



**HAL**  
open science

## High temperature annealing behavior of femtosecond written FBGs in Ge-doped fused silica optical fibers

Alexandre Lerner, Thomas Blanchet, Romain Cotillard, Matilde Sosa, Nicolas Roussel, Géraud Bouwmans, G. Laffont

► **To cite this version:**

Alexandre Lerner, Thomas Blanchet, Romain Cotillard, Matilde Sosa, Nicolas Roussel, et al.. High temperature annealing behavior of femtosecond written FBGs in Ge-doped fused silica optical fibers. EWOFs 2023 - European Workshop on Optical Fibre Sensors, May 2023, Mons, Belgium. pp.1264331, 10.1117/12.2678399 . cea-04298736

**HAL Id: cea-04298736**

**<https://cea.hal.science/cea-04298736>**

Submitted on 21 Nov 2023

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

# High temperature annealing behavior of femtosecond written FBGs in Ge-doped fused silica optical fibers

A. Lerner<sup>a,b</sup>, T. Blanchet<sup>a</sup>, R. Cotillard<sup>a</sup>, M. Sosa<sup>a,c</sup>, N. Roussel<sup>a</sup>, G. Bouwmans<sup>b</sup>, G. Laffont<sup>\*a</sup>

<sup>a</sup>Université Paris-Saclay, CEA, List, F-91120, Palaiseau, France; <sup>b</sup>Univ. Lille, CNRS, UMR 8523 – PhLAM – Physique des Lasers Atomes et Molécules, F-59000 Lille, France; <sup>c</sup>Institut de Chimie Moléculaire et des Matériaux d’Orsay, Université Paris Saclay, F-91405 Orsay, France

## ABSTRACT

We investigate the behavior and stability of fiber Bragg gratings written by femtosecond laser pulses in Ge-doped fused silica optical fibers, using both the phase mask and point-by-point techniques, during their annealing at 1200°C for 30 min and subsequent aging at 1000°C during 43 hours. Bragg wavelength drifts and reflected peak amplitude variations were shown to drastically differ depending on the writing scheme and thermal history. Particularly, we show that amplitude decay of point-by-point gratings at 1200°C may be easily mitigated by tuning the writing pulse energy. Future work may be pursued in order to finely unravel the high temperature mechanisms regarding the stability of fs-written fiber Bragg gratings used as temperature sensors in order to improve measurement stability and accuracy.

**Keywords:** Optical fiber sensors, Fiber Bragg Gratings, Femtosecond lasers, Fused silica, High temperature, Annealing

\*guillaume.laffont@cea.fr

## 1. INTRODUCTION

Optical fiber sensors based on Fiber Bragg Gratings (FBGs) are nowadays used in multiple research or industrial fields in order to thermally monitor specific environments where typical measurement methods as pyrometry or thermocouples may be limiting. They are particularly notable for their multiplexing capacity and immunity to electromagnetic interferences. High temperature ( $T > 600^\circ\text{C}$ ) measurements can be performed using regenerated FBGs<sup>1</sup> (RFBGs) or femtosecond laser written FBGs<sup>2</sup> (fs-FBGs). Different fiber compositions can be used in that prospect, the majority of the literature relying on telecom-grade Ge-doped fused silica optical fibers.

Most often written in as-drawn optical fibers, fs-FBGs do not require a specific treatment to become robust versus high temperature, as it is the case for RFBGs. Nonetheless, annealing is necessary in order to stabilize their optical properties (peak amplitude and wavelength) and to eliminate the influence of the residual stresses induced by the rapid cooling during the fiber manufacturing<sup>3</sup>, but also the stress fields added during the fs laser photo-inscription<sup>4</sup> itself, and finally the glass intrinsic structural relaxation<sup>5</sup>.

The two main writing schemes for fs-FBGs manufacturing are based on the phase-mask (PM) and point-by-point (PbP) techniques. The first leads to so-called type II defects in the form of porous nanoplanes of densified fused silica glass extending across the optical core and partially through the cladding<sup>6</sup>. The second leads to the formation of type III features in the form of void spherical or ellipsoidal structures surrounded by a densified silica shell, with a typical characteristic length of few hundred nanometers<sup>7</sup>.

Both these types of gratings were demonstrated to withstand temperatures up to 1000°C and above, with stability (regarding the drift in reflected peak amplitude and Bragg wavelength ( $\lambda_B$ )) highly dependent on the inscription technique and writing parameters<sup>8,9</sup>. Multiple authors recently carried out extensive reviews of the thermal stability of these types of gratings and their implication for temperature measurement<sup>5,10,11</sup>.

The fs-written structures stability as well as the glass structural and stress relaxation are strongly affected by the visco-elastic properties of the glass. To stabilize their properties, stress relaxation is commonly performed by annealing the gratings at sufficiently elevated temperatures. While the typically reported heat treatment temperatures for fs-FBGs are in the range of 1000–1070°C<sup>11</sup>, it appears that the optimal annealing point ( $T_a$ , corresponding to a viscosity  $\eta < 10^{12}$  Pa.s) of the fused silica glass is around 1100°C, as was measured at  $\sim 1114^\circ\text{C}$  by Shao *et al.*<sup>12</sup> using RFBGs in Ge-doped fused silica fiber.

Pre-annealing the fiber at 1100°C before writing a PM fs-FBG was shown by Li *et al.*<sup>13</sup> to drastically reduce the wavelength drift once aged at 1200°C. Nonetheless, annealing the fiber prior to inscription leads to an increased brittleness of the glass, which is not compatible with some writing setups, as on-line inscription in draw-towers for example.

Exceeding the 1000°C limit can potentially lead to noticeable collapse or erasure of the written defect, by allowing glass flow through the decrease in viscosity<sup>5</sup>. This phenomenon was observed by Shi *et al.*<sup>14</sup> on PbP fs-FBGs written in different fiber compositions submitted to isothermal annealing over the range 950–1150°C. They established an empirical relationship between the FBGs coupling strength decay and the glass viscosity, demonstrating the viscosity-driven collapse of the void structures composing the gratings.

The underlying mechanisms at stake at temperatures around the annealing point – in both the fused silica matrix and the modified volume – shall be understood in order to correctly anneal these sensors for harsh environments monitoring, or develop new fiber compositions and inscription methods to improve the measurement stability at high temperatures.

In that prospect, we manufactured arrays of fs-FBGs in Ge-doped silica optical fibers using both the PM and PbP technique, and studied the influence of an annealing at 1200°C ( $> T_a$ ) for approximately 30 min on their subsequent stability at 1000°C during 43 hours.

## 2. EXPERIMENTAL PROCEDURE

### 2.1 Fiber Bragg Gratings manufacturing and characteristics

Three arrays of 3-mm long second-order FBGs were manufactured with each of the writing techniques (PM and PbP) in mechanically stripped SMF-28 optical fiber. The PM setup is comprised of a Ti:Sapphire fs laser (800 nm,  $t_{\text{pulse}} = 35$  fs) which pulses are focused in the core of the fiber through a phase mask by a cylindrical lens. A piezoelectric actuator allows scanning the fiber cross-section. One FBG centered at ~1552 nm with a reflectivity within range of 80–90 % was inscribed for each PM array.

The PbP setup is comprised of a frequency doubled Yb fs laser (515 nm,  $t_{\text{pulse}} = 160$  fs) focalized into the core of the fiber using an immersion microscope objective. A three-axis translation stages setup allows scanning the core and writing the structure. Two FBGs were written in each PbP array: one at a low pulse energy (LE) resulting in a reflectivity of 25–35 % centered at ~1520 nm, and another written at higher pulse energy (HE) with a reflectivity of ~85 % centered at ~1550 nm.

### 2.2 Thermal testing

Thermal testing was performed by positioning the optical fibers inside a tubular furnace (TF1 16-60-300, Carbolite). Proper thermal mapping ensured that the FBGs were in a  $\pm 2.5^\circ\text{C}$  stability area of the heated tube length. The FBGs spectral response were monitored using a si255 (Luna Innovations) optical sensing instrument.

The furnace was first heated from Room Temperature (RT) up to 1200°C in order to maintain certain gratings at this setpoint for 30 min, then set to 1000°C for 43 h. In order to assess the influence of the high temperature annealing on the subsequent behavior at 1000°C, the FBG arrays were divided in three batches as follows. Arrays PM1 and PbP1 were positioned in the furnace during the 1200°C step for ~30 min, then rapidly cooled down to ~200°C to quench them, and were subsequently repositioned in the furnace once stabilized at 1000°C. PM2 and PbP2 were placed in the furnace at RT at the beginning of the experiment, and underwent the full thermal cycle. To control the behavior of as-inscribed FBGs – *i.e.* no thermal exposure since fiber drawing – arrays PM3 and PbP3 were introduced in the furnace at the beginning of the 1000°C step.

## 3. RESULTS AND DISCUSSION

### 3.1 Annealing at 1200°C

Figure 1 presents the Bragg Wavelength Shift (BWS) and reflected peak power amplitude variation ( $\Delta A_{\text{mpi}}$ ) for the studied FBGs, with the reference point taken at RT before the experiment. During the annealing of ~30 min at 1200°C, the PM and PbP FBGs show drastically different behaviors regarding the evolution of their amplitude. The amplitude decay of both PM FBGs (Figure 1a) show a similar dynamic. The PM grating that underwent temperature rise from RT to 1200°C (PM2) sees its amplitude plateauing after approximately 23 min at 1200°C, cumulating a total attenuation of –24 dB.

PbP FBGs also show noticeable amplitude decays at 1200°C (Figure 1b). Their evolution is consistent with the results presented by Shi *et al.*<sup>14</sup>, but decay time comparison would be challenging given that the authors interrogated the grating in transmission contrary to the present results. Contrary to PM FBGs, the decay dynamics for PbP FBGs are dependent on

the glass thermal history. PbP2 FBGs, that underwent gradual heating up to 1200°C (in about 4 h) show higher amplitude decay rate than PbP1 FBGs directly positioned in the 1200°C environment from RT. It appears that writing energy (LE/HE) also has a strong influence on the reflected amplitude decay rate of such PbP FBGs. After 30 min at the annealing temperature, the PbP2 grating written at “low pulse energy” underwent an attenuation of about -9.7 dB, whereas the “high pulse energy” grating shows a -2.3 dB reduction in amplitude only. We note that PbP2 LE completely erased after such exposure, and that PbP2 HE is severely attenuated (Figure 1b). This phenomenon may be expected since decrease in coupling strength appears to be related to the micro-voids collapse<sup>14</sup>, and because higher writing pulse energies lead to larger dimensions of the inscribed type III defects. Hence, tuning the inscription energy allows adjusting the high temperature behavior of these features, the counterpart being for example increased insertion losses from scattering.

Fs-FBGs are obviously not designed to operate for extended durations at temperature above the fused silica intrinsic annealing point  $T_a$  (about 1100°C), but it appears that under specific configurations of writing energy or inscription technique, they may be designed to withstand these levels for a few hours. These timespans may be sufficient to perform stress relaxation or grating stabilization.

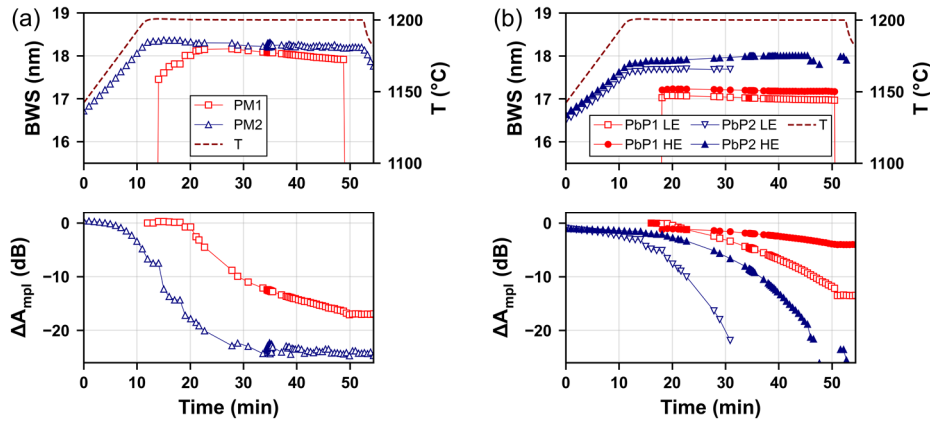


Figure 1: BWS and amplitude variations of (a) PM fs-FBGs and (b) PbP FBGs during the 1200°C annealing. Colors correspond to identical thermal history. Reference values for BWS and  $\Delta A_{mpl}$  calculation were extracted at RT before the experiment.

### 3.2 Aging at 1000°C

As previously described, two FBGs arrays were rapidly cooled down from 1200°C to ~200°C, quenching the fused silica glass. Once brought back to 1000°C, these three gratings (PM1, PbP1 LE, PbP1 HE) show an identical Bragg wavelength shift (BWS) behavior, independently of the writing technique and parameters (Figure 2). This evolution follows a stretched exponential function, comparable to what was observed by Ryu and Tomozawa<sup>15</sup> when studying the fictive temperature evolution of fused silica fibers quenched from 1200°C and subsequently annealed at 1000°C. Quenching the fiber hence fixed an identical material configuration – regarding stress and fictive temperature – for both samples. This means that the 1200°C annealing completely removed the residual stresses induced by the fiber drawing and the diverse fs laser exposures. We also note that monitoring the Bragg wavelength may be a way to monitor the glass fictive temperature evolution in specific experimental configurations.

As previously shown on Figure 1, FBGs that underwent the full thermal cycle present a severe amplitude reduction at 1200°C. This induces noise in peak detection during the 1000°C aging in Figure 2 (PM2/PbP2 HE). We observe that their relaxation reaches lower BWS values for both PM and PbP FBGs compared to as-inscribed and quenched gratings, but further studies are necessary to conclude on their long-term behavior after such harsh thermal exposure.

As-inscribed FBGs (PM3/PbP3) positioned directly in the furnace at 1000°C from RT show various Bragg wavelength drift behaviors. In the case of PbP3 gratings, it tends to stabilize (here for  $t \geq 20$  h) to a slope of about +1.1 pm/h (~0.06°C/h), whereas for PM3 a drift of about +7.5 pm/h (~0.4°C/h) is observed. As a comparison, quenched FBGs previously discussed show a wavelength drift of about +0.4 pm/h (~0.02°C/h). The diversity of drift values may indicate that, additionally to fiber drawing residual stress relaxation – supposedly identical for all gratings, inscribed on fiber sections from the same spool – stress relaxation inherent to the fs-written structure exists. The PM technique modifies the fused silica on a larger volume than PbP-induced features, which would be consistent with the observed behavior in PM3, leading to potential improved stability for as-inscribed fs-written PbP FBGs compared to PM gratings in the present study.

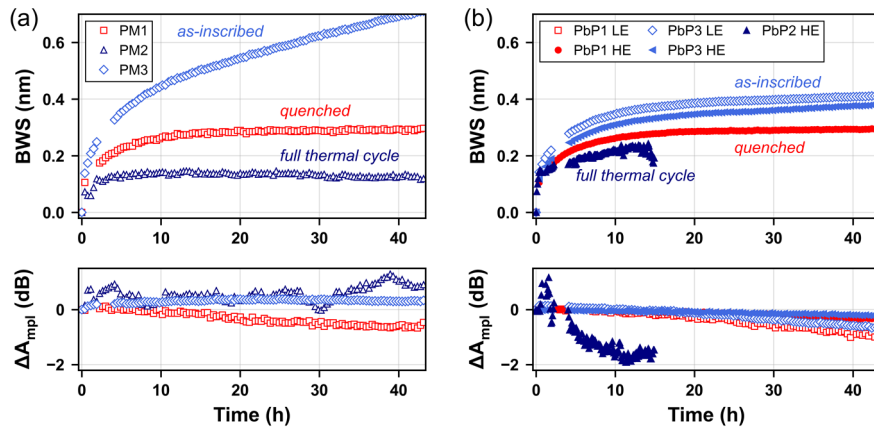


Figure 2: BWS and amplitude variations of (a) PM and (b) PbP FBGs during the aging at 1000°C. Colors correspond to identical thermal history. Reference values for BWS and  $\Delta A_{mpl}$  were extracted once the furnace stabilized at the setpoint.

## CONCLUSIONS

Annealing fs-FBGs at 1200°C showed significantly different behaviors depending on the writing technique. Dependencies on the thermal history and inscription parameters were shown for PbP FBGs and will be further studied, whereas the amplitude decay dynamics of PM FBGs was shown to depend on the temperature only. As-inscribed PbP FBGs show lower Bragg wavelength drift compared to PM FBGs, which may be explained by the difference in fs laser-modified volume. Rapid cooling of the fused silica from 1200°C allowed to demonstrate the necessity of heat treatment over the fused silica annealing point in order to completely relax residual stresses induced during the fiber drawing and fs-FBGs inscription. Further studies will be carried out to quantify the impact of heat treatment strategy and writing parameters on high temperature behavior of these FBGs in order to use them as temperature probes in harsh environments.

## REFERENCES

- [1] Laffont, G., Cotillard, R., Roussel, N., Desmarchelier, R. and Rougeault, S., "Temperature Resistant Fiber Bragg Gratings for On-Line and Structural Health Monitoring of the Next-Generation of Nuclear Reactors," 6, *Sensors* **18**(6), 1791 (2018).
- [2] Chanet, N., Corre, Y., Cotillard, R., Gaspar, J., Laffont, G., Pocheau, C., Caulier, G., Dechelle, C., De Gentile, B., Destouches, C., Dubus, L., Gally, F., Hernandez, C., Missirlan, M., Richou, M., Roussel, N. and Santraine, B., "Design and integration of femtosecond Fiber Bragg gratings temperature probes inside actively cooled ITER-like plasma-facing components," *Fusion Engineering and Design* **166**, 112376 (2021).
- [3] Paek, U. C. and Kurkjian, C. R., "Calculation of Cooling Rate and Induced Stresses in Drawing of Optical Fibers," *J American Ceramic Society* **58**(7-8), 330-335 (1975).
- [4] Bhardwaj, V. R., Corkum, P. B., Rayner, D. M., Hnatovsky, C., Simova, E. and Taylor, R. S., "Stress in femtosecond-laser-written waveguides in fused silica," *Opt. Lett.*, **OL 29**(12), 1312-1314 (2004).
- [5] Cavillon, M., Lancry, M., Poulmellec, B., Wang, Y., Canning, J., Cook, K., Hawkins, T., Dragic, P. and Ballato, J., "Overview of high temperature fibre Bragg gratings and potential improvement using highly doped aluminosilicate glass optical fibres," 4, *J. Phys. Photonics* **1**(4), 042001 (2019).
- [6] Hnatovsky, C., Silva, K. D., Abdurkerim, N., Walker, R. B., Ding, H. and Mihailov, S. J., "Nanoscale morphology and thermal properties of low insertion loss fiber Bragg gratings produced using the phase mask technique and a single femtosecond laser pulse," *Opt. Express*, **OE 30**(26), 47361-47374 (2022).
- [7] Marshall, G. D., Williams, R. J., Jovanovic, N., Steel, M. J. and Withford, M. J., "Point-by-point written fiber-Bragg gratings and their application in complex grating designs," 19, *Opt. Express*, **OE 18**(19), 19844-19859 (2010).
- [8] Martinez, A., Khrushchev, I. Y. and Bennion, I., "Thermal properties of fibre Bragg gratings inscribed point-by-point by infrared femtosecond laser," 4, *Electronics Letters* **41**(4), 176-178 (2005).
- [9] Grobncic, D., Smelser, C. W., Mihailov, S. J. and Walker, R. B., "Long-term thermal stability tests at 1000 °C of silica fibre Bragg gratings made with ultrafast laser radiation," 5, *Meas. Sci. Technol.* **17**(5), 1009-1013 (2006).
- [10] Dedyulin, S., Timakova, E., Grobncic, D., Hnatovsky, C., Todd, A. D. W. and Mihailov, S. J., "Accurate Measurements of a Wavelength Drift in High-Temperature Silica-Fiber Bragg Gratings," 1, *Metrology* **1**(1), 1-16 (2021).
- [11] Grobncic, D., Hnatovsky, C., Dedyulin, S., Walker, R. B., Ding, H. and Mihailov, S. J., "Fiber Bragg Grating Wavelength Drift in Long-Term High Temperature Annealing," 4, *Sensors* **21**(4), 1454 (2021).
- [12] Shao, L.-Y., Canning, J., Wang, T., Cook, K. and Tam, H.-Y., "Viscosity of silica optical fibres characterized using regenerated gratings," *Acta Materialia* **61**(16), 6071-6081 (2013).
- [13] Li, Y., Yang, M., Wang, D. N., Lu, J., Sun, T. and Grattan, K. T. V., "Fiber Bragg gratings with enhanced thermal stability by residual stress relaxation," 22, *Opt. Express*, **OE 17**(22), 19785-19790 (2009).
- [14] Shi, J., Xiao, S., Yu, Y., Sun, W., Luo, T., Chen, T., Han, F. and Feng, X., "Correlation between glass viscosity and the high-temperature lifetime of silica fiber Bragg gratings directly written by a femtosecond laser," *OSA Continuum*, **OSAC 3**(12), 3468-3481 (2020).
- [15] Ryu, S.-R. and Tomozawa, M., "Structural Relaxation Time of Bulk and Fiber Silica Glass as a Function of Fictive Temperature and Holding Temperature," *Journal of the American Ceramic Society* **89**(1), 81-88 (2006).