Growth of rare-earth doped oxyde nanoparticles in silica fibers", 9th Symposium "SiO2: Advanced Dielectrics and Related Devices

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ABSTRACT

Rare earth (RE) doped silica-based optical fibers with transparent glass ceramic (TGC) core was fabricated through the well-known modified chemical vapor deposition (MCVD) process without going through the commonly used stage of post-ceramming. The main characteristics of the RE-doped oxyde nanoparticles, their density and mean diameter in the fibers are dictated by various parameters discussed during this presentation. Low scattering losses induced by nano-scale particles and alteration of the spectroscopic properties of the erbium ions were observed. These initial studies should be useful in incorporating new doped materials in order to realize active optical fibers for constructing lasers and amplifiers.

PACS Keywords: silica, nanoparticles, erbium, alkaline earth.

1 INTRODUCTION

Developing of new rare-earth (RE)-doped optical fibers for power amplifiers and lasers requires continuous improvements in the fiber's spectroscopic properties (like gain and efficiency characteristics, resistance to spectral hole burning and photodarkening,…) besides reduction in device size and economical efficiency. Silica glass as a host material for fibers has proved to be very attractive. However some potential applications of RE-doped fibers suffer from limitations in terms of spectroscopic properties resulting from clustering or inappropriate local environment when doped into silica.

The route of interest here consists of using silica as a mechanical host and support of the fiber optical waveguide, and of embedding RE-ions within oxide nanoparticles of composition and structure different from those of silica, and small enough to induce acceptable scattering loss. We have proposed a straightforward technique allowing to embed RE ions within *in-situ* grown oxide nanoparticles in silica-based preforms [1]. The implemented principle is the spontaneous phase separation process. Two key advantages of this process are that (i) nanoparticles are grown *in-situ* during the course of the fabrication process and (ii) there is no need (and associated risks) of nanoparticles manipulation by an operator. Further, the process takes advantage of the high compositional control and purity typical of the MCVD technique.

In the present paper, we report on the growth of erbium-doped MO-silicate nanoparticles, that are stable after the fiber drawing stage using the MCVD and solution doping techniques. This demonstrates the potentiality of this fabrication technique for applications such as fiber amplifiers.

2 EXPERIMENTAL DETAILS

Preforms were fabricated by the conventional MVCD technique. In this process, gaseous chlorides ($SiCl_4$, $GeCl_4$, $POCl_3$) are passed through a rotating silica tube, heated by an external burner in translation along the tube. Due to the high temperature, chlorides oxidize, forming particles which deposit on the inner wall. This porous layer turns into a glassy layer when the burner passes over it (the tube external temperature is around 1500 °C). In the final stage, the tube is collapsed into a rod at a temperature higher than 1800°C. fibers were obtained by stretching preforms in a drawing tower at temperatures higher than 2000 °C under otherwise normal conditions. Opto-geometric properties of the perform (or fiber) are determined by adjusting the composition and number of layers. In our samples, phosphorous and germanium concentrations are ~0.5 mol% and 1 mol%, respectively.

Erbium and alkaline-earth ions were incorporated through the solution doping technique. An alcoholic solution (of desired strength of $ErCl_3.6H_2O$ or $MCl_2.6H_2O$) is soaked for two hours in the unsintered core layer. After removing the solution, the layer is dried and sintered. Erbium concentration is estimated through absorption spectra to be around 300 ppm Is is comparable with the concentration in standard Erbium-Doped Fiber Amplifier. Two alkaline-earth concentrations in the solution were prepared: 0.1 and 1 mol/l. The core diameter in the preforms was measured with a preform analyzer (York Technology P 101) to be 1 mm. It is about 8 µm in fibers.

3 NANOPARTICLES CHARACTERIZATIONS

Nanoparticles were characterized through scanning electron microscopy (SEM) in the backscattered electrons mode. Typical SEM picture from the exposed core section of cleaved fibers is shown on Fig. 1 (Mg concentration in the solution is 0.1 mol/l).

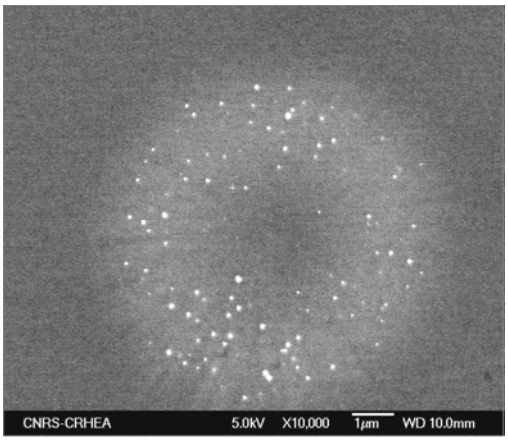

Figure 1: SEM picture of a Mg-doped fiber

The gray disk corresponds to the fiber core. Nanoparticles, visible as bright spots, are only observed when alkaline-earth ions (Mg, Ca or Sr) are added. They show an important compositional contrast compared to the silica background. The dark central part of the core is caused by the evaporation of germanium element; this is a common artifact of the MCVD technique that can be corrected through process optimization.

The statistical histograms of the size distribution of the NP for 0.1 mol/l Mg-doped preform and fiber are presented in Fig. 2. The mean particle size is 50 nm in the preform and 40 nm in the fiber. Those histograms show that the NP are preserved during the drawing step, in spite of the high temperature of this stage (~2000 °C). Moreover, the size distribution is slightly affected by this step : the mean size decreases from 50 nm downto 40 nm, in the preform and the fiber, respectively, while the core diameter is divided by 100.

When the Mg solution concentration increases from 0.1 to 1 mol/l, the mean particle diameter almost doubles to reach 80 nm. In this fiber, NPs up to 160 nm were observed. Ca and Sr incorporations lead also to particle with a mean diameter of 100 nm, even for a concentration in the solution of 0.1 mol/l.

Regarding light attenuation, fibers prepared with 0.1 mol/l of Mg are compatible with applications as losses as low as 0.4 dB/m were measured [2]. Other fibers, with 100-nm NP, lead to high attenuation level, above 100 dB/m.

Erbium spectroscopy is modified by the presence of the NP. Two behaviors are observed : in the case of low Mg concentration, Erbium spectroscopy is related to silica-based sample, while for high Mg concentration, Er spectroscopy is clearly related to phosphate environment [2].

During the presentation, we will discuss the influence of the MCVD parameters (alkaline-earth concentrations, deposit temperature, sintering temperature, collapsing stage) on the growth of NP in order to control their sizes. Relation with erbium spectroscopy will also be reported.

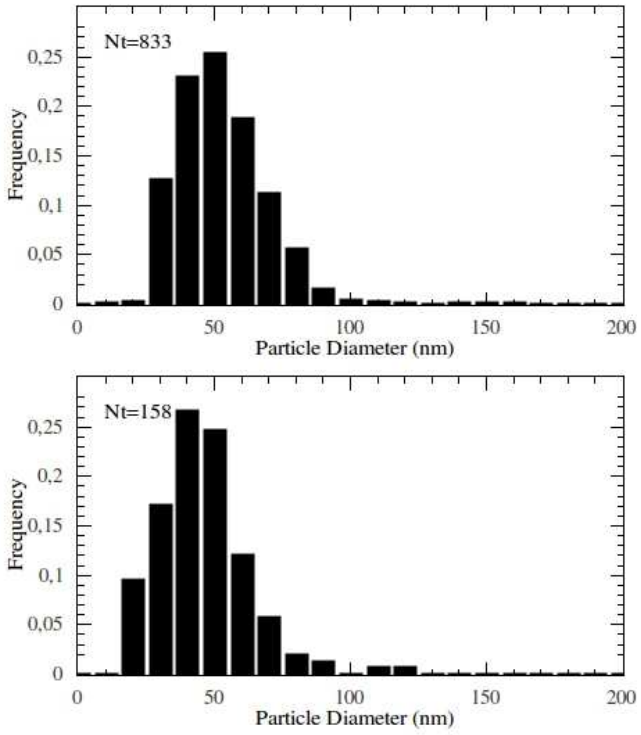

Figure 2: Histograms of the particle size in Mg-doped preform (top) and optical fiber (bottom).

4 CONCLUSION

A method to fabricate nanostructured Er^{3+}-doped fibers entirely through the MCVD process is demonstrated. By adding magnesium to the silica-based composition, nanoparticles of 40 nm in diameter are obtained through *in situ* growth without requiring a separate process to realize nanoparticles (such as post-process ceramming). Such fibers have low-loss. Moreover, a broadening of the emission spectrum by as much as almost 50% is observed with attractive features to realize gain flattened fiber amplifiers. More generally, this concept has great potentials as possible solutions to nowadays issues in amplifying fibers, including (but not limited to) amplifiers intrinsic gain flattening and/or spectral hole burning, amplifiers irradiation strengthening, photodarkening in fiber lasers, etc.

REFERENCES

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