# Hydrogen loading in tin-phosphosilicate fibres: a method to achieve enhanced thermal stability in fibre-Bragg-grating based devices

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Abstract: Enhanced photosensitivity has been observed in hydrogen-loaded tin-phosphosilicate fibres by using a 248 nm excimer laser. Isothermal measurements up to 860 K demonstrated significant advantages over fibre gratings written in conventional H-loaded fibres. Gratings written in this fibre require a considerably shorter post-fabrication thermal annealing in order to satisfy the stability requirements of telecom components.

**Key Words:** Gratings, Thermal stability, Materials, Photosensitivity.

# 1. INTRODUCTION

Devices based on fibre Bragg gratings require high photosensitivity and extremely small tolerance on the optical properties over a long time scale in order to be assembled in reliable WDM components. High photosensitivity in standard telecom optical fibres is achieved with the so-called hydrogen (or deuterium) loading process, which can increase the photosensitivity by more than two orders of magnitude [1]. Since its first use in germanosilicate fibres, hydrogen loading has been successfully applied to many silicate fibres [2], including phosphorus, aluminium or cerium doped or codoped fibres. No improvement has been observed when the dopant is nitrogen [3]. It has to be remarked that high photosensitivity at 248 nm has been observed in phosphorusdoped silica fibres only when gratings were written in heated fibres [4]. A weak effect has been observed in unheated Hloaded phosphosilicate fibres when exposed to the KrF laser [5]. A drawback of all the gratings written in hydrogenloaded fibres is the extremely poor temperature stability. In fact in most fibres the photo-induced refractive index change starts to be erased at 100 °C in one hour. Tin doping has been shown to be a powerful method to achieve high photosensitivity [6]. Moreover, the enhanced stability of gratings written in tin-silicate fibres does not require postfabrication annealing to satisfy the requirements of telecom components [7].

In this paper we study the effect of hydrogen loading on tindoped fibres, with particular stress on the excellent thermal stability.

# 2. HYDROGEN LOADING

The tin-codoped phosphosilicate fibre used in the experiments was fabricated by modified chemical vapour deposition (MCVD). SnCl<sub>4</sub> was the precursor used to introduce Sn in the optical fibre preform. The fibre external diameter, numerical aperture and cut-off were d=110µm, NA~0.14 and  $\lambda_c$ ~1.42 µm respectively. The fibre was placed in a hydrogen-loading cell at 165 bars and 25 °C for 14 days. The photosensitivity of samples taken from the vessel was tested by writing gratings at ~1.55 um with a "Lambda Physik" line-narrowed KrF-laser (mod. EMG150) and a phase-mask. The exposure time, laser repetition rate and pulse fluence were t<sub>exp</sub>=30 minutes, RR=20 Hz and I<sub>p</sub>~300 mJ/cm<sup>2</sup> respectively. In order to let the hydrogen outdiffuse, the fibre samples were left at room temperature for a period of 7-14 days before being spliced to a fibre-coupled diode. Spectra were collected using an optical spectrum analyser. Figure 1 compares the reflectivity spectrum of the grating written both in the hydrogen-loaded and in the pristine fibres.

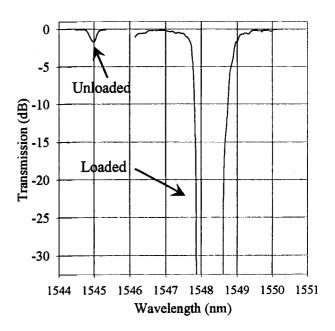


Fig. 1: Transmission spectra of gratings written in loaded and unloaded tin-phosphosilicate fibres. Grating length, exposure time, laser repetition rate and pulse fluence are L=2mm,  $t_{\rm exp}$ =30 minutes, RR=20 Hz and  $I_p$ ~0.3 J/cm² respectively. The loading process was carried out leaving the fibre in the hydrogen-loading cell at 165 bars and 25 °C for 14 days.

Similarly to what has previously been observed in fibres doped with Ge, Al and Ce, hydrogenation enhances photosensitivity to UV laser radiation in the tin-doped phosphosilicate fibre. While in the unloaded fibre the maximum reflectivity was <2dB, in the H-loaded fibre it was > 35 dB. The induced refractive index modulation ( $\Delta n_{mod}$ ) was evaluated by fitting the reflectivity curve  $R(\lambda)$  with [8]:

$$R(\lambda) = \frac{1}{1 + \frac{1}{\kappa^2 L^2 \operatorname{sinc}^2(\gamma L)}}$$
 (1)

where L is the grating length,  $\kappa=\pi\cdot\Delta n_{mod}/\lambda$  the "ac" coupling constant,  $\lambda$  the wavelength,  $\gamma=(\kappa^2-(\sigma+\delta)^2)^{1/2}$  the propagation constant inside the grating,  $\sigma=2\pi/\lambda\cdot\Delta n_{ave}$  the "dc" coupling constant,  $\delta=\beta-\pi/\Lambda$  the detuning,  $\beta$  the propagation constant in vacuum,  $\Lambda$  the grating pitch and  $\Delta n_{ave}$  the induced average refractive index change.  $\Delta n_{mod}$  was estimated to be  $\sim 1.4\cdot 10^{-3}$  and  $\sim 1.3\cdot 10^{-4}$  in the loaded and unloaded fibres.

Figure 2 compares the  $\Delta n_{\rm mod}$  growth of gratings written in a fibre loaded for 3 days and in a pristine fibre.

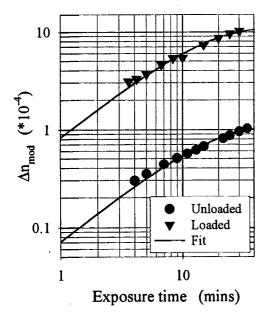


Fig. 2: Temporal evolution of  $\Delta n_{mod}$  in hydrogen-loaded (3 days at 165 bars and 25  $^{0}$ C) and pristine tin-phosphosilicate fibres.

The experimental data have been fitted with an exponential rising to the maximum:

$$\Delta n_{\text{mod}} = \Delta n_{\text{sat}} \cdot (1 - e^{-t/\tau}) \tag{2}$$

where  $\Delta n_{sat}$  is the asymptotic value of  $\Delta n_{mod}$  for long exposure times,  $\tau$  is the time constant of the refractive index growth and t the exposure time. The fit parameters are reported in table 2. While  $\Delta n_{sat}$  differs by an order of magnitude, the  $\tau$  does not show any significant modification.

	$\Delta n_{sat}$	τ (min)
Unloaded	1.10.10-4	14.7
Loaded	1.11.10-3	12.8

Tab. 1: Fit parameters for the data of fig. 2.

#### 3. THERMAL STABILITY

The grating thermal stability was studied using the so-called master curve method [9]. It has been shown that this accelerating aging technique is a more general and preferable method as compared to the parallel power-law method for gratings written in hydrogen-loaded fibres [10]. In this approach the grating decay is recorded at different temperatures and all the data are combined to give a master curve from which it is possible to predict the grating reliability in different conditions. The aging parameter  $E_{\rm d}$  (called demarcation energy) is defined as [9]:

$$E_{d}=k_{B}Tln(vt) \tag{3}$$

where  $k_{\rm B}$  is the Boltzmann's constant and T the temperature.  $\nu$  represents an attempt frequency and is determined during the data fit.

In our experiment, gratings were written as previously explained in samples of fibre hydrogen loaded for 6 days at 165 bars. Real time spectra of the gratings placed in the furnace, were recorded by means of a white light source, a coupler and an optical spectrum analyser. The time evolution of the integrated coupling coefficient  $\eta$  (defined as  $\eta{=}\Delta n_{mod}$  / $\Delta n_{mod}(0)$ , where  $\Delta n_{mod}(0)$  is the initial value of  $\Delta n_{mod}$ ) is shown in figure 3 for four different temperatures.

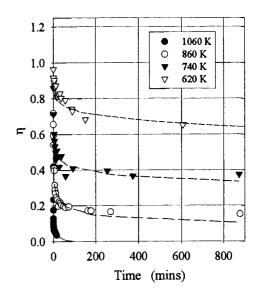


Fig. 3: Decay of the gratings written in H-loaded tinphosphosilicate fibre for different furnace temperatures. The fibres were hydrogen-loaded at 165 bars and 25  $^{\circ}$ C for 6 days. Gratings were written by exposing the fibre for 30 mins at  $I_p$ =0.3J/cm<sup>2</sup>.

It is worth stressing the enhanced thermal stability of these gratings. While gratings written in unloaded germanosilicate fibres had ~27% erased in 60 mins at 350 °C [9], in the same conditions the gratings written in the hydrogen-loaded tinphosphosilicate fibre had a change  $\eta \sim 24\%$ . The experimental data were fitted using  $v=2.5\cdot10^{10}$  min<sup>-1</sup>, considerable smaller than the value found for hydrogen-loaded germanosilicate fibres [10]. The master curve was approximated by the equation  $\eta = (1 + e^{Ed-1.65})^{-1}$  small decays. From the curve it is possible to evaluate what is the thermal decay of a grating after 25 years at 80 °C (E<sub>d</sub>=1.23eV):  $\eta$ =0.966, meaning that ~3.4% of the original grating has been wiped away. Table 2 compares the values obtained in this paper to the results published on the stability of gratings written in germanosilicate [9], in boro-germanosilicate [10] and in tinsilicate [7] fibres.

Fibre	1–η	Ref.
HL-SPS	0.034	This paper
GS	0.0782	[9]
BGS	0.138	[10]
SS	0.0014	[7]

Tab. 2: Estimation of the fraction of the initial grating erased after 25 years at 80 °C. HL-SPS, GS, BGS and SS represent hydrogen-loaded tin-phosphosilicate, germanosilicate, borogermanosilicate and tin-silicate, respectively.

As expected, the decay is considerable higher than the one observed in unloaded tin-doped fibres (<1%); nevertheless it is better than the values of degradation reported in literature for unloaded traditional fibres.

# 4. CONCLUSIONS

In summary, hydrogen loading has been shown to enhance the photosensitivity of tin-doped phosphosilicate fibres. The photo-induced refractive index change of the gratings written in hydrogen-loaded fibres was one order of magnitude stronger than the gratings written in the unloaded fibres. The thermal stability of gratings written in hydrogen-loaded tinphosphosilicate fibre has been tested with iso-thermal annealing and proved that >15 % of the initial photosensitivity survived a treatment at 860 K for 14 hours. An estimation of the grating fraction surviving 25 years at 80 °C shows that hydrogen loading of tin-codoped phosphosilicate fibres has a significant advantage over conventional fibres. Gratings written in this fibre require a less-severe post-fabrication thermal annealing in order to satisfy the stability requirements of telecom components.

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