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Realization of optical fiber regenerated gratings by rapid cooling and split annealing

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Rapid cooling, or quenching, during regeneration of seed gratings in standard single-mode silica optical fiber is explored. It is shown that regeneration can be broken up into stages in time. The novel, to the best of our knowledge, method of "split annealing" offers a unique tool for optimizing regeneration and studying fundamental glass science within a one-dimensional bi-material system. We demonstrate regeneration at temperatures as high as $T = 1200°C$ for the first time as well as opening up an approach suited to batch processing of regenerated gratings.

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Regenerated fiber Bragg gratings (RFBGs) have shown excellent high-temperature stability, exceeding $T = 1000°C$ [1,2]. They yield cleaner spectra when compared to other high-temperature stable gratings, such as Type-II gratings [3], and have negligible lower-order cladding mode losses enabling easy multiplexing of sensors in practical applications. The major concern, however, is that at higher temperatures stress relaxation between the core and cladding reduces fiber strength, making the glass more brittle and requiring more care in handling. In addition, the strength of the gratings is typically about $R \sim 10–18\%$ of the seed grating, when regenerated at any temperatures $T > 850°C$. The glass relaxation mechanism behind regeneration is covered in [4]. A general review of other literature describing the regenerated grating formation and their applications reported during the last one and half decades is given in Ref. [5]. Long-term stability of RFBGs at elevated temperatures has also been studied in the recent past [6–8]. It has already been established that thermal processing methodologies have a major influence on regeneration strength. The rationale behind this is understandable. There are various background contributions that can impact glass relaxation including: stress relaxation in bi- and tri-material systems such as optical fibers and around dopant sites usually in the core [4,9], the diffusion of added dopants such as hydroxyl and hydrides [10,11], formed with hydrogen bonding when gratings are written in the presence of hydrogen, and "water" impurities at elevated temperature [12]. All affect glass density which can be characterized in part by silica ring size, folding, and strains. The annealing of irradiation-induced defects [13] also relaxes local glass structure by removing strain and allowing potential diffusion. This latter component is important because glass defects allow excitation of the glass network at longer wavelengths using lasers allowing a degree of periodic micro-processing that is not possible by direct thermal treatment alone. These features can be lumped into net structural changes that impact the glass transition—this transition will differ where the glass has been processed differently during regeneration from seed gratings.

Therefore, knowledge about the influence of temperature, T, on regeneration is critical to optimizing and tuning regeneration and realizing stronger RFBGs. Typical annealing methodologies, such as isochronal annealing or a combination of isochronal and isothermal annealing adopted so far for grating regeneration, have failed to explore this relaxation [5]. This is because all the background processes in these cases experience a continuously varying thermal ambiance which is determined by the ramping rate and set dwelling time during combined isochronal and isothermal annealing. Later, a method of fast regeneration by rapid annealing allowed regeneration exclusively under isothermal conditions [14]. This facilitated further insight into grating regeneration with fundamental understanding of a bi-material glass host of an optical fiber under rapid heating. Using this method of rapid annealing, we demonstrated for the first time that similar seed gratings inscribed in standard Ge-doped single-mode fibers can be regenerated at different temperatures under isothermal condition [15]. The time to regenerate gratings was found to be longer at lower temperatures with a gradual enhancement of regeneration efficiency. It was also shown that regeneration of gratings during annealing has three distinct phases: (i) the "erasing phase" during which seed grating R diminishes until submerged in the measured noise floor; (ii) the "incubation phase" where the grating peak remains unobservable until it emerges out of the noise floor again; and (iii) the "regeneration phase" in which R can be seen increasing from then onwards until subsequent saturation.

At lower temperatures, when the quenching rate is slower, regeneration efficiency is more pronounced, but it takes a significantly longer time. On the other hand, at higher temperatures, faster quenching means faster regeneration but with reduced regeneration efficiency. This is because average thermal diffusion of structure between periodic regions is increased, i.e., the fringe contrast between laser-processed grating regions is washed out more effectively with higher temperatures. Considering the complexity of the entire process, it is therefore necessary to better understand and optimize the temperature dependence of each phase independently so that the entire regeneration process can be optimized both in terms of manufacturing time (less is better) and regeneration efficiency (higher is better) by combining isothermal treatment at different phases. Addressing this challenge forms the basis of the work reported in the present paper.

Here, we report novel rapid quenching, or cooling, of the grating on regeneration during thermal treatment at a constant temperature. We demonstrate for the first time that by rapidly cooling the grating, optimal changes leading to regeneration can be arbitrarily "frozen in" at room temperature. This allows separation of the regeneration into various stages, showing that the regeneration process can be broken into phases that can be carried out arbitrarily in time in steps. This makes it possible to process and study each phase selectively at a different T, a novel concept we name "split annealing". Using judicious thermal treatment at different phases we show that it is now possible to produce strong regenerated gratings at temperatures beyond $T = 900°C$.

Regeneration was conducted at different values of T using seed gratings ($L = 10$ mm) fabricated with $\lambda = 213$ nm laser (repetition rate = 15 kHz, $E \sim 1.3$ µJ/pulse, scanning speed $v = 0.02$ mm/s) in SMF-28e fiber (H_2 loaded @ $P = 1500$ psi, $T = 100°C$, $t = 24$ hours). The peak attenuation at λ_{Bragg} for all the seed gratings was $R \sim -50$ dB. For strong gratings, the FWHM (@ $R = -0.5$ dB) of reflection bandwidth was kept to $\Delta\lambda = (0.52 \pm 0.02)$ nm, ensuring similar AC index modulation. The bulk (DC) index change during writing was determined by monitoring the Bragg wavelength, λ_{Bragg}, and shift, $\Delta\lambda_{Bragg}$. For all the seed gratings, $\Delta\lambda_{Bragg}$ during inscription was $\sim (15 \pm 2)$ pm ensuring a similar fringe contrast. A tubular oven (OTF-1200X, MTI Corporation, USA) was used for thermal processing of the seed gratings. A standard FBG interrogator (SI-720, Micron Optics Inc., USA) recorded R and transmission spectra during annealing.

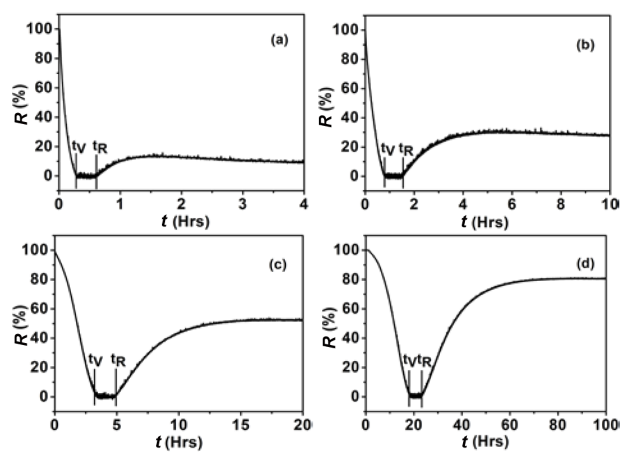

Fig. 1. Isothermal annealing at (a) $T = 900°C$, (b) $T = 850°C$, (c) $T = 800°C$, and (d) $T = 750°C$.

Table 1. Record of t_V, t_R, and t_R–t_V During Continuous Annealing under Isothermal Conditions

T (°C)	t_V (h)	t_R (h)	t_R–t_V (h)	R (%)
900	~0.30	~0.67	~0.37	~13
850	~0.80	~1.67	~0.87	~30
800	~3.48	~4.9	~1.44	~50
750	~17.06	~24.6	~7.50	~80

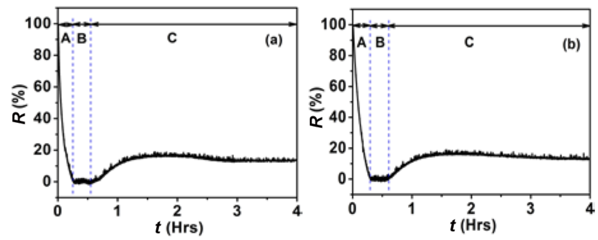

Fig. 2. Split annealing at $T = 900°C$. At the end of phases 'A' and 'B,' the grating was rapidly cooled and kept at RT for (a) $t = 1$ h, and (b) $t = 24$ h before being re-inserted into the oven. 'C'is the regeneration phase.

Table 2. Record of t_V, t_R, and t_R–t_V During Continuous and Split Mode Annealing under Isothermal Conditions

T (°C)	Annealing Mode	t_V (h)	t_R (h)	t_R–t_V (h)	R (%)
	Continuous	~0.30	~0.67	~0.37	~13
900	**Split (1 h)**	~0.30	~0.65	~0.35	~16
	Split (24 h)	~0.30	~0.69	~0.39	~15

Firstly, four seed gratings were regenerated by rapid continuous annealing under isothermal conditions. The seed gratings were inserted in a preheated oven set at $T = 900$, 850, 800, and 750°C, respectively. Figure 1 shows the peak reflectivity, R, as a function of time, t, during the annealing. The time t_V is the time taken to complete the erasing phase, i.e., the time when R is submerged in the noise floor. Similarly, t_R denotes the time when the regeneration phase starts, i.e., when the peak R reappears after it was inserted in the oven. The incubation phase when R is in the noise floor is characterized by t_R–t_V. It is apparent from the results that regeneration efficiency and regeneration time depends upon the T at which the seed gratings were annealed. Table 1 shows the details.

In the next experiment, a similar seed grating (sample 1) was inserted in the preheated oven ($T = 900°C$). Instead of allowing the grating to pass through all the three phases, it was rapidly cooled down by quickly pulling it out of the oven around t_V, i.e., once the grating peak had vanished at the end of the erasing phase. The grating was kept at RT for $t = 1$ hour before being re-inserted into the preheated oven ($T = 900°C$) for rapid annealing and to allow the seed grating to isothermally progress through the incubation phase. The grating was withdrawn again around t_R once the incubation phase was over at the moment when the grating peak just reappeared. The sample was again kept at RT for an hour before it was further inserted in the preheated oven ($T = 900°C$) for annealing for the final regeneration phase. The peak R recorded during all three rapid annealing phases carried out in steps were concatenated and are plotted as a function of time, t (Fig. 2). The parameters for these two experiments are summarized in Table 2. It was interesting to observe that the final regeneration strength remains comparable

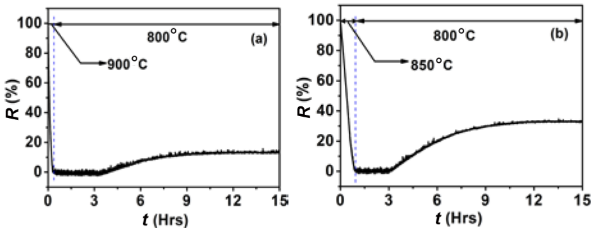

Fig. 3. (a) Erasing the seed grating at $T = 900°C$ and incubation and regeneration at $T = 800°C$ attained $R \sim 15\%$ reflectivity; (b) erasing the seed grating at $T = 850°C$ and incubation and regeneration at 800°C attained $R \sim 31\%$ reflectivity.

when treated under similar isothermal conditions, irrespective of the annealing approach, either continuous or in split mode where the process is divided into stages. This staged processing, or split annealing, was verified by repeating the experiment at $T = 900°C$ where the seed grating was kept at RT for 24 hours after rapidly cooling the fiber at the end of each stage.

Splitting the entire regeneration process was further verified by repeating the experiment at $T = 850°C$ and keeping the grating at RT for times of $t = 24$, 72, and 168 hours, respectively, at intermediate stages. (Results may be seen in Supplement 1 for reference.) These experimental results substantiate that the background processes during regeneration of the grating by rapid isothermal annealing can be intermittently frozen in and then can be restarted at will. In all cases, the final regeneration efficiencies were similar to when the seed grating was regenerated through continuous annealing. The ability to freeze in a glass structure arbitrarily during regeneration paves the way to decouple all three phases of grating regeneration, allowing a novel approach to regeneration and its implementation in practice by the method of split annealing. It makes it possible to study the influence of annealing the seed at any temperature without T being the same in each phase.

Our investigations to study the influence of annealing temperature on regeneration by splitting the annealing process was divided in two parts. In the first part, the seed gratings were erased at high temperature and subsequently incubated before being regenerated at a lower temperature. It is already established that processing the gratings at lower temperature yields strong RFBGs but the time taken to regenerate is appreciably longer. Being able to erase the grating faster and subsequently incubating and regenerating the grating at a slower rate without losing the RFBG strength would be desirable from a manufacturing perspective. Two samples (sample 2 and sample 3) were erased at $T = 900°C$ and 850°C, respectively, then taken out of the oven after the erasing phase and kept at RT for $t = 24$ h. Sample 2 and sample 3 individually were then incubated and regenerated at $T = 800°C$. The peak R as a function of time t was recorded during the erasure, incubation, and regeneration phases for these two samples and are shown in Fig. 3. It is interesting to note that by continuously annealing a seed grating throughout at $T = 800°C$, RFBGs with $R \sim 50\%$ were obtained (Table 1), whereas erasing the seed gratings at $T = 900°C$ (sample 2) and at $T = 850°C$ (sample 3) and subsequently incubating and regenerating these samples at $T = 800°C$ produced weaker gratings. Sample 2 and sample 3 had $R \sim 15\%$ and $\sim 31\%$, respectively. It is also important to note that this matches the values when the samples were annealed continuously at $T = 900°C$ and $T = 850°C$ (Table 1). Therefore, annealing the grating at low

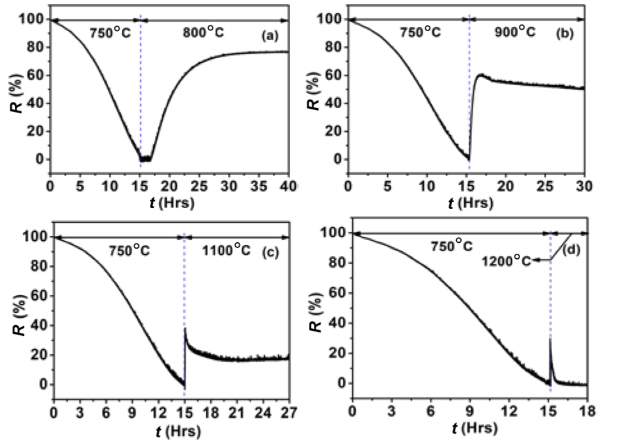

Fig. 4. (a) Erasing the seed grating at $T = 750°C$ and incubation and regeneration at $T = 800°C$; (b) incubation and regeneration at $T = 900°C$; (c) incubation and regeneration at $T = 1100°C$, and (d) incubation and regeneration at $T = 1200°C$.

temperature only during the incubation and regeneration phases does not help produce strong RFBGs. Rather, it is the temperature at which the seed is erased that matters most. This is consistent with frozen in glass changes during initial quenching.

In the subsequent investigation, the annealing conditions were reversed. The seed gratings were erased at a lower temperature than the temperatures at which the gratings were later incubated and regenerated. Four seed gratings ('sample 4' to 'sample 7') were erased at $T = 750°C$ then incubated and regenerated individually after insertion into a preheated oven. Sample 4 was annealed at $T = 800°C$ during incubation and regeneration, sample 5 at $T = 900°C$, sample 6 at $T = 1100°C$ and sample 7 at $T = 1200°C$. The results of these experiments are shown in Fig. 4 where the measured R as a function of t for all these samples is shown. In Fig. 4(a), incubating and regenerating the seed at $T = 800°C$ produced $R \sim 80\%$ regeneration in $t \sim 15$ h since it was erased (t_v). This level of grating strength could be achieved by continuously annealing the seed grating throughout at $T = 750°C$ and the time taken for incubation and regeneration was ~ 50–55 h after t_v [see Fig. 1(d)]. In Fig. 4(b), an RFBG with a reflectivity of $R \sim 60\%$ was produced by incubating and regenerating the seed grating at $T = 900°C$. It is important to note that it was never possible to produce an RFBG with a reflectivity $R > 10$–18% by annealing the seed continuously at $T = 900°C$ [see Fig. 1(a)], whereas by split annealing, a strong RFBG could be produced at $T = 900°C$. The result shown in Fig. 4(c) demonstrates that RFBGs can be obtained even at $T = 1100°C$ annealing in split mode. The figure shows that reflectivity rose to $R \sim 40\%$ during regeneration but subsequently came down and settled at $R \sim 20\%$. Importantly, with rapid annealing of the seed gratings continuously at any temperature at or above $T \sim 1000°C$, no regeneration was observed. The result shown in Fig. 4(d) is also significant. It appears that for this glass host, the limit of grating regeneration has been reached. The figure shows that the regeneration with $R \sim 30\%$ was immediately observed as soon as the seed grating, which was erased earlier at $T = 750°C$, was inserted in an oven which was preheated at $T = 1200°C$. However, the regenerated grating structure that can be produced at this temperature was not stable and the fringe contrast was washed out, indicating that glass viscous flow is significant.

In summary, novel "split annealing" allows an integrated demarcation of stages in regeneration for producing RFBGs. A number of advantages were demonstrated. The quality and strength of RFBGs have been substantially enhanced with the regeneration limits identified in the glass system analyzed. We have confirmed the role of the amorphous nature of glass processing and the impact that has on regeneration. By freezing in glass structure at will, the process of split annealing allows optimization of the regeneration process. Much of this optimization can be accounted for by reducing the impact of viscous flow in washing the grating fringe out. Glass processing is occurring in periodically varying regions simultaneously, leading to viscous flow between the regions that are inhibited at lower T and at faster quenching rates, optimizing the fringe contrast obtained. As well as optimizing the regeneration process with total control and understanding, split annealing enables the process to be adapted to a manufacturing environment. This is a new paradigm in optical fiber and waveguide grating production and can be extrapolated to complex laser patterning of glass in multi-material systems such as those anticipated to contribute toward additive manufacturing. It exploits the well-established glass processing science of amorphous networks that have continuum structural states but within the added confines of multiple periodic processing sites in an approximate one-dimension model bi-material glass system. The bi-material system gives rise to two glassy transition temperatures determined in part by the presence of glassy dopants (such as Al which raises the glass transition) and impurities in the core and the original fiber drawing conditions. It is noted that stress contributions arise in integrated optics from deposition on a substrate and so similar opportunities to exploit regeneration exist. Exploring second regeneration with these strong RFBGs regenerated beyond 1000°C will also be of interest [16]. The stage is set for the mass production of optimized ultrahigh temperature fiber components such as the batch production of RFBGs as required. Many gratings can be stored after fabrication and the seed gratings collectively erased at low temperature, a process that can take a longer time. This avoids long-term storage of the final product which is brittle. The samples can then be quickly, identically, and optimally incubated and regenerated at any desired time and temperature as required. They can also be regenerated inside prepackaged high-temperature components where such packaging could not be done after regeneration, for example in extended space operations where extreme environments demand extreme packaging.

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Disclosures. The authors declare no conflict of interest.

Data availability. Data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon request.

Supplemental document. See Supplement 1 for supporting content.

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