

## Optical quality improvement of rare-earth doped silica layers by CO<sub>2</sub> laser irradiation

Gil Atar<sup>a,b,\*</sup>, David Eger<sup>a</sup>, Ariel Bruner<sup>a</sup>, Bruno Sfez<sup>a</sup>, Menachem Nathan<sup>b</sup>

<sup>a</sup> Applied Physics Division, Soreq NRC, Yavne 81800, Israel

<sup>b</sup> Department of Electrical Engineering—Physical Electronics, Faculty of Engineering, Tel Aviv University, Ramat Aviv, Tel Aviv 69978, Israel

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### ABSTRACT

We investigate different types of structural defects in 20–30 μm thick Yb/Al-codoped fused silica layers on a pure fused silica substrate, and show that all types of structural defects can be treated in a single laser heat treatment. Laser-induced processes of defect elimination include diffusion of micro-voids, amorphization of crystallites and roughness reduction via surface-tension-driven mass flow. The physical mechanisms for defect elimination are analyzed in terms of onset temperature and typical time of elimination. Results of such treatment include complete amorphization, reduction of surface roughness by an order of magnitude to about 15 nm, and a remarkable improvement of more than an order of magnitude in optical cross-transmittance. The treatment thus provides a possible route for producing lasers and high-power integrated optics components using silica-on-silica technology.

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### 1. Introduction

Over the past decade, CO<sub>2</sub> laser treatment was proven to be a versatile tool for inducing surface modification of glass for various applications. In such treatment, a shallow layer of glass undergoes melting, flowing and resolidification. CO<sub>2</sub> laser processing was used to shape and perfectly smooth ultra-high-Q silica toroid microcavity [1] and for direct writing of long-period-gratings on silica fibers by inducing a local change in refractive index (probably by means of residual stress relief [2]). The fast thermal cycle associated with laser processing was shown to affect the fictive temperature of silica glass and its resistivity to wet etching [3].

Several studies dealt with the use of CO<sub>2</sub> lasers to reduce surface roughness in various types of glasses [4–7]. Important contributions were made by Mendez et al. [8] who successfully used a CO<sub>2</sub> laser to polish micro-optical elements, and by Nowak et al. [9] who ran a comprehensive investigation of the underlying mechanism of laser polishing, i.e. surface-tension-driven mass flow. CO<sub>2</sub> laser treatment was also successfully used to induce amorphization/crystallization of glass–ceramics [10], and in

annealing for mitigation of high power laser-induced damage in fused silica (FS) [11,12]. Each study above targeted a single type of structural defect, hence requiring a particular set of spatial and temporal characteristics of the laser induced temperature field. No report was made on the use of a CO<sub>2</sub> laser to improve the optical quality of sintered FS which contains several kinds of structural defects.

In this study, we investigate the structural defects in 20–30 μm thick sintered FS layers and show that a CO<sub>2</sub> laser can be used to improve its optical quality by removing trapped micro-voids via diffusion, by inducing amorphization of micro-crystallites and by significantly reducing surface roughness via surface-tension-driven mass flow, all in a single process.

Rare-earth doped FS is commonly used as core material in large mode area (LMA) fibers, which today constitute one of the leading platforms for high power lasers [13]. In an effort to fabricate an on-chip waveguide analogous to the LMA fiber, we recently reported a process for producing a 20–30 μm thick Yb/Al-codoped FS layer using silica-on-silica technology [14]. The fabrication scheme is a modified version of the fiber fabrication method and is based on direct nanoparticle deposition (DND) of Yb/Al-codoped silica nanoparticles, followed by high temperature sintering and CO<sub>2</sub> laser heat treatment. In the DND process (a version of the traditional flame hydrolysis), a 100 μm thick layer of doped silica nanoparticles (soot) is deposited on a pure FS substrate (Corning UV-grade 7980, 12 × 12 × 6.35 mm<sup>3</sup>). The soot contains controlled Yb and Al doping levels to achieve desired absorption and Δn values

Abbreviations: FS, fused silica; LMA, large mode area; DND, direct nanoparticles deposition; WG, waveguide; PWG, planar waveguide; CW, continuous wave; LPF, low pass filter.

\* Corresponding author at: Applied Physics Division, Soreq NRC, Yavne 81800, Israel. Tel.: +972 8 9434131, mobile: +972 50 6292272; fax: +972 9 9434503.

E-mail address: [gilatar@soreq.gov.il](mailto:gilatar@soreq.gov.il) (G. Atar).

and its density is 3–5 times lower than FS, i.e. 0.4–0.7 g/cm<sup>3</sup>, depending on deposition temperature. In the process of sintering, the sample is heated to 1500–1600 °C, where the soot layer undergoes amorphous densification to reach a final thickness of 20–30 μm. Reactive ion etching can then be applied to form the desired shape of the waveguide and the process can be repeated for depositing a clad layer with highly controlled  $\Delta n$  value.

Ideally, the product of the sintering stage is a homogeneously dense amorphous matrix of doped FS. However, in practice the layer contains significant amount of structural defects which serve as scattering centers for guided light. Therefore, the sintered FS is merely a semi-transparent glass layer (much like the glass rod obtained in the case of fiber production) and an additional processing step is required to eliminate the structural defects and improve the optical transmittance of the core material. In the case of fiber production, all structural defects in the sintered FS are eliminated by melting the glass and consequentially drawing it into a fiber at temperatures above 2000 °C. In the case of on-chip waveguide (WG) fabrication, since no mechanical drawing is performed, an alternative method is required to apply a high temperature heat treatment and induce sufficient glass flow to eliminate all structural defects and obtain a highly transparent core material. The objective of this study was to investigate the different types of structural defects present in sintered Yb/Al-codoped FS, analyze potential routes for its elimination and to develop a simple and innovative method to improve the optical quality of sintered doped FS on pure FS substrate.

A possible way to perform the heat treatment would be to use a conventional furnace to melt the sintered FS. However, simply heating the sample to a temperature above 2000 °C would result in overall melting of both the sintered core layer and the holding substrate, leading to a collapse of the planar structure. An apparent solution would be to use a different material than pure FS for the substrate. A material such as silicon carbide features a higher melting point than FS and may seemingly act as a solid substrate in the process of melting the sintered layer. However, even a small mismatch between thermal expansion coefficients may give rise to cracks, as fast heating and cooling rates are essential to avoid crystallization of the FS. Furthermore, any contact with a foreign material at such high temperature might result in severe contamination. Therefore, the choice of substrate material is practically restricted to pure FS, which calls for a controlled method to heat only the top layer of the FS sample to very high temperatures while maintaining the substrate solid. The CO<sub>2</sub> laser is attractive as a tool for this purpose, because of the short absorption length of its 10.6 μm wavelength in FS. The CO<sub>2</sub> laser cannot be applied directly on the soot layer due to the strong scattering of the soot. As a result, the soot heats very fast and non-uniformly causing uncontrollable ablation.

Accomplishing efficient elimination of all types of structural defects present in sintered FS would enable thick and highly doped FS WGs to be produced on FS substrates. The on-chip waveguide technology offers great flexibility in optical circuit design using microelectronics methods. Such capability to generate and manipulate high power laser light on a silica chip may provide, in principle, novel schemes for pumping and beam combining, an important advantage over fiber technology.

In Section 2, the various types of structural defects are investigated and the heat-activated physical mechanisms for eliminating them are analyzed in terms of onset temperatures and typical duration. In Section 3, considerations for choosing the laser heating work-mode are detailed, as well as a description of the experimental system and process parameters for eliminating all three types of structural defects in a single process. Qualitative and quantitative measurements, showing a remarkable improvement in optical quality following the CO<sub>2</sub> laser treatment, are discussed in Section 4.

## 2. Structural defects analysis

### 2.1. Micro-scale voids and crystallites

Fig. 1a shows a dark-field transmission micrograph of the sintered doped FS layer. The nearly perfect circles are identified as residual voids, i.e. inclusions in the FS layer. Their different sizes and gray levels indicate they do not represent a surface phenomenon, but that the voids are distributed through the entire depth of the 30 μm thick layer. Voids are formed during sintering, as glass particles move towards one another and join together, creating larger glass particles with internal closed spaces. Once a void is formed, a process of diffusion is believed to govern the outgassing of the trapped gas during the high temperature sintering [15]. In this process, a constant decrease in the size of the void gradually occurs as densification takes place. However, the duration of the sintering process is limited by a simultaneous process of crystallization. Thus, the sintering is often terminated prematurely, leaving micrometric scaled residual voids.

The polygon-like shapes seen in Fig. 1 are identified as crystallites, i.e. sites of solid silica. The undesired process of crystallization occurs since the foreign phase in the FS soot acts as nucleation sites and may lead to crystal growth. As mentioned, in order to avoid excessive growth of crystalline sites, the sintering process is often terminated prematurely, thus the FS layer is characterized by a moderate presence of both crystallites and residual voids. In order to identify the particular crystalline phase of silica seen in Fig. 1a,

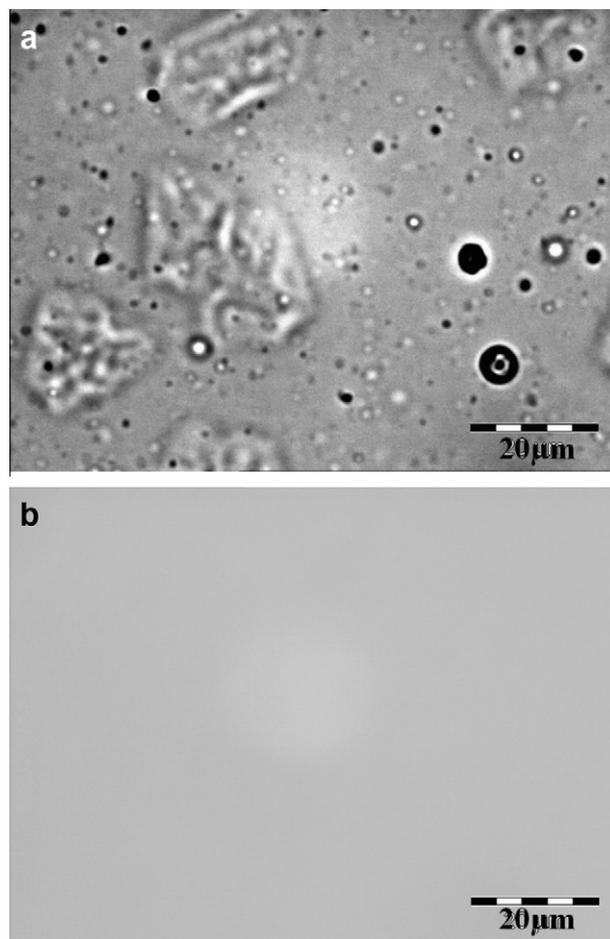


Fig. 1. Dark-field transmission micrographs showing crystallites and voids in the as-sintered FS layer (a), and a homogeneous FS layer following CO<sub>2</sub> laser treatment (b).

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