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Stephen C. Warren-Smith, Erik P. Schartner, Linh V. Nguyen, Dale E. Otten, Zheng Yu, David G. Lancaster, and Heike Ebendorff-Heidepriem

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Stability of grating-based optical fiber sensors at high temperature

Stephen C. Warren-Smith, Erik P. Schartner, Linh V. Nguyen, Dale E. Otten, Zheng Yu, David G. Lancaster, and Heike Ebendorff-Heidepriem

Abstract—**We present a comparison of four different grating-based optical fiber high temperature sensors. Three of the sensors are commercially available and include a heat treated, twisted (chiral) pure-silica microstructured optical fiber, a femtosecond laser written Bragg grating in a depressed cladding single mode fiber and a regenerated fiber Bragg grating. We compare these to an in-house fabricated femtosecond laser ablation grating in a pure-silica microstructured optical fiber. We have tested the sensors in increments of 100°C up to 1100°C for durations of at least 24 hours each. All four sensors were shown to be operational up to 900°C, however the two sensors based on pure-silica microstructured fiber displayed higher stability in the reflected sensor wavelength compared to the other sensors at temperatures of 700°C and higher. We further investigated high temperature stability of silica suspended-core fibers with femtosecond laser inscribed ablation gratings, which show improved stability up to 1050°C following thermal annealing. This investigation can be used as a guide for selecting fiber types, packaging, and grating types for high temperature sensing applications.**

Index Terms—**Optical fiber sensors, fiber Bragg gratings, temperature measurement.**

I. INTRODUCTION

Optical fibers are attractive for temperature sensing as they are small, light-weight, immune to electromagnetic interference, resistant to corrosion, and can provide either distributed or multi-point sensing [1, 2]. Grating based temperature sensors are particularly attractive for applications where localized sensing is required and can act as an immediate replacement to existing electrical (e.g. thermocouple) sensors. However, conventional fiber Bragg grating (FBG) fabrication techniques, whereby ultra-violet light is used to induce periodic refractive index modulations in photosensitive glass, can operate to only several hundred degrees Celsius before failing [3].

Grating based sensors that operate at higher temperatures are an area of active research. One approach is to anneal a conventional FBG in a doped-silica single-mode fiber (SMF) to form what is known as a regenerated FBG [4-7]. These gratings have been demonstrated to survive at temperatures up to 1295°C [8]. Alternatively, twisted fibers can be heat treated to induce permanent chiral gratings such as long period gratings and Bragg diffraction gratings [9-11]. Another promising technique is the use of a femtosecond (fs) laser to write damage (type II) gratings [12, 13]. We have previously shown this latter technique is particularly effective if used to create surface ablation FBGs in pure-silica microstructured optical fibers (MOF) [14].

The optical fiber material type is also a critical parameter when considering the high temperature operation of optical fiber sensors. Significant attention has been given to FBGs inscribed into sapphire crystal fibers, which can operate in excess of 1400°C [15, 16]. However, sapphire crystal fiber is generally limited in length due to high loss and is unstructured (cladding-less) fiber. This results in a high numerical aperture fiber that supports the propagation of a large number of higher-order modes, thus giving a broad FBG reflection spectra. This broad reflection limits the multiplexing capability, such as to three FBGs within a 100 nm spectral bandwidth [17] and restricts the temperature measurement resolution.

In this paper, we focus our attention to commercially available and simple-to-fabricate silica-based optical fibers, which are suitable for long-length multiplexed sensing and can be readily integrated with standard telecommunications equipment. The different grating types considered in this paper have previously been shown to survive very high temperatures; however, an important factor to consider is the stability of the reflected wavelength. This ultimately dictates the temperature sensing error and thus usability of the sensors for specific applications. In this paper, we test and directly compare four different high temperature gratings, three of which are commercially available, and report on their long-term high-temperature wavelength stability. In light of our results, we discuss the potential mechanisms for drift at extreme temperatures, which includes stress-relief in the glass material, devitrification, dopant diffusion, and interaction with packaging materials.

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S. C. Warren-Smith, E. P. Schartner and H. Ebendorff-Heidepriem are with the Institute for Photonics and Advanced Sensing and the School of Physical Sciences and the ARC Centre of Excellence for Nanoscale BioPhotonics, The University of Adelaide, Adelaide, South Australia 5005, Australia (e-mail: stephen.warrensmith@adelaide.edu.au).

L. V. Nguyen and Z. Yu are with the Institute for Photonics and Advanced Sensing and the School of Physical Sciences, The University of Adelaide, Adelaide, South Australia 5005, Australia.

D. Otten and D. G. Lancaster are with the Laser Physics and Photonic Devices Laboratories, School of Engineering and Future Industries Institute, University of South Australia, Mawson Lakes, SA 5095, Australia.

II. SENSOR DESCRIPTION AND OPTICAL MEASUREMENT

Four optical fiber temperature sensors were tested, including three commercially available sensors, and one in-house fabricated sensor. In this paper we refer to the sensor by the grating and fiber type, noting that a complete sensor also consists of packaging (described below).

a. *Chiral grating in pure silica MOF (Chiral Photonics, HTS-1000, United States of America).* The sensor consisted of a 15 mm grating that was made by twisting a pure-silica MOF and passing it through a small heat zone at a temperature above the softening point of silica [18, 19]. While the pitch of the grating is the order of tens of microns, the reflection spectrum has been established as being generated through diffraction of the core fundamental mode into free space rather than as a long period grating with coupling to higher order modes [10]. While having been demonstrated as being stable at temperatures up to 900°C [18, 19], a limitation of this sensor design is that there is limited scope for multiplexing due to the broad grating response. The sensor comes sheathed in a glass capillary, with the capillary over-sheathed with a nickel-chromium-iron-molybdenum alloy capillary tube and was used as received. The sensor was manufacturer-specified to have a maximum temperature limit of 900°C and have been demonstrated as stable within 0.0005°C/hr at this temperature [19].

b. *Regenerated FBG in doped silica core SMF (Cal-Sens, Spain).* Regenerated FBGs are formed by first creating a standard UV-inscribed FBG in hydrogen-loaded doped-silica SMF. The grating is then annealed at high temperature to form a secondary regenerated grating, which generally has reflectivity in the order of 10-15 dB lower than the original seed grating [6]. The mechanism by which these gratings are formed is still debated, but current theories suggest a combination of atomic diffusion, stress-induced densification, and micro-crystallization (devitrification) at the core-cladding interface is occurring at the elevated annealing temperatures [5, 8]. The sensor comes sheathed in a ceramic tube and was used as received. The sensor was manufacturer specified to be suitable for temperature monitoring up to 1000°C.

c. *fs-written FBG in doped-silica depressed-cladding SMF (Loptek, Germany).* The sensor consisted of a fs-laser written type-II FBG in a depressed-cladding (fluorine doped) pure silica core SMF. The fiber comes coated with polyimide, sheathed in stainless steel, and was used as received. The sensor was supplied with a calibration to 400°C and was specified as being suitable for use up to 1000°C.

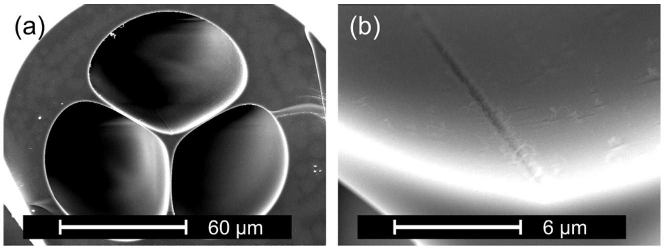

Fig. 1. Scanning electron microscope image of (a) the SCF cross-section, and (b) the femto-second laser ablation grating. Reprinted from [14].

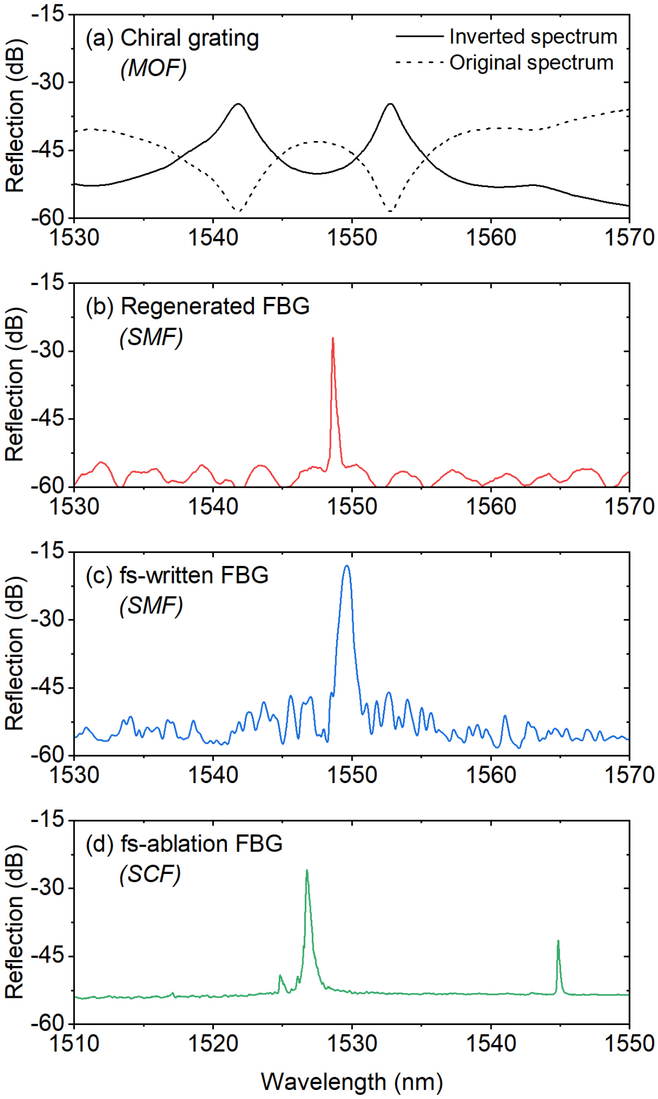

Fig. 2. Reflected spectra from (a) a chiral diffraction grating in a pure-silica MOF, (b) a regenerated FBG in a hydrogen loaded doped-silica core SMF (c) a fs-written grating in a doped-silica depressed cladding SMF, and (d) a fs-ablation grating in a pure-silica SCF. In (a) the original spectrum is shown as a solid-black line, the inverted spectrum shown as a dashed-black line was used in the measurements to be compatible with the peak tracking software used. The color of each spectrum corresponds with the results shown in Sec. 3.

d. *fs-ablation FBG in pure silica suspended-core microstructured optical fiber (SCF) (University of Adelaide, Australia).* The sensor was an in-house fabricated pure-silica SCF [Fig. 1(a)] with a fs-laser written ablation grating [Fig. 1(b)], with similar specifications as previously reported [14]. Briefly, the grating was fabricated using a wavelength-doubled (524 nm), ultra-fast laser (IMRA DE0210) with pulse duration < 250 fs. The pulse frequency was set to 100 kHz and down sampled to 1 kHz using a pulse picker. The pulse energy was approximately 100 nJ. The laser pulses were focused from a 4 mm diameter beam using a 50× microscope objective (Nikon MUE13500) onto the surface of the core of the SCF by focusing through the cladding. The fiber was then translated along its axis to produce an FBG with a pitch of 1.064 μm giving a second order FBG at 1527 nm. The fiber was then spliced to SMF28E using an arc splicer (Fujikura FSM-100P), and the distal end of the fiber sealed using the splicer to prevent contamination of the internal fiber holes. The fiber was packaged within an in-house drawn fused silica glass capillary. The capillary end was fused, again to prevent contamination of the fiber surface.

The reflection spectrum of each sensor was recorded using an optical fiber interrogator based on a swept laser source (National Instruments, PXIe-4844). Example spectra at room temperature for each sensor are shown in Fig. 2. The peak reflected wavelength of the sensors was recorded throughout the duration of the experiments. Note that the chiral grating has two spectral dips, rather than a reflected peak, thus the spectrum was inverted (shown in red) and the long wavelength peak was selected for tracking.

III. STABILITY AT HIGH TEMPERATURE

The optical fiber sensors were inserted into the centre of a tube furnace (ModuTemp) and co-located with a K-type thermocouple (Fig. 3). The furnace set-point was increased in increments of 100°C every 24 hr, with the exception of being held at 700°C for approximately 300 hr, and being held at 1000°C and 1100°C for 48 hr each in order to test stability over longer periods of time. The reflected wavelength of each grating was tracked continuously and the drift over time is shown in Fig. 4 for temperatures of 600°C and above. At temperatures below 600°C none of the sensors exhibited drift beyond the resolution of the optical sensor interrogator used (4 pm). Note that the figures show a slightly shorter duration than described above as the period where the furnace temperature was stabilizing is not shown here.

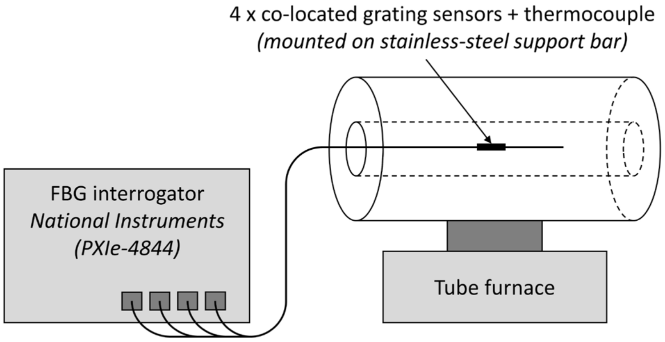

Fig. 3. Experimental setup for comparing the high temperature stability of the four sensors.

The results show that all four sensors are relatively stable up to 600°C, while at 700°C and above the two sensors based on doped fibers (b and c) undergo significant drift. Over 300 hr at 700°C the fs-laser written FBG in the depressed cladding fiber (c) experienced a net drift of approximately 0.40 nm (1.3 pm/hr) and the regenerated FBG (b) drifted by 0.15 nm (0.5 pm/hr). This corresponds to a temperature measurement error of approximately 2.4°C/day and 1.4°C/day, respectively, based on a temperature response of 13.4 pm/°C.

The two sensors based on pure silica MOFs, the chiral grating (a) and the fs-laser ablation FBG (d), show negligible drift up to 900°C. At 1000°C and 1100°C drift was observed for all sensors. However, this drift did not increase as a one-to-one function with temperature. For example, the regenerated FBG showed a greater drift at 900°C compared to 1000°C, indicating a level of annealing at the lower temperatures.

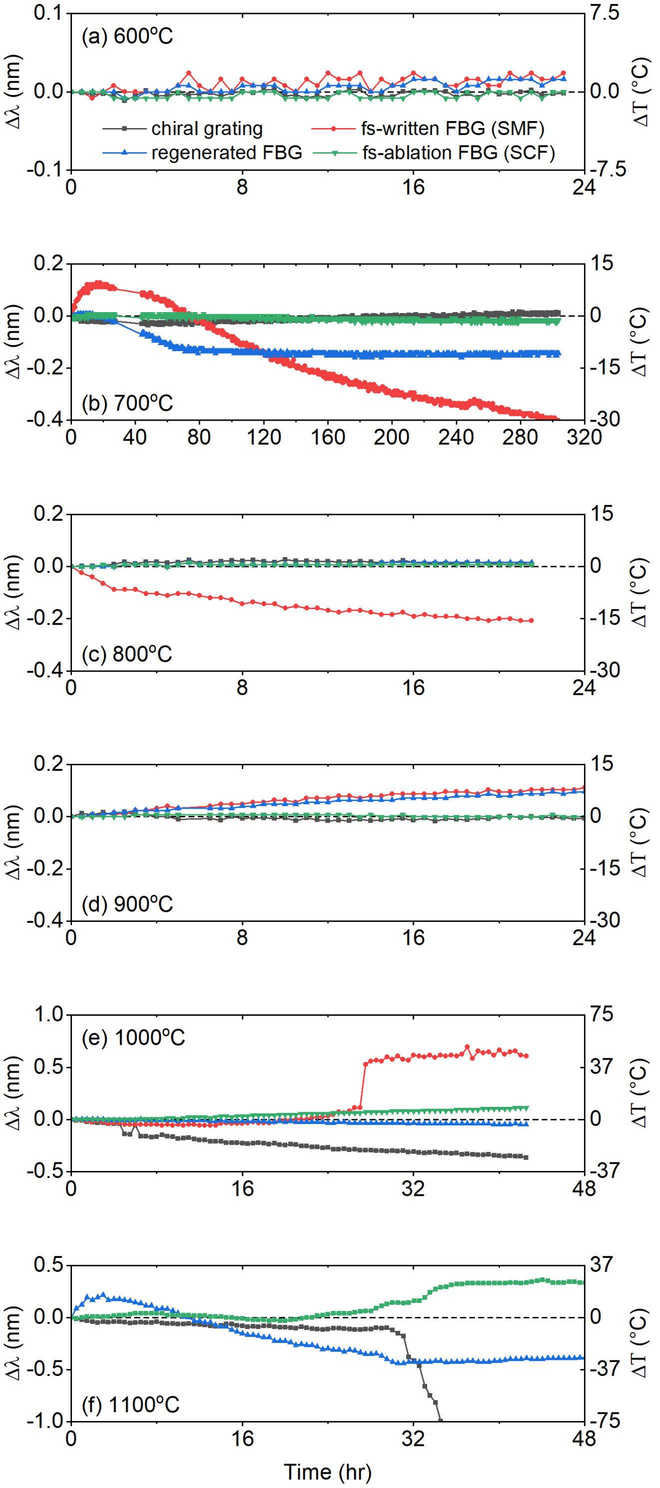

Fig. 4. Drift in the peak wavelength position of the four sensors when held at sequentially higher temperatures. Note that the sensors were held at 700°C for an extended duration of 300 hr. The secondary y-axis (ΔT) is based on the measured sensitivity of the SCF sensor from room temperature to 700°C of 13.4 pm/°C and is shown as an approximate indicator of the temperature error

While the focus of this paper is on the wavelength drift, rather than reflectivity, we note that the fs-laser written FBG within silica-core step-index fiber showed significant degradation at 1000°C, and had complete grating erasure before reaching 1100°C. The remaining gratings exhibited slow degradation in FBG reflection strength at 1100°C. Given that the maximum calibrated temperature sensing range for each supplied sensor was 900°C, 1000°C and 400°C for the chiral, regenerated and fs-laser gratings, respectively, the manufacturer specifications have been found to be generally consistent with the measured survivability and drifts measured.

These results indicate that chiral and fs-laser ablation FBGs in pure silica fiber offer good wavelength stability at high temperature. As these fibers do not rely on dopants for their wave-guiding properties they will not exhibit dopant diffusion, which is known to affect stability of optical fiber sensors at high temperature [20] or used advantageously to create thermally expanded core fibers [21]. However, at temperatures approaching the annealing point of silica it is seen that drift will begin to occur, which is likely a combination of relaxation of stresses formed during fiber drawing and grating writing, and potentially the devitrification (growth of β-cristobalite) of silica glass [22].

Devitrification of silica glass may play a prominent role in regards to the surface treatment and protection of the optical fiber sensors. Temperatures up to 1100°C are below the glass transition temperature of silica and extrapolated crystal formation rates from higher temperatures [22] predicts internal crystallization rates of vitreous silica that are negligible. However, this is under the condition of a contamination free surface. Surface contaminants can considerably accelerate crystallization [23] and are a well-known cause of fiber fragility [24]. This suggests the importance of packaging for optical fiber high temperature sensors, both in terms of ensuring a clean surface during packaging and preventing further contamination after packaging. We observe that the two sensors that demonstrated the best stability in this study, the chiral grating-based sensor and the SCF sensor, were packaged in silica glass capillary. This provides a cleaner and smoother surface due to being fire-polished when drawn, and will not shed particles as can be the case for ceramics or oxidized stainless steel at high temperature. The silica capillary also does not have a thermal expansion mismatch with the optical fiber.

TABLE I
DIFFERENCE IN FBG REFLECTION WAVELENGTH ACROSS TWO CONSECUTIVE ANNEALING CYCLES

FBG	$\Delta\lambda_1$ (pm)	$\Delta\lambda_2$ (pm)	ΔT_1[a] (°C)	ΔT_2[a] (°C)
1	156	32	8.9	1.8
2	116	8	6.6	0.5
3	156	28	8.9	1.6
4	296	52	16.9	3.0

[a] The temperature difference values (ΔT) are based on the average measured sensitivity of the annealed SCF sensors in the following experiment (Fig. 3) from 1000°C to 1100°C (17.5 pm/°C) and is for a guide only.

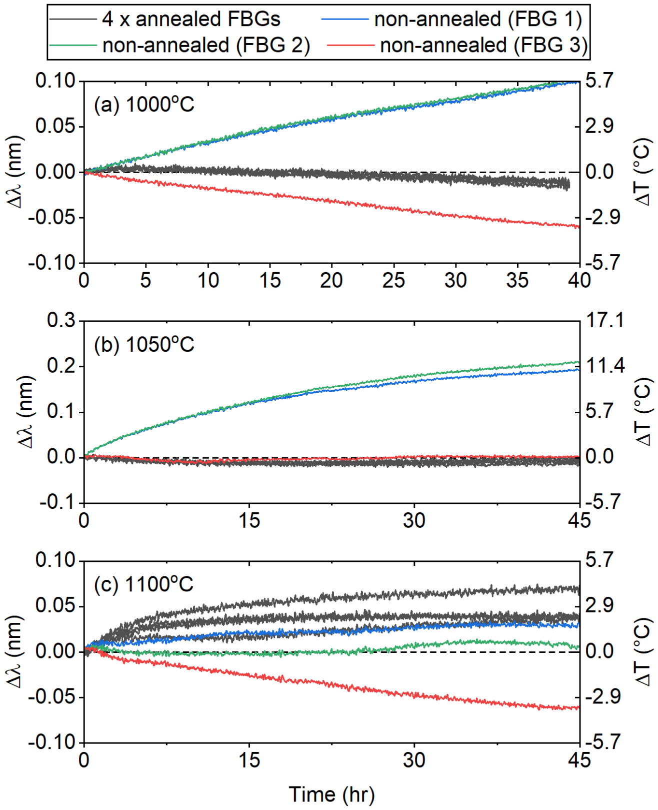

Fig. 5. Two-day comparison at 1000°C, 1050°C and 1100°C after SCFs with fs-laser ablation FBGs were annealed compared to three that were non-annealed. The secondary y-axis (ΔT) is based on the average measured sensitivity of the annealed SCF sensors from 1000°C to 1100°C (17.5 pm/°C) and is for a guide only. Note that the scale on the y-axis in (b) is different to (a) and (c) to accommodate the larger drift values.

IV. ANNEALING OF SILICA SCFS

We prepared seven new nominally-identical SCFs with fs-laser ablation FBGs to investigate the cause of drift at higher temperatures (≥ 1000 °C), in particular whether this drift is primarily due to stress, which can be removed via annealing, or crystallization, which cannot. These gratings were prepared with similar specifications as those used in the test described in Sec. 3, but with a grating length of 2 mm instead of 10 mm and a room-temperature reflection wavelength of 1550 nm. The shorter grating length was used due to improvements found in the FBG reproducibility when writing shorter lengths, as the fiber is effectively straighter over the shorter length, and is not expected to have different thermal properties compared to the longer 10 mm gratings.

The sensors were packaged as described above in silica capillaries and placed into a tube furnace (Across International, TF1700) with the gratings at the centre position of the furnace and the temperature referenced with an R-type thermocouple. Four of the fibers were annealed by twice raising the temperature above the strain point of fused silica and holding for four hours, followed by cooling to 1000°C at 1°C/min and then passively cooling to room temperature (≤ 10°C/min). The remaining three sensors were not annealed. The difference in grating wavelength position after each annealing step compared to before each annealing step, Δλ, is shown in Table 1 and shows significantly reduced, though non-zero, drift over the second annealing step. Increased annealing duration time, or repeated annealing cycles, is expected to further reduce the drift in FBG reflection wavelength, as has been shown for other microstructured optical fiber temperature sensing configurations [25].

The FBGs were then heated consecutively to temperatures of 1000°C, 1050°C and 1100°C, and held for 48 hours each. Note that the annealed and non-annealed sensors were tested in independent trials, but with the same experimental conditions. The resulting drift recorded in the FBG reflection wavelength is shown in Fig. 5. Note that the time duration shows less than 48 hours, as time taken to stabilize the furnace has not been included.

The results of Fig. 5 show that thermal annealing above the strain point (1125°C for fused silica) is an effective strategy for improving the thermal stability of fs-laser ablation FBGs in pure silica SCFs. In particular, the annealed sensors (4 × grey curves in Fig. 5) exhibit significantly less variability in the drift compared to the non-annealed sensors (3 × colored curves in Fig. 5). Noting, however, that the non-annealed sensors showed improved stability at 1100°C compared to 1050°C, likely due to a degree of thermal stabilization that occurred while being previously held at 1000°C and 1050°C for 48 hr each.

Drifts no greater than 1 pm/hr were measured following thermal annealing, with average drifts of 0.36 pm/hr, 0.15 pm/hr and 1.0 pm/hr recorded at temperatures of 1000°C, 1050°C, and 1100°C, respectively. The average sensitivity of the gratings to temperature over the range of 1000°C to 1100°C was measured to be 17.5 pm/°C, thus the average drift values correspond to 0.5°C/day, 0.2°C/day and 1.4°C/day at temperatures of 1000°C, 1050°C, and 1100°C, respectively.

At 1100°C a small decay in the grating reflection strength was also measured, at approximately 0.1 dB/day. Detailed analysis of the mechanism for grating decay, particularly at higher temperatures, is beyond the scope of this study and is the subject of continued investigation.

The thermal annealing allows the stresses in the glass induced by fiber drawing and fs-laser writing to be relaxed. It is known that fs-laser ablation results in a two-component modification. The physical defects (ablation holes) caused by multi-photon ionization are a permanent modification, but this is surrounded by a region of increased density due to thermal changes [26, 27]. Thermal annealing likely removes stresses related with the latter, forming a thermally stable index-modulation based only on irreversible removal of material.

V. CONCLUSIONS

Four grating-based optical fiber temperature sensors, including three commercially available sensors, were tested at high temperature for stability of the reflected wavelength. Two sensors were based on doped silica fibers, which showed stability up to 600°C, with measurable drift at 700°C and above. The two sensors that were based on pure-silica MOFs showed good stability up to 900°C.

The in-house fabricated SCF with fs-laser ablation FBGs offers high temperature stability equivalent to a sensor drift of less than 0.5°C/day up to 1050°C after annealing. This is higher than comparable thermocouple sensors, such as 1.5°C drift over 1000 hr at 1000°C using type K thermocouples (0.036°C/day) [28], However, the potential for multiplexing of sensing points along a single fiber means that FBG high temperature sensors are likely to play an important role in applications requiring a high density of measurement points.

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Stephen C. Warren-Smith completed his PhD in 2011 at the University of Adelaide, Australia, on the topic of microstructured optical fiber chemical sensing. He was then employed from 2011 to 2014 as an Australian Research Council (ARC) Super Science Fellow at the Institute for Photonics and Advanced Sensing and the School of Chemistry and Physics at the University of Adelaide, working on optical fiber bio-sensing for women's health applications. In 2015-16 he worked as a European Union Marie Curie International Incoming Fellow at the Leibniz Institute of Photonic Technology, Jena, Germany, on a project investigating the micro/nano-structuring of optical fibers for sensing. Since October 2016 he is with the University of Adelaide as a Ramsay Fellow.

Erik P. Schartner completed his PhD in 2011 on a project entitled "Hydrogen peroxide sensing with microstructured optical fibers: fuel, wine and babies." Since the completion of his PhD, Erik has worked on an industry linked project with Cook Medical, on the development of novel optical fiber probes for measurements of pH and temperature in the local medium surrounding embryos, and following that as a researcher within the Centre for Nanoscale Biophotonics looking at developing fiber sensors for biomedical applications. He is currently working as a Research Fellow in the Institute for Photonics and Advanced Sensing, and the Centre for Nanoscale Biophotonics. His current research projects involve application of fiber sensors to ultra-high temperature industrial applications, and the deployment of novel optical fiber sensors in bio-applications.

Linh V. Nguyen obtained a PhD in Information and Communications from Gwangju Institute of Science and Technology (GIST), South Korea, in 2009. His graduate works focused on the development of fiber-optics devices for applications in fiber sensing, fiber lasers and all-optical signal processing. In 2010 he moved to Edith Cowan University (ECU) in Western Australia to work on the development of fiber-optics sensors for applications in desalination and seawater-related industries. He joined the Institute for Photonics & Advanced Sensing (IPAS), School of Physical Sciences at the University of Adelaide in 2011 as an Australian Research Council (ARC) Super Science Fellow leading a project toward development of rapid protein sensing and blood typing platform at crime scenes and subsequently on a project developing tools for in-field surveillance of pathogens in a Researcher/Chief Investigator capacity. Currently he is working on the development of densely multiplexed high temperature sensors for industrial applications.

Dale E. Otten is currently a part-time Research Fellow within the Laser Physics and Photonics Devices Laboratory (LPPDL) of the University of South Australia (UniSA), and is also CEO of the laser manufacturer Red Chip Photonics (RCP). His career is characterized by many transitions between academia and industry. After obtaining a BSc. (Hons) in Chemistry from the University of Adelaide (UoA) in 2002, he then spent several years in industry developing ion selective electrodes for analytical instruments in Seattle, WA, USA. In 2010 he obtained his PhD at the University of California, Berkeley, in the ultrafast nonlinear spectroscopy of liquid-vapor interfaces. He then spent 6 years at Coherent Scientific in Adelaide, Australia successfully servicing the research laser market in both a business development and technical capacity. In 2016 Dale joined RCP as its CEO and is actively commercializing the outputs of the LPPDL in this role, in tandem with is position at UniSA.

Zheng Yu completed both a B. Eng with first class honours in Mechanical Engineering and M. Sc in Optics and Lasers at the University of Adelaide, Australia. He has worked in the fields of medical optics and lasers for over 10 years. Since May 2017 he has been employed at the University of Adelaide as a research officer in the Institute for Photonics and Advanced Sensing.

David G. Lancaster has established and co-leads the Laser Physics and Photonics Devices Laboratories group at the University of South Australia. The laboratory's research areas include short to mid-infrared waveguide lasers; ultra-fast laser processing of optical materials; optical fiber based sensors; integrated photonic devices; and advanced photonic manufacturing. David completed a Ph.D. in experimental laser physics at Macquarie University (1997), followed by 3 years as a Postdoctoral Fellow at Rice University developing mid-infrared spectroscopic instruments for trace gas sensing. In 2000 he joined the Australian Defence Science and Technology Organisation as a senior research scientist where he focused on short to mid-infrared lasers for application within Electronic Warfare Systems. David is currently Professor of Laser Engineering at the University of South Australia, a founder and

CTO of Red Chip Photonics, and has published over 60 journal papers and several patents.

Heike Ebendorff-Heidepriem received the Ph.D. degree in chemistry from the University of Jena, Germany, in 1994. She received the Weyl International Glass Science Award and the prestigious Marie Curie Individual Fellowship of the European Union in 2001. During 2001-2004 she was with the Optoelectronics Research Centre at the University of Southampton, UK. Since 2005, she has been with the University of Adelaide, Australia. Currently, she leads the Fibres and Photonics Materials research group and is the Deputy Director of the Institute for Photonics and Advanced Sensing. She is also the Deputy Director of the Optofab node of the Australian National Fabrication Facility (ANFF) and Senior Investigator of the ARC Centre of Excellence for Nanoscale BioPhotonics (CNBP). Her research focuses on the development of novel optical glasses, specialty optical fibers, hybrid glasses and fibers, surface functionalization and sensing approaches.