Optoelectronics Research Centre, University of Southampton, Southampton, U.K. From 1995 to 2013, he was a CNRS Research Scientist, and since 2013 the CNRS Research Director (Centre National de la Recherche Scientifique) with LPMC (Condensed Matter Physics Laboratories), UNS. Since 2001, he has been leading the Optical Fiber team with LPMC, now InPhyNi (Institute of Physics in Nice). He has coauthored more than 80 publications and proceedings, and more than 160 conferences papers. His research interests include specialty optical fibers, spectroscopy of rare-earth in glasses and glass-ceramics, fiber lasers, fiber components for telecommunications, and fiber sensors. He is in charge of the Specialty Optical Fiber fabrication facility with InPhyNi.