

Study of spectral and annealing properties of fiber Bragg gratings written in H₂-free and H₂-loaded fibers by use of femtosecond laser pulses

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Abstract: The spectral and annealing properties of a series of fiber Bragg gratings (FBGs) written in both H₂-loaded and H₂-free fibers by use of 800nm femtosecond laser pulse irradiation and created through a phase mask, have been investigated. It is found that type II FBGs inscribed in H₂-loaded fibers exhibit superior spectral quality when compared with those written in H₂-free fibers. Isochronal annealing tests shows that type II FBGs written in H₂-free fibers have the highest thermal stability, followed (in order of stability) by H₂-loaded type II, H₂-free type I and then H₂-loaded type I FBGs. The thermal stability of the H₂-loaded type II FBGs can effectively be increased by using a high temperature pre-annealing treatment. After the treatment, type II FBGs written into both H₂-free and H₂-loaded fibers can sustain long-term annealing (for more than 12 hours) at temperatures of more than 1000 °C while their high reflectivities can still be maintained. This demonstrates the real potential of the FBGs developed and investigated in this work to be used as the ideal sensing elements for a series of high temperature measurement applications.

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1. Introduction

Fiber Bragg gratings (FBGs) have many applications in optical fiber communication systems and also in optical fiber sensors [1-5]. They are usually fabricated by use of UV laser irradiation which is based on a single-quantum photochemical mechanism and the refractive index modulation thus produced critically depends on the intrinsic photosensitivity of the fiber materials. The gratings fabricated in this way often exhibit poor stability at high temperatures, of more than 500 °C. In recent years, optical devices fabricated by use of femtosecond laser pulse irradiation have attracted considerable attention and scientific interest for a number of potential applications. This method involves a multiphoton absorption process which can induce a local refractive index change, ranging from 10^{-5} to 10^{-2} , even in non-photosensitive transparent materials [6-8]. As a result, FBGs have been successfully written in various types of optical fibers, and their characteristics have been examined [7-20]. Varying the laser intensity used to inscribe the gratings result in both type I-IR and type II-IR FBGs (where the IR suffix denotes the use of light from an infra red (IR) laser in the grating formation) being obtainable [16] and thermal stability tests carried out demonstrate that type II-IR FBGs exhibit excellent stability above 1000 °C. This very positive result for a number of fiber sensor applications is likely the result of a nonlinear self-focusing process where ultra high peak power locally affects the glass structure [17]. However, the type II-IR FBGs written in non-photosensitive fibers have poor spectral quality as the "damage" region produced by the ultrafast laser exists not only in the core but also in the cladding.

In the work, the spectral and annealing properties of FBGs written in both H₂-free and H₂-loaded fibers by use of 120 fs, 800 nm laser pulses, through a phase mask, have been

investigated. It is found that type II-IR gratings inscribed in H₂-loaded fibers exhibit better spectral quality than those written in H₂-free fibers. Moreover, the stability of the type II-IR gratings can be substantially increased at a very high temperature (of more than 1100 °C) if a pre-annealing treatment is implemented, thereby making them ideal sensor elements for very high temperature monitoring applications.

2. Experimental setup

The experiment to write the gratings investigated in this work was performed using of a Ti: sapphire laser system consisting of an oscillator (Mai Tai) and an amplifier (Spitfire Pro). The amplified Ti: sapphire laser emits pulses of 120 fs with linearly polarized light at a central wavelength of approximately 800 nm (TEM₀₀ spatial mode, repetition rate of 1 kHz) and a 1/e Gaussian beam radius $\omega_0 = 2\text{mm}$. The maximum pulse energy of the laser output was ~1 mJ, which could be attenuated by rotating a half waveplate followed by a linear polarizer. The laser beam was focused using a cylindrical lens with a focal length of 60mm through a silica phase mask into the fiber. Considering the case of Gaussian beam optics, the width of the focal spot size would thus be $7.6\mu\text{m}$ which may be calculated using $\omega \approx \lambda f / \pi \omega_0$, where λ is the wavelength, f is the focal length of the focusing lens, and ω_0 is the incident beam diameter. As no extra apodization technique was used for the Gaussian profile beam, the exposure was not uniform.

The phase mask used in this series of experiments (Ibsen Photonics) was optimized for 800 nm illumination, with the first-order diffraction efficiency of 72.8 %. The diffraction angle for 800 nm light was 48.33°, so the regions of the ±1 order beams overlapped coherently within the distance up to 0.36 mm from the phase mask. Since ~ 20.6 % of the zeroth order diffraction cannot be blocked, partial annealing occurs during the growth of the grating.

H₂-loaded SMF-28 fibers used in the work reported were loaded with hydrogen (at a temperature of 85 °C and a pressure of 3000 psi (approximately 200bar)) for 24h. During the grating inscription, the fiber was positioned in close proximity to the phase mask (being within a distance about 300 μm) by use of a high-precision four-axis translation stage. The laser focus was adjusted in a way that allowed the beam to enter the fiber core to ensure the efficient grating inscription.

The annealing properties of the fabricated gratings were studied by inserting the fiber into an ISOTHERMAL PEGASUS^{PLUS} 1200 tube furnace, which created the temperature range that was used of between 150 and 1200 °C (with stability of between ± 0.05 to ± 0.2 °C). Along with a standard (reference) probe, gratings were loosely placed into the isothermal enclosure (a metal block) so that no external stresses were applied to the grating. The temperature in close proximity to the grating was monitored using a thermocouple probe. The annealing test was performed on ambient air, and the reflection spectrum of the grating, monitored during the annealing process, was measured by use of a super wideband light source (Amonics ALS-CWDM-FA) and an optical spectrum analyzer (YOKOGAWA AQ6319) with a resolution of 0.02 nm.

3. Results and discussion

The results of the tests carried out on the series of gratings fabricated as discussed above are now considered. It was found that there were two distinct intensity thresholds for grating inscription in H₂-free SMF-28 fibers, corresponding to creating type I-IR and type II-IR FBGs respectively [9]. In the experiments undertaken, two different intensity thresholds for FBG inscription also were observed in H₂-loaded SMF-28 fiber and the FBGs thus fabricated possessed different features in terms of their spectral quality and thermal stability.

3.1 FBG inscription results

In this work, type I-IR and type II-IR FBGs have been fabricated in both H₂-free and H₂-loaded SMF-28 fibers by use of 120 fs, 800 nm femtosecond pulses, irradiating the fiber through a phase mask, and the corresponding threshold intensities and the exposure dose required are shown in Table 1. The typical reflection spectra of the FBGs obtained are presented in Fig. 1.

Table 1. Experimental results of irradiation of several fibers with 120 fs, 800 nm laser pulses

Fiber	Grating	Reflectivity, dB	Threshold intensity, $10^{12} W/cm^2$	Exposure dose, kJ/cm^2
H ₂ -free SMF-28	Type I	10	8.4	756
	Type II	13.2	13.2	26.4
H ₂ -loaded SMF-28	Type I	12.3	3.6	324
	Type II	13.5	12	24

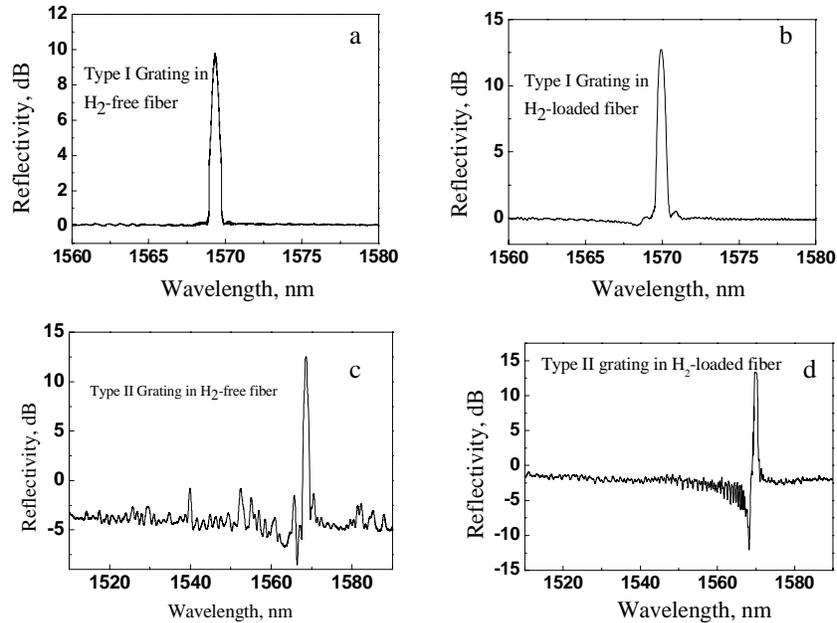


Fig. 1. Reflection spectra of the FBGs fabricated (a) in a H₂-free SMF-28 fiber with 350 μ J pulse energy; (b) in a H₂-free SMF-28 fiber with 550 μ J pulse energy; (c) in a H₂-loaded SMF-28 fiber with 150 μ J pulse energy; (d) in a H₂-loaded SMF-28 fiber with 500 μ J pulse energy.

A type I-IR FBG with reflectivity of 10 dB, spectral bandwidth of 0.45 nm and an out-of-band insertion loss of < 0.1 dB was inscribed in the H₂-free SMF-28 fiber with the pulse energy of 350 μ J (intensity of about $8.4 \times 10^{12} W/cm^2$) and ~ 20 min exposure time, i.e. about 1.2 million pulses, (as shown in Fig. 1(a)). Assuming a uniform FBG (of 4 mm length), the corresponding amplitude of the refractive index modulation in the fiber core was estimated to be $\Delta n_{ind} = 4.2 \times 10^{-4}$. When the pulse energy was increased, type II-IR FBGs were fabricated in this same fiber by the use of a pulse energy of 550 μ J (an intensity of about $1.32 \times 10^{13} W/cm^2$) and using several seconds exposure time (as shown in Fig. 1(b)), the FBG obtained exhibited the reflectivity of 13.2 dB, an out-of-band insertion loss of ~ 5 dB and a 3dB bandwidth of ~ 0.98 nm. Under similar exposure conditions, type I-IR and type II-IR FBGs were also written

in H₂-loaded fibers with a pulse energy of 150 and 500 μJ (as shown in Figs. 1(c) and (d) respectively). Hydrogen loading results in a dramatic enhancement of the photosensitivity of the Ge-doped fibers, which leads to a significantly lowering of the grating writing threshold. The 3dB bandwidth of the type II-IR gratings was observed to increase from 0.42 nm (type I-IR grating) to 0.88 nm, this being due to the reduction in the effective length of the grating [16]. The low out-of-band insertion loss was observed to be ~ 3 dB.

The relatively poor quality of the reflection spectra of the type II-IR FBGs may result from the strong coupling into the cladding modes due to the non-uniform refractive index change across the core, which originates from the multiple-beam interference produced by the femtosecond pulses focused on the fiber through the phase mask. However, the type II-IR FBGs written in H₂-loaded fibers (as shown in Fig. 1(d)) still have better spectral quality than those in the H₂-free fibers, which is probably related to the enhanced photosensitivity of the fiber core and the relatively small laser intensity employed. The dominant mechanism for inscription of FBGs using IR femtosecond radiation is widely thought to result from the multiple-photon absorption [7-9], which would not happen unless the laser pulse intensity is high enough to reach the threshold. In the case of doped fibers, material modification would only occur in the core by adjusting the pulse intensity precisely if the multiple-photon ionization (MPI) threshold in the core was lower than that in the cladding [14]. Hydrogen loading can greatly improve the absorption of laser energy in the core region, which leads to a lower MPI threshold of the core. As a result, the structural modifications or damage caused is mainly located in the fiber core region and little effect is seen beyond the core-cladding interface.

With precise alignment of the system, type II-IR FBGs can be fabricated in both H₂-free and H₂-loaded fibers using a laser pulse exposure of a few seconds (about several thousand pulses), as the large refractive index modulation caused happens quite rapidly. However, it is difficult to determine an accurate value of the time required for grating formation as the grating growth is sometimes accompanied by the erasure [10].

3.2 Annealing and temperature sensitivity results

The thermal stability of the FBGs written using the femtosecond laser pulses is dependent on the inscription conditions, most notably the pulse duration, the pulse energy and the fiber alignment [11, 16-17]. In general, type I-IR and type II-IR FBGs show better thermal stability when compared with that obtained from UV induced gratings, typical of excimer lasers. In particular, type II-IR FBGs can sustain high temperatures up to 1000 °C while maintaining a high reflectivity due to the thermally ultrastable defects produced by the high intensity laser pulses [17]. However the exact mechanism of type II gratings formation under high-intensity IR excitation is still unknown.

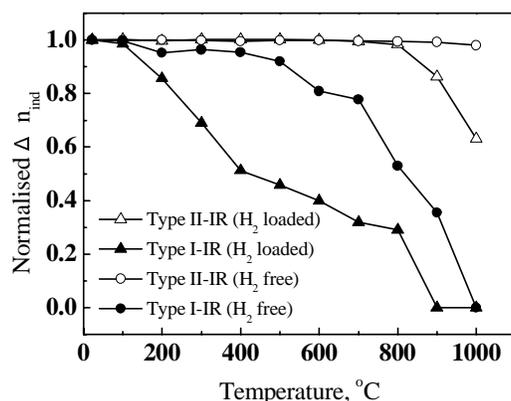


Fig. 2. Short-term annealing study of FBGs written in two types of fibers. (a) in a H₂-free fiber with 350 μJ pulse energy; (b) in a H₂-free fiber with 550 μJ pulse energy; (c) in a H₂-loaded fiber with 150 μJ pulse energy; (d) in a H₂-loaded fiber with 500 μJ pulse energy.

In the experiments carried out, the thermal stability of the FBGs obtained in both H₂-loaded and H₂-free SMF-28 fibers were characterized by the isochronal annealing approach. The decay in the grating reflectivity can be represented more scientifically in terms of the decay in the refractive index modulation of the gratings written into these fibers [5]. Fig. 2 shows the variation of the normalized refractive index modulation Δn_{ind} when the fibers were subjected to short-term thermal exposure (30 min at each temperature) at 100 °C, 200 °C and then progressively to 1000 °C with a temperature increment of 100 °C.

When the temperature is increased, it was observed that in both H₂-free and H₂-loaded fibers, type II FBGs were more thermally stable than type I FBGs. It was observed particularly that type II-IR FBGs written in H₂-free fibers had the highest thermal stability, this being followed by H₂-loaded type II FBGs and then H₂-free type I FBGs. H₂-loaded type I gratings had the poorest thermal stability. A 50% decrease of the normalized Δn_{ind} was reached at temperatures of 400 °C and 800 °C, for the H₂-loaded and the H₂-free SMF-28 fibers, respectively. In particular, the stability of the H₂-loaded FBGs was seen to decrease rapidly and the grating completely disappeared when the temperature approached to 900 °C. In summary, type I-IR FBGs can be easily fabricated (especially in hydrogenated fibers), have high spectral quality and good reproducibility but they have relatively low thermal stability. The index change induced in type I-IR gratings is likely due to the highly nonlinear defect formation resulting from a multi-photon absorption process. These defects, like those associated with type I-UV exposures, can be annealed out below the glass transition temperature. However, an increase of the type II grating strength with temperature was not observed in both H₂-free and H₂-loaded samples, this result being different from that reported in References [17] and [18]. This is probably caused by the phase mask used, which was not zeroth order suppressed and it was estimated that ~22 % of the zeroth order diffraction could not be blocked. Such a zeroth order diffraction beam created an annealing effect during the growth of the grating and as a result, the “weakly” induced index change was annealed out under conditions where the irradiation time was sufficiently long.

While type I FBGs created by IR pulses due to multi-photon absorption were almost erased as the temperature exceeded 900 °C, the type II FBGs revealed ultra-high thermal stability. Type II-IR gratings written in H₂-loaded fibers were almost unaffected by thermal exposure at temperatures of up to 800 °C as were those written in H₂-free fibers. Above this a portion of the refractive index change of this grating was annealed out, resulting in a degradation of grating reflectivity from $\Delta n_{ind} = 1.7 \times 10^{-3}$ to 1.2×10^{-3} within a few minutes of the temperature reaching 900 °C. This was probably due to a large portion of the initial total index change coming from an annealable index change rather than a permanent damage type index change [17]. However the type II-IR gratings written in H₂-free fibers remained stable at temperatures up to 1000 °C because of their ultra-stable local damage structure.

It can also be observed from Fig. 2 that the FBGs fabricated in H₂-free fibers have relatively good thermal stability, and the type I-IR FBGs can sustain temperatures of 500 °C with 96 % of its initial Δn_{ind} remaining, showing an almost negligible decay rate while type II-IR FBGs can retain more than 98 % of its initial Δn_{ind} after a 30 min exposure at a temperature of 1000 °C.

The short-term annealing test described above shows that type II-IR gratings written in both H₂-free and H₂-loaded fibers are extremely stable below 800 °C. A long-term thermal stability test for type II-IR gratings was also carried out by heating the gratings to 700 °C and then they were kept at that temperature for 12 hrs. The results obtained show that there was almost no degradation of the grating strength in both of the fibers. The gratings were subsequently heated to 1000 °C (and remained at that temperature for 12 hrs) with the evolution of the grating reflectivity and the resonant wavelength being recorded, as shown in Figs. 3(a) and 3(b). The calculated Δn_{ind} determined from the reflection spectra are plotted as a function of annealing time. It can be noted that the thermal stability of the H₂-loaded type II-IR FBGs are substantially increased over the high temperature range considered arising from

the 700 °C annealing treatment and thus could sustain high temperature of up to 1000 °C. After 12 hours, there was only a slight degradation of the grating strength for the duration of the test. This property is similar to that of type R (regenerated) gratings created during high temperature annealing of type I gratings [21, 22]. However, the tested gratings are indeed type II “damage” gratings in H₂-loaded fibers (the damage morphology of the fiber cross section in the grating region has been observed during the experimental work). In the experiment carried out here, the resonance wavelength was also measured each hour during the annealing process, and shown to be approximately constant regardless of the annealing time at 1000 °C (as shown in Fig. 3(b)). A closer look at this figure (the insert) reveals the slow drift toward longer wavelengths for both H₂-free and H₂-loaded gratings and that the former varied rapidly.

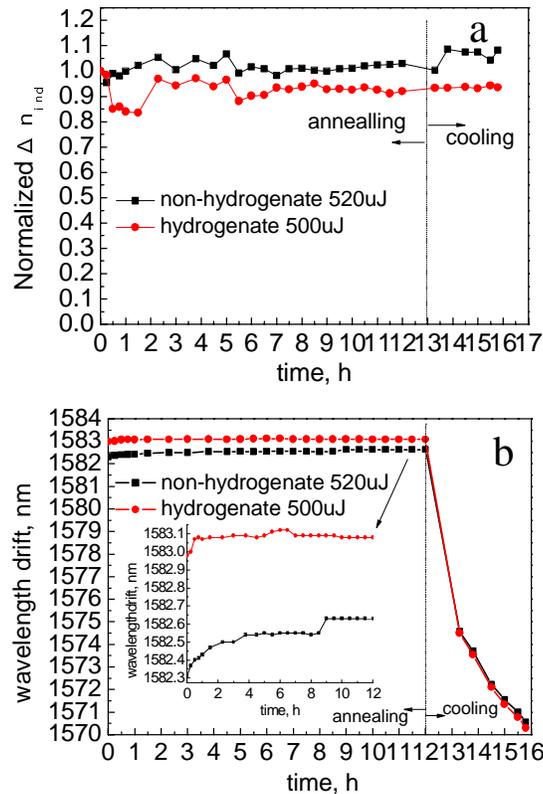


Fig. 3. Isothermal evolution of the reflection and resonant wavelength of type II-IR FBGs inscribed in non-hydrogenated and hydrogenated fibers over a 12 h period at 1000 °C annealing followed by 4 h cooling. (a) the response in terms of reflectivity; (b) the wavelength drift response.

Type II-IR gratings are created when the laser intensity is greater than the damage threshold of the particular glass, an effect which is similar to that in type II-UV “damage” gratings. Since a high pulse intensity can be provided, the total stress relief obtained through local fracture enables an ultra stable grating structure. The accurately localized damage can also produce gratings with high spectral quality [19, 23]. In addition, the large local refractive index change leads to filamentation which arises in part from self focusing effects by the generated plasma and can be used to prevent collateral damage, which is more familiar from the use of long pulse lasers [23].

The evolution of the reflectivity and the resonant wavelength of type II-IR FBGs inscribed in both H₂-free and H₂-loaded fibers can also be observed during the annealing process. Fig. 4 demonstrates the reflection spectra at the beginning and the end of the long-term annealing

process at 1000 °C, along with the original spectra both at room temperature and then after 4 hrs cooling. The spectral quality for both gratings was found to be greatly improved arising from the high temperature annealing treatment. The reason for this is that the thermally activated defects induced by the high intensity pulses are annealed out, leaving a very smooth interface between the structurally altered region and the unaffected region within the material.

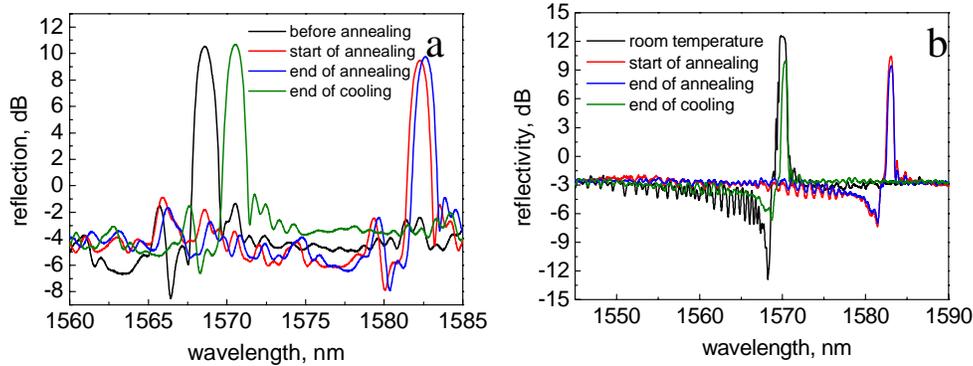


Fig. 4. Spectral evolution of Type II-IR FBGs inscribed in (a) non-hydrogenated and (b) hydrogenated fibers at the four times shown during the annealing process.

In the next stage of the investigation, the temperature was subsequently increased beyond 1000 °C and stabilized at 1150 °C. The gratings, however, were gradually erased, as expected, during this very high temperature heat-treatment process. Type II-IR FBGs inscribed in the hydrogenated fibers decayed rapidly and disappeared within several minutes, while the H₂-free FBGs decreased relatively slowly and the corresponding spectral response was recorded in Fig. 5. The FBG written in the H₂-free fiber decreased rapidly and reached zero dB after a period of 442 min. It should be noted that the grating was not completely erased as there was ~ 5 dB insertion loss of the grating (seen from the insert in Fig. 5(a)). The Bragg resonance λ_{Bragg} of the grating shifted slowly towards a shorter wavelength with annealing time (as shown in Fig. 5(b)), which coincided with the grating decay. However, the shift was not monotonic and fluctuations in the resonance wavelength were observed during the annealing process. λ_{Bragg} is related to the effective index and the grating period, both of which change with the temperature: however their high temperature behavior is still not well understood.

4. Conclusion

Different types (type I-IR and type II-IR) of FBGs have been fabricated in both H₂-free and H₂-loaded SMF-28 fibers by use of 800 nm femtosecond laser pulses, illuminating through a phase mask. The temperature sustainability and the spectral characteristics of the FBGs thus created were examined in some detail and cross-compared. It was found that type II-IR FBGs inscribed in H₂-loaded fibers exhibit better spectral quality when compared with gratings written in H₂-free fibers, a result which probably arises due to the fact that the structural change is confined to the Ge-doped core region of the fiber. Furthermore, the stability of type II-IR FBGs written in H₂-loaded SMF-28 fibers is substantially increased at the high temperature considered through pre-annealing heat treatment at 700 °C and the highest temperature reached before erasing is 1150 °C, a similar result to that for type II FBGs written in H₂-free fibers. A permanent drift of the central wavelength at a temperature of 1150 °C is observed along with a very significant reduction of the grating strength. The long-term annealing test undertaken demonstrates the high potential of type II-IR FBGs to be used as fiber optic sensing elements well suited to applications where very high temperatures (up to 1100 °C) are experienced.

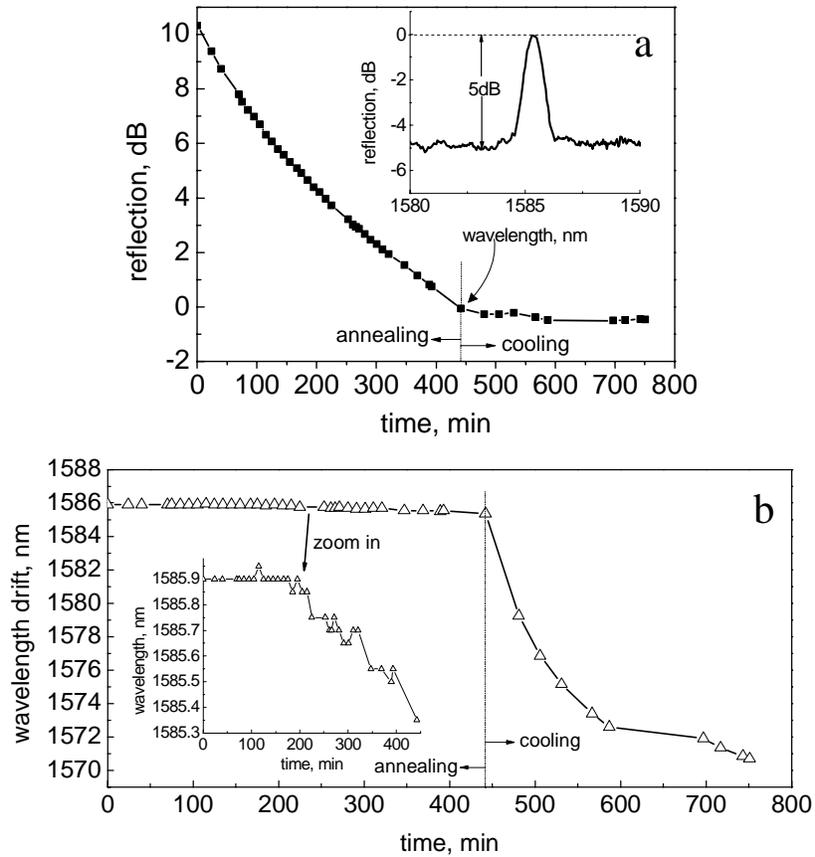


Fig. 5. Isothermal evolution of (a) the reflection and (b) resonant wavelength of Type II-IR FBGs inscribed in H₂-free fibers during 442 min (at 1150°C) annealing and 180 min cooling.

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