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# Investigation of High Temperature Measurements Repeatability with Sapphire Fiber Bragg Gratings

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**Abstract:** Thermal cycling tests of a sapphire Fiber Bragg Grating inscribed in a sapphire fiber with a phase mask and a femtosecond laser are performed up to 1150°C in order to assess the repeatability of high temperature measurements.

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## 1. Introduction

Optical Fiber Sensors (OFSs) are promising alternatives to conventional (electric) sensors for use in harsh environments, because of their small size and weight (low intrusiveness), passiveness and possibility of multiplexing that simplifies deployment. They may be classified according to whether they are multiplexed or distributed. Distributed OFSs are based on light scattering mechanisms within the guide such as Rayleigh, Brillouin or Raman [1]. They cannot be used for temperatures higher than 850°C [2], mainly because of Blackbody Radiation (BR) that superimposes over the useful scattered signal and eventually degrades the Signal-to-Noise Ratio (SNR). Raman-based distributed temperature sensing also suffers from dramatic drop in sensitivity at elevated temperatures.

Conversely, point OFSs (either multiplexed or not) such as Fiber Bragg Gratings (FBGs) [3], Long-Period Gratings (LPGs) [4], Fabry-Perot Interferometers (F-PIs) [5] or BR sensors may operate up to higher temperatures depending on fiber material. Silica-based OFSs are limited to 1300°C by a suitable choice of dopants [6]. Higher temperatures may be reached using single-crystal Sapphire ( $\text{Al}_2\text{O}_3$ ) fibers since the melting point of sapphire is about 2040°C [7]. For instance, silica-based FP-I operate up to 1000°C [5] while sapphire-based FP-I may be used at temperatures as high as 1900°C [7]. The use of LPGs is actually limited by the necessity to operate in transmission and the large Full Width at Half-Maximum (FWHM) that hampers spectral multiplexing. Finally, it is worthwhile noticing that silica-based BR sensors cannot operate between Room Temperature (RT) and 500°C [8] as the BR is in the infrared region where silica is not transparent.

Contrary to previous sensor types, FBG sensors may be spectrally multiplexed along a single guide. The ability of FBG sensors to withstand high temperature is determined by the written process, according to Canning's classification [3]. The maximal temperature reached is 1295°C with a regenerated FBG inscribed in a co-doped B/Ge silica-based fiber [9]. Type II or III gratings are inscribed with fs-laser and interferometric methods – for type II – and Point-by-Point (PbP) – type II and III – in any fiber composition and they can withstand temperature up to 1000°C [10], [11], [12]. Over such a large temperature span, the temperature-induced Bragg Wavelength Shift (BWS) is often adjusted using higher order polynomial relationships.

The prospect of using FBG sensors at higher temperatures motivated a growing research activity on Sapphire FBG (S-FBG) over the past 20 years. S-FBG can be inscribed using femtosecond laser with an interferometric method such as phase mask [13], Talbot interferometer [14] or by focalizing the laser beam directly inside the fiber core (PbP) [15] or Line-by-Line (LbL) [16] techniques.

T. Habisreuther *et al.* [14] demonstrated the ability for the S-FBG to withstand temperature as high as 1900°C. The gratings were inscribed with the Talbot interferometer technique. Moreover, X. Xu *et al.* [16] wrote S-FBGs with the LbL technique and monitored the grating behavior on a complete oven cycle up to 1612°C. They observed different temperature coefficients between heating and cooldown phases.

M. Busch *et al.* [17] inscribed S-FBGs with a fs-laser centered around 800 nm and a phase mask. Then, the authors performed 9 thermal cycles during 20 min. They observed hysteresis in the Bragg shifts vs temperature between 1200°C and 1745°C. The accuracy in Bragg shift monitoring was about  $\pm 0.5$  nm at 1200°C, which translates in a temperature uncertainty of  $\pm 15^\circ\text{C}$ .

These previous results show that the repeatability for high temperature measurements is of great matter of concern with S-FBG sensors, with the aim to provide satisfactory and useful temperature monitoring for industrial applications.

In this work, we have inscribed S-FBGs in a 100- $\mu\text{m}$  diameter sapphire fiber using the phase mask technique and a fs-laser and we performed thermal cycling tests in order to evaluate the repeatability of the temperature measurements up to 1150°C.

## 2. Experimental Setups

### 2.1 S-FBG photo-inscription

The gratings were inscribed with a Titanium-Sapphire (Ti:Sa) fs-laser (Coherent, Astrella) which delivers 35 fs pulses around 800 nm at a frequency of 1 kHz with an energy per pulse set at 650 mJ for the grating inscription. The beam was focalized inside the uncoated 100- $\mu\text{m}$  diameter sapphire fiber using a cylindrical lens with a focal length of 18 mm. The focal line was parallel to the fiber axis and a piezo actuator moved the lens with a sinusoidal movement, perpendicularly to the fiber axis at a frequency of 0.2 Hz, over a span of 20  $\mu\text{m}$ . The phase mask was placed between the lens and the fiber and at about 1 mm from it. The 20 cm long sapphire optical fiber (Micromaterials, USA) was packaged by IDIL (Lannion, France). In order to reduce back reflections, one end was connected with a FC/APC connector and the other end was polished with an angle of 8°.

The S-FBG was interrogated in reflection with an ASE source (density power of 0.1 mW/nm from 1520 nm to 1620 nm), a 105- $\mu\text{m}$  core multimode 2x1 coupler and the reflected signal was analyzed with an Optical Spectrum Analyzer (OSA, Yokogawa, AQ6376D) with a spectral resolution of 20 pm. The measurement time was about at 90 s.

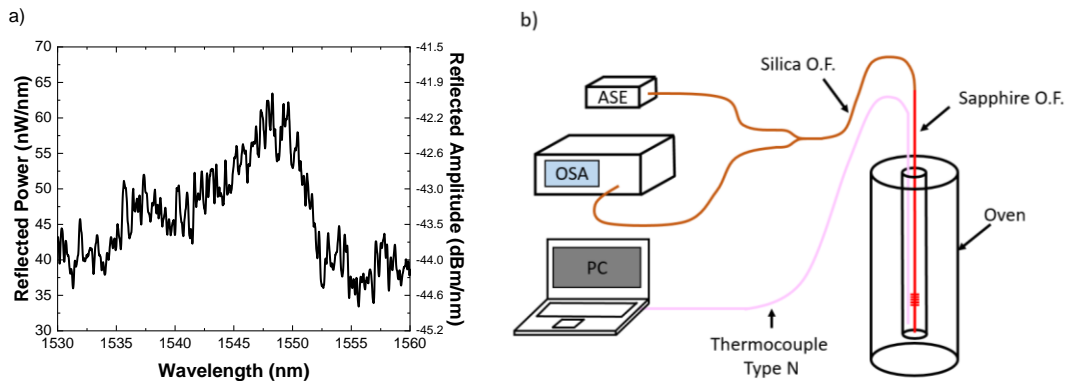


Figure 1. a) Typical spectrum of S-FBG recorded at RT, b) Setup for thermal annealing tests

S-FBG spectra are wider (10 nm at mid-height, fig. 1.a) than those of silica-based FBG and present a triangular shape due to the multimodal nature of the sapphire fiber (it can be seen as the distribution of Bragg gratings attributed to different effective indexes). The maximum of the Bragg peak is located at about 1547 nm. The spectra show ripples stemming from either the light source, the coupler and the multimodal behavior of the fiber (modal content). Ripples were removed by smoothing the spectra envelope, which was realized with a moving average over 500 pm. Finally, we estimated the Bragg wavelengths with the help of the -1 dB bandwidth method, as described by Tosi *et al.* [18].

### 2.2 Thermal tests

The experimental setup is shown in Fig. 1.b. The S-FBG was plunged inside a vertical oven (Jofra, CTC-1200A from Ametek), whereas the fiber is only subjected to its own mass. The fiber was heated up to a temperature of 1150°C with a heating slope of about 40°C/min, the temperature was held at 1150°C during 40 min and cooldown lasted several hours. We repeated this cycle 10 times. During the two-week experiment, two type N thermocouples were used to record the oven temperature near the S-FBG.

## 3. Results

### 3.1 Sensor calibration

The grating calibration was performed on the last cycle, during the cooldown phase since the temperature inside the oven is not homogenous during the heating phase. A 3<sup>rd</sup> order polynomial calibration equation is adjusted (red curve on Fig. 2). For comparison, the average value of two thermocouple measurements were also shown (black curve). A

maximal uncertainty of more than 30°C is observed at a temperature of about 1150°C, *i.e.* when cooldown starts. This is due to an inhomogeneous temperature distribution within the oven that leads to a large difference between both S-FBG and thermocouple indications. For all other temperatures, the uncertainty was below 15°C.

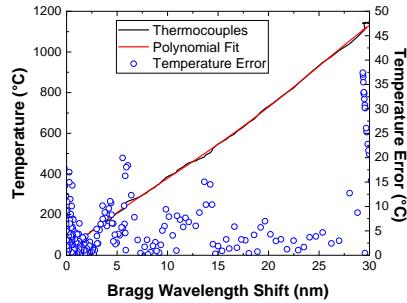


Figure 2. Calibration curve of the S-FBG, thermocouples (black), polynomial fit (red) and absolute error (blue circle – Y-right axis)

### 3.2 Thermal Cycling

The S-FBGs were plunged stress free inside the oven. For all thermal cycles, the uncertainty in absolute temperature is less than 50°C, excepted during the transient heating phase ( $\sim 40^\circ\text{C}/\text{min}$ ) because the temperature distribution is not homogeneous (Fig. 3). For the first 4 cycles, the grating wavelength is not stable and we note an uncertainty as large as 50°C. Moreover, for the first cycles a BWS of 30°C ( $\sim 0.6$  nm) is observed between the beginning and the end of the annealing phase (15 h) witnessing for a hysteresis effect.

For the second and third cycles, the Bragg wavelength is stable at RT, showing an uncertainty less than 25°C. Between the 4<sup>th</sup> and 5<sup>th</sup> cycles (72 h to 96 h) as well as during between the 5<sup>th</sup> and 6<sup>th</sup> (96 h to 168 h) the Bragg peak continues to shift even at RT, with an uncertainty varying from 10°C to 50°C. For the cycles 6 to 10, the Bragg wavelength is almost constant with an average uncertainty less than 20°C. During the two-week long period, the grating amplitude decreased by -1 dB/nm (about 10 nW/nm).

As for silica based-FBG, a thermal annealing at temperature higher than the operating temperature must be performed to stabilize the grating [19]. However, the thermal cycle was not efficient enough to stabilize the S-FBG and at least five thermal cycles up to 1150°C during 40 min must be performed, since the grating is stable for the next five cycles. Moreover, even at a temperature of 1150°C the fiber surface itself degrades due to the contact with the atmosphere, leading to loss due to the light scattering at sapphire-air interface. This perturbation has been observed previously [18].

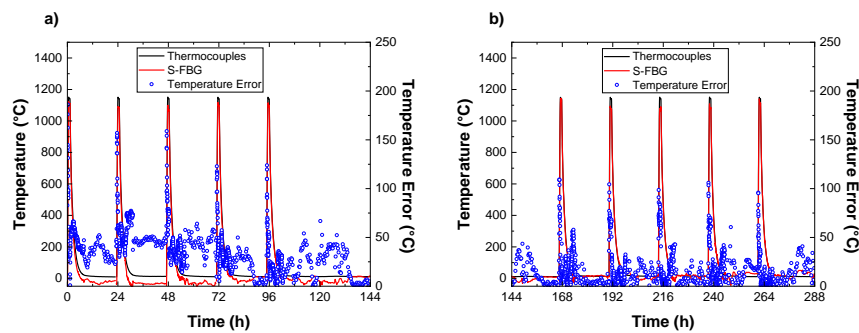


Figure 3. Temperature measured by the thermocouples (black) and S-FBG (red) and the absolute uncertainty (blue circle) between the thermocouples and the S-FBG for the first five cycles (a) and the last five cycles (b).

## 4. Conclusion

We have inscribed FBGs inside sapphire fibers with a Ti-Sa fs-laser emitting around 800 nm and a phase mask. We performed thermal cycling tests up to 1150°C to assess for the repeatability of the temperature measurements. This test confirms the existence of a hysteresis effect in the Bragg-to-temperature response of the S-FBG that translates in a temperature shift of 50°C at RT. We show that a thermal annealing process is required to remove this hysteretic behavior and to achieve repeatable high temperature measurements. A minimum of 5 cycles is necessary. At the end

of the thermal cycling process, the uncertainty in temperature remains below 20°C. This uncertainty in temperature is partly due to statistics (depends on OSA parameters and recording time) and to spectral instabilities in ASE spectrum and modal content of the sapphire fiber that is not controlled. Another important consequence is the degradation of the Bragg spectrum due to alteration of the sapphire surface by air contamination. S-FBGs will be sheltered within alumina capillaries for high temperature measurements.

Future developments involve tests up to 1500°C, with thermal cycling and long-term aging (few days) in order to address high-temperature applications such as high-temperature processes, chemical reactor and engine monitoring.

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