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# Bragg gratings in ternary SiO<sub>2</sub>:SnO<sub>2</sub>:Na<sub>2</sub>O optical glass fibres

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#### **Abstract**

Bragg gratings have been written in an optical fibre with a core made of ternary  $SiO_2:SnO_2:Na_2O$  glass and a  $SiO_2$  cladding. The presence of  $Na_2O$  allows for higher concentrations of  $SnO_2$  which is believed to be responsible for the photorefractive response of this composition. In these preliminary experiments significant refractive index modulations, up to  $6.2 \cdot 10^{-4}$ , have been achieved using a 248 nm excimer laser and a phase mask to write gratings for reflectivity at around 1.5  $\mu$ m. The induced refractive index changes show enhanced temperature stability and there is no sign of any erasure up to temperatures exceeding 600 °C.

UV photosensitivity of glass materials (i.e. change in refractive index induced by exposure to optical radiation) is a fundamental property for the realisation of many optical devices. Bragg gratings in optical fibres/waveguides, obtained via exposure to a periodic light pattern on a photosensitive core, have attracted much attention since their first demonstration because of their many applications such as sensors, dispersion compensators and laser mirrors [1]. On the other hand, the definition of channel waveguides using UV writing on glass substrates such as silica on silicon, are becoming increasingly important for the realisation of multifunctional integrated optical components [2]. Telecom SiO<sub>2</sub> optical fibres which have low GeO<sub>2</sub> content (~3%) show small refractive index changes when exposed to UV radiation, whereas the UV photoinduced index change in standard germanosilicate waveguides is sometimes not sufficient for writing complex structures with a high degree of integration, which would require small bending radius together with low loss. It is thus necessary to increase the photosensitivity of optical fibres/waveguides through post-fabrication methods (such as hydrogen/deuterium loading and flame brushing) and co-doping (B2O3, SnO2 and rare earths) [3]. SnO<sub>2</sub> has been used mainly as a codopant to increase the photosensitivity of germanosilicate and phosphosilicate glasses [4-6], and more recently SiO<sub>2</sub>:SnO<sub>2</sub> optical fibres have also been investigated [7]. Ref. 7 has shown that small concentrations (~0.15 mol %) of SnO<sub>2</sub> in the silica network, without the need of adding any other co-dopant, give permanent refractive index changes with a high degree of photorefractivity. A comparison with SiO2:GeO2 fibres shows that GeO<sub>2</sub> concentrations nearly two order of magnitudes higher are needed to produce the same photorefractivity under similar UV irradiation conditions (although SiO2:GeO2 fibres need shorter exposure times). Compared to other techniques, the use of SnO2 keeps absorption in the third

telecom window at 1.5 µm low, provides better temperature stability of the grating, is less time consuming and potentially cheaper. Unfortunately the incorporation of SnO<sub>2</sub> in silica presents several problems. A limit for binary glasses based on SiO<sub>2</sub>:SnO<sub>2</sub> is given by the crystallization process which takes place for SnO<sub>2</sub> concentrations of ~1% mol. Crystallization needs to be avoided during fibre/waveguide fabrication to keep the optical loss small. Another problem with the production of SnO<sub>2</sub> doped or co-doped silica fibre via MCVD (or solution doping) techniques is related to the high volatility of SnO<sub>2</sub> at the temperature required for preform collapse which prevents high SnO<sub>2</sub> concentration in the fibre. From previous works [4-6] co-dopants such as GeO2 and P2O5 can allow incorporation of SnO<sub>2</sub> up to ~1 mol%. Following reports that alkaline elements can increase the solubility of SnO<sub>2</sub> in bulk silica glass (up to 20 mol% without crystallization) [8], we propose to use Na<sub>2</sub>O to increase the concentration of SnO<sub>2</sub> in silica fibres. Bragg gratings have been written in a optical fibres with SiO<sub>2</sub>:SnO<sub>2</sub>:Na<sub>2</sub>O core and SiO<sub>2</sub> cladding which were produced using the rod-intube (RIT) technique [9]. In these preliminary experiments refractive index modulations up to 6.2·10 <sup>4</sup> have been achieved using a 248 nm excimer laser and a phase mask. Compared to gratings written in germanosilicate and borosilicate optical fibres, the gratings written in SiO2:SnO2:Na2O show much greater temperature stability (up to temperatures well above 600 °C there is no sign of any erasure). Such features are important for certain applications, e.g. optical sensors working at high temperatures and high power lasers and amplifiers where residual absorption effects or multiphoton absorption could produce significant temperature increase.

The fibre used in these experiments was produced using RIT [9] by collapsing a Suprasil cladding

onto a small cylindrical glass core rod whilst pulling. The core glass was produced by melting powders of Na<sub>2</sub>O, SiO<sub>2</sub> and SnO<sub>2</sub> in a Pt crucible at 1500  $^{\circ}$ C for 60 mins, consolidating at 1750  $^{\circ}$ C for 60 mins and casting on a Cu mold at room temperature. The molar composition of the batch powders was: [SiO<sub>2</sub>]=75%, [SnO<sub>2</sub>]=5%, [Na<sub>2</sub>O]=20%. The glass was drilled with an ultrasonic drill. The cylinder (1.5 mm diameter and 50 mm long) was cleaned in an ultrasonic bath and etched in hydrofluoric acid to reduce the surface roughness. The refractive index was measured with an Abbe refractometer and found to be  $n_{Na}$ =1.52. The cladding, a suprasil tube with outer diameter (OD) of 34 mm and internal diameter (ID) of 1.6 mm, was collapsed onto the core at 2000  $^{\circ}$ C whilst pulling the fibre (the final fibre had OD=74  $\mu$ m).

Gratings for reflectivity at ~1.55 µm were written in the fibre using a pulsed KrF excimer laser (wavelength 248 nm) working at 30 Hz and a phase mask. Pulse duration and fluence were estimated to be 20 ns and 140 mJ/cm<sup>2</sup>. The growth of the refractive index modulation is shown in fig.1 and tends to values of ~6.2·10<sup>-4</sup> after 120 minutes of exposure. Measurements at different intensities (pulse energy) have been carried out in order to understand whether the photosensitivity of this sample is driven by a one or two-photon absorption process. As fig. 2 shows the slope of the initial growth of the refractive index modulation as a function of pulse energy on a log-log scale is ~1.1, indicating that the photorefractive response of this glass is based on a one photon process [7,10].

Initial temperature stability studies have shown that the gratings written in this glass are more stable than those written in silica based fibres made of other glass compositions. Fig. 3 shows the results

of temperature stability studies made on gratings written into the SiO<sub>2</sub>:SnO<sub>2</sub>:Na<sub>2</sub>O fibre. In these measurements the reflectivity (hence refractive index modulation) of the grating was measured during step heating: the sample was heated up by steps of ~45 °C (starting from 205 °C) in 2 minutes and kept at that temperature for the subsequent 28 minutes, before increasing the temperature again by another step. It is clear from fig. 3, which shows the main reflectivity peak at several temperatures, that the main peak shifts to longer wavelengths as the temperature increases, whereas the reflectivity starts decreasing above 600 °C. Fig. 4 shows a comparison for four different fibre compositions, making evident the enhanced stability of gratings written in SiO<sub>2</sub>:SnO<sub>2</sub> and SiO<sub>2</sub>:SnO<sub>2</sub>:Na<sub>2</sub>O compared to those written in germanosilicate (SiO<sub>2</sub>:GeO<sub>2</sub>) and borogermanosilicate (SiO<sub>2</sub>:GeO<sub>2</sub>:B<sub>2</sub>O<sub>3</sub>).

In conclusion, we have reported the fabrication of optical fibres based on the ternary glass composition SiO<sub>2</sub>:SnO<sub>2</sub>:Na<sub>2</sub>O which can allow for SnO<sub>2</sub> concentrations in SiO<sub>2</sub> significantly above the crystallization limit of ~1 mol %. Compared to other dopants (e.g. GeO<sub>2</sub>, P<sub>2</sub>O<sub>5</sub>) the introduction of sodium oxide increases the solubility of SnO<sub>2</sub> as well as not causing any significant background refractive index change in the silica matrix (this last feature may be important for the realisation of photosensitive optical fibres and waveguides which are compatible with current telecom fibres). The remarkable temperature stability shown by the photoinduced refractive index modulations in SiO<sub>2</sub>:SnO<sub>2</sub>:Na<sub>2</sub>O optical fibres may be very important for the realisation of gratings and other devices which are subjected to high operating temperatures, e.g. sensors or high power lasers/amplifiers. The high temperature stability may also indicate traps with high activation energy

for the photoinduced defects [11] and this could be relevant for applications where intrinsic absorption effects (one or more photon for high light intensities) could erase the induced refractive index modulation. Further improvements will be necessary to fabricate optical fibres with levels of refractive index modulation  $>10^{-3}$ .

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### **Figures**

- 1. Growth of refractive index modulation ( $\Delta n_{mod}$ ) resulting from exposure to 248 nm KrF laser at 30 pulses/s with 140 mJ/pulse·cm<sup>2</sup>.
- 2. Initial modulation growth  $(\delta \Delta n_{mod}/\delta \Delta t)$  rate as a function of KrF laser energy per pulse. The continuous line is the best linear fit. The resulting slope is ~1.1, indicating that the process is one photon driven.
- 3. Temperature stability of gratings written in SiO<sub>2</sub>:SnO<sub>2</sub>: Na<sub>2</sub>O fibre: the main reflectivity peak is shown at several temperatures. The sample was heated up in steps of ~45 °C (starting from 205 °C) in 2 minutes and kept at that temperature for 28 minutes, before increasing the temperature again with another step.
- 4. Comparison of temperature stability of gratings written in different core glass compositions:  $SiO_2:SnO_2: Na_2O$  (SSN),  $SiO_2:SnO_2$

Fig. 1

