## The drawing stage as a crucial step to engineer nanoparticles in optical fibers

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## Abstract

The evolution of optical fiber applications has always been supported by the evolution of materials and the development of new associated manufacturing processes (1). The paradigmatic example is the improvement of the transparency of silica fibers obtained via the development of chemical vapor processes leading to the tremendous development of telecommunications. Among the diversity of materials studied, optical fibers containing nanoparticles were proposed about twenty years ago to improve luminescence properties via the encapsulation of rare earth ions or transition metals (2). More recently, such optical fibers have shown a strong potential for the realization of sensors (temperature, stress, chemical environment, dosimetry, etc.) (3). All these applications depend on our ability to control the characteristics of the nanoparticles in the fibers (4). To achieve this goal, we focus in this presentation on the drawing stage of the optical fiber preform.

The preforms were prepared by the MCVD (Modified Chemical Vapor Deposition) process. A porous layer of silica doped with germanium was deposited inside a silica tube. This porous layer was soaked with an alcoholic solution in which erbium and lanthanum chlorides were dissolved. Different concentrations of lanthanum were used (0.7 to 0.175 mol/l). The solution was removed after 2 hours and the porous layer was densified before collapsing the tube into a preform of 1 cm in diameter. As final step, preforms were drawn under various conditions by varying the temperature and the drawing speed. The diameter of the fibers is 125  $\mu$ m.

Lanthanum ions are added to initiate the formation of nanoparticles via phase separation mechanisms involving the instability of the SiO2-La2O3 binary system at the high temperatures inherent to the MCVD process (up to 2100  $\circ$ C). Scanning electron microscopy (SEM) analyses show that the increase of the La concentration leads to an increase of the average particle size from 75 to 250 nm. These particle sizes lead to a very strong light scattering, characterized by the whitish color of the core.

In order to study the effect of drawing on the nanoparticles, different conditions were tested by varying the temperature between 1800 and 2050  $\circ$ C and the drawing speed between 15 and 30 m/min. For all fibers, the final diameter was maintained at 125  $\mu$ m. Energy Dispersive X-Ray (EDX) analysis revealed that the nanoparticles are lanthanum silicates, more specifically of La2SiO7 composition, while being amorphous. Considering the elongation of the nanoparticles observed previously (5), the longitudinal sections of the fiber cores were

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analyzed by SEM as well as by SEM coupled with focused ion beam (SEM/FIB) cutting for a 3D analysis of a volume of the optical fiber core (6). These analyses show that an increase in the drawing temperature or a decrease in the drawing speed leads to a decrease in the particle size. A low drawing temperature or a fast drawing speed leads to an elongation of the particles or even to their fragmentation. These elongation and fragmentation mechanisms will be discussed in the presentation based on known fluid dynamics mechanisms and Rayleigh-Plateau instabilities. To analyze the effects of the drawing conditions, a numerical model has been developed to follow the thermal history of the fiber throughout the drawing process and to determine the capillary number. The confrontation between the numerical results and the experimental results will be discussed during the presentation.

All these results allow to highlight that the drawing step is a crucial step for the characteristics of nanoparticles in optical fibers and that it allows to realize a real engineering of their size and shape.

## References

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Keywords: Optical fibers, nanoparticles, phase separation, Rayleigh, Plateau instability