A Raman Spectroscopic Study of FBG Regeneration

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Abstract: Fibre Bragg grating regeneration involves, at its simplest, the annealing and resurrection of a seed FBG through thermal annealing. Micro-Raman spectroscopy provides evidence that inscribing the seed structure has changed the thermal history of the glass so that whilst the annealing is identical in all areas, the local glass relaxation is different in the UV irradiated regions. Notably, there is a significant decrease of the D₁ and D₂ defects bands within the core and the inner cladding of regenerated FBG revealing the complex nature of this two- or three materials system.

OCIS codes: (060.3738) Fibre Bragg gratings, photosensitivity; (060.2370) Fibre optics sensors; (160.6030) Silica

1. Introduction

Improving fibre Bragg grating (FBG) technologies to operate in harsh and extreme environments is a rapidly growing field of research. A new generation of devices that operate in these extreme environments (particularly those above 800 °C), including with additional challenges such as radiation and intense optical fields (e.g. high power lasers), must be capable of withstanding gradual annealing and degradation, or aging, over a suitable period of time while preserving their intrinsic advantages (multiplexing capabilities, electromagnetic immunity, low intrusiveness, mechanical reliability) over other, conventional technologies such as thermocouples. Much of the answer to improving these technologies lies with optimizing inherent glass structure and properties, particularly viscous flow, chemical and structural migration and stress relaxation and fundamentally recognising the opportunities multi-materiel systems provide. Overall, FBG regeneration is one of the two main current approaches (the other being femtosecond laser writing) that can enable photonic technologies to operate in such harsh environments at elevated temperatures. In its simplest recognizable expression, regeneration involves the annealing and resurrection of a structure patterned into a glass through optical or thermal treatment. This pattern, or seed structure, has changed the thermal or optical history of the glass so that whilst the annealing is identical in all areas, the local response is different, equivalent to a local variation in quenching. FBG regeneration has been interpreted (often without recognising relaxation and the multi-material system being used) in different ways including dopant chemical migration (fluorine evolved into oxygen in order to explain performances above 900 °C for example), crystallization and glass structural changes. One local approach to study structural relaxation is to spectroscopically follow structural changes that are associated with a density change. In this paper, we used Raman microspectroscopy to investigate the mechanisms of FBG thermal regeneration in B-codoped germanosilicate GF1 optical fibres.

2. Experimental details

Bragg gratings were inscribed in B-codoped germanosilicate GF1 optical fibres. The composition of these fibres is summarised in the Table below. Fibre Bragg gratings were produced by direct writing through a 10 mm long optical phase mask using an ArF laser ($\lambda = 193$ nm; pulse fluence: $f_{pulse} = 95$ mJ/cm²; cumulative fluence $f_{cum} = 113$ J/cm²; RR = 30 Hz; pulse duration $\tau_w = 15$ ns). Before grating writing, the fibres were hydrogen (H₂) loaded ($T = 80^{\circ}$ C, P =180 bar, t = 4 days), avoiding type In formation. To perform the regeneration process some gratings were placed in a computer-controlled oven. The temperature was ramped to $T = 850 \,^{\circ}$ C over t = 60 min before dwelling at this temperature for t = 40 min; during this dwell time the grating decays and regenerates. For the sake of comparison, a pristine fibre was also placed into the furnace acting as a reference "thermally-treated fibre". Subsequently, the cross-section of the samples were analyzed at the cleaved end facet of the sample using micro-Raman spectroscopy (Renishaw, inVia spectrometer) with an excitation wavelength of $\lambda = 532$ nm and a x100 objective (NA = 0.9)

Fibre	Outer cladding (φ ~ 125 μm)	Inner Cladding (φ ~ 40 μm)	Core (φ ~ 9 μm)
GF1	SiO ₂ only	$[P_2O_5] \simeq 11 \text{ mol\%}, [F] \simeq (3 - 4) \text{ mol\%}$	[GeO ₂] ~ 33 mol%, [B ₂ O ₃] ~ 20 mol%

3. Results and discussion

Glass structural relaxation often leads to noticeable changes in fictive temperature, T_f [ref] and thus glass density but also stress relaxation (or increase in some cases). By monitoring Raman D₁ and D₂ bands and their ratio,

information's about densification/expansion or stress relaxation in glass can be tentatively obtained in a number of specific cases. These two peaks are associated with breathing modes of four (D₁) and three (D₂) planar silica-oxygen rings and their numbers correspond to some stored energy per volume unit that increases with the glass density when there is no other structure modification. All the Raman spectra were normalized to the main band intensity (at $v = 429 \text{ cm}^{-1}$), the D₁ ($v \sim 495 \text{ cm}^{-1}$) and D₂ ($v \sim 605 \text{ cm}^{-1}$) peak heights were measured after Gaussian decomposition. The resulting profile of the D₂ amplitude and D₁/D₂ ratio along the fibre cross section are shown in Figure 1.



Fig. 1. Raman spectra of the GF1 fibre core: pristine, seed FBG and regenerated FBG. Thermally-treated GF1 fibre without any irradiation is also shown for sake of comparison.

As Figure 1(Left) shows minor changes for D₂ peak at $\nu \sim 605$ cm⁻¹ were observed in the core when comparing the pristine fiber and the thermally treated one. Having a look on the D₁/D₂ amplitude ratio shown in Fig. 1(Right), we can see a spectacular increase of the ratio revealing a structural relaxation characterized by a decrease in glass density (expansion). Note there is also a significant decrease of D₂ within the inner-cladding after thermal treatment that is consistent with compressive stress relaxation. However whereas the relaxation of the initial tensile stress in the core should leads to a decrease of D₁/D₂ ratio, we rather observe the signature of a glass expansion. In GF1 fiber, both core and inner-cladding have softening dopants (boron and phosphorus) that lower T_g significantly relative to the pure silica outer-cladding, which does not soften much. Further, the thermal expansion coefficient of the doped core is an order of magnitude larger than the inner cladding suggesting expansion is proportionally larger in the core. Now comparing the seed UV FBG and the pristine fiber, we can see an increase for both D peaks i.e. D₂ at $\nu \sim 605$ cm⁻¹ in Fig. 1(left) and D₁ (not shown here) in the fiber core and a slight decrease of the D₁/D₂ ratio, which reflects the expected UV-induced densification. In contrast we observe the signature of a glass expansion within the inner cladding of the seed UV FBG.

After regeneration both D peaks are observed to strongly decrease in height (and in area as well) when compared to the seed UV FBG. Gaussian decomposition also reveals an increase of the D_1/D_2 ratio from 2 to 3 in the fiber core after the regeneration. In addition there is a slight change of the shape of the main band around 390cm⁻¹ (not shown here) that indicates an increase of the 6-membered rings. Significantly high changes are also observed within inner-cladding even compared to the thermally-treated fiber. In addition, the Raman profile data seems to indicate a substantive expansion of the inner-cladding dimensions.

4. Conclusion

For GF1 fibre an overall relaxation at the fibre core/cladding interface is expected, driven by the larger tensile stress from the outer pure silica cladding on the inner-cladding and core. Both the inner-cladding and the core materials appear to expand as revealed by a decrease of glass density local indicators, namely the D_1 and D_2 bands but also by the increase of the D_1/D_2 ratio after regeneration and the blue-shift of the main band. Thus it seems the regeneration is largely driven by the dominant tensile stresses.

Additional evidence for a net reduction of density can be seen as a blue shift in the final regenerated grating Bragg wavelength at room temperature from the starting seed Bragg wavelength provided no load is present on the fibre, where this can lead to period elongation with glass softening. As has been proposed originally [ref37 S. Bandyopadhyay, J. Canning, M. Stevenson, K. Cook, "Ultra-high temperature regenerated gratings in boron codoped germanosilicate optical fibre using 193nm", Opt. Lett., **33** (16), 1917-1919, (2008)], the evidence presented here supports the

notion that regenerated grating occurs at the interface; recent etching works also indicate strong changes at the interface [ref38].

@. Acknowldgements: raman microfacility USYd

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