Investigation of the long-term stability of arc-induced gratings heat treated at high temperatures

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A long-period grating written in the SMF-28 fibre was heat treated at 1000 °C for 15 days. The spectrum of the grating shifted to longer wavelengths and the resonances depth decreased as a result of structural relaxation. The background loss increased considerably for times longer than 200 h, and this loss is caused by devitrification of the fibre.

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1. Introduction

High temperature sensors are needed in a wide spectrum of technological areas from classical metallurgy to modern green energy production (production of hydrogen through thermolysis), aeronautics (in aircrafts turbines), and spacecraft industry. Fibre optic sensors with their inherent characteristics such as electromagnetic immunity, low weight and volume and the possibility of multi-physical parameter interrogation of spatial distributed gratings offer competitive advantages over the conventional ones [1]. In this context, the development of high temperature sensors based on fibre gratings is of great interest. Long-period fibre gratings (LPFGs) produced by the electric arc technique have already demonstrated good stability at high temperatures during 24 h annealing [2]. In this paper, we assess the effect of heat treatment on arc-induced gratings at 1000 °C for two weeks.

2. Experimental results

A LPFG was arc-induced in the Corning SMF-28 fibre using the following set of fabrication parameters: an axial tension of 5.1 g, 40 arc discharges with an electric current of 9 mA and 1 s duration each and a grating period of 540 μm. The fibre with the grating written on it was then placed inside an 18 cm long tubular oven (a weight of 2.2 g was used to prevent bending) and was heated up to 1000 °C, at a heating rate of ~5 °C/min. It is known that, for this fibre, stress relaxation takes place for temperatures above 600 °C and, therefore, the fibre was first heated continuously up to 500 °C and then for each step of 100 °C the grating was kept for 10 min at that temperature to achieve thermal equilibrium. The grating remained at 1000 °C during 15 days, and its spectrum was recorded periodically by using a white light source and an optical spectrum analyzer with a resolution of 1 nm.

The evolution of the grating spectrum during the long-term heat treatment is shown in Fig. 1. Fig. 1a shows its evolution during the first 20 h of annealing. As can be seen, the spectrum moves towards longer wavelengths, the resonance depth decreases as a result of structural relaxation. The background loss increased considerably for times longer than 200 h, and this loss is caused by devitrification of the fibre.
Note, however, that the LP$_{11}$ mode was initially over-coupled (the product of the coupling constant at the wavelength of the resonance centre and the grating length was larger than $\pi/2$) while the LP$_{12}$ and LP$_{13}$ modes became over-coupled during the first 20 h of annealing [4]. In Fig. 1b it is observed an overall increase in the background loss that is specifically related to scattering caused by the devitrification process (the process of crystallization in a formerly amorphous glass) [5]. It is also observed the evolution of the transmission spectrum to an irregular wavelength dependence, its origin is not fully understood, as will be discussed below. It is known that during annealing in air at high temperatures, the hydroxide anion OH$^-\,$ is absorbed from water vapour leading to a reduction of the glass viscosity, to an increase in the OH$^-\,$ band near 1.4 $\mu$m (see, for instance, Fig. 4), and it also enhances crystallization [6]. Another factor that contributes to crystallization is oxygen loading that also occurs during annealing in air at 1000 °C, and it saturates in about 200 h [7]. It should be noted that after 219 h the background loss increases at a rate of ~1.7 dB/day, which is an order of magnitude higher than the value obtained during the first 200 h. For the last 100 h of the heat treatment, the identification of the cladding modes was only possible due to the consecutive data points shown in Fig. 2.

Fig. 2 shows the time dependence of the resonance wavelengths and the corresponding resonance depth for three cladding modes during annealing for two weeks. All the resonances shift to longer wavelengths as a result of structural relaxation, however at about 285 h the LP$_{12}$ and LP$_{13}$ resonances start moving in the opposite direction (Fig. 2a). We suppose that after 200 h of heating [8], the glass structure has already approached equilibrium and therefore the subsequent shift to shorter wavelengths can be due to crystallization [9] or diffusion [10]. First, during prolonged annealing at such high temperature, silica based glass changes to a more organized structure such as cristobalite. This process is initiated at the surface of the fibre due to dust, flaws or scratches and advances towards the fibre core [11]. The crystallization rate increases in the presence of water and oxygen [5]. On the other hand, dopants such as germania also accelerate the crystallization since it decreases the viscosity and the point defects in the glass structure may act as nucleation centres leading to crystallization starting at the core and evolving to the cladding region. In our particular case, we do not know which process (in the core or in the cladding) is more important. During the crystallization process, the refractive index of the glass increases[12]. Second, for heating temperatures and times discussed in this study, the diffusion of the core dopant would lead to an increase of the core radius ($R_{co}$) by more than 1 $\mu$m [10] and to a decrease of the core-cladding refractive index difference ($\Delta n_{co-cl}$). The increase in the core radius shifts the resonances to longer wavelengths while the decrease in the core-cladding refractive index difference shifts the resonances in the opposite direction. The combined effect results in the observed blue shift of the resonance wavelengths as was confirmed by
computer simulations using the Apollo v2.2 software (Fig. 3). For this simulation, we assumed that the formation of the grating is due to the core-shift mechanism and that during diffusion the refractive index profile follows a “top-hat” shape which is valid for the beginning of the diffusion process (changes of the order of 1%), that is, \( \Delta n_{\text{diff}} = (R_0 / R_{\text{fib}})^2 \times \Delta R_{\text{co-cl}} \) where \( R_{\text{fib}} \) and \( \Delta R_{\text{co-cl}} \) are respectively, the new core radius and the new core-cladding index difference during the diffusion process. The initial values of \( R_0 = 4.18 \mu m \) and \( \Delta R_{\text{co-cl}} = 4.85 \times 10^{-3} \) (at 1550 nm), which are close to the parameters of the standard fibre, have been obtained by taking into account the effect that the arc discharge has on the fibre properties [13] and they have been used to fit the spectrum in Ref. [3]. The computer simulation also indicates that diffusion contributes to an increase of the coupling strength. This may be explained by an increase of the overlap integral as a result of an increase of the core radius, since the core-cladding refractive index decreases. Therefore, the observed decrease of the resonances depth in Fig. 2b during the heat treatment can only be explained by the fact that all the resonances are over-coupled. Note that the resonances depth shown in Fig. 2b was obtained after subtracting the background loss.

In order to separate the effects of the heat treatment on the fibre and on the grating, an 18 cm long piece of fibre without grating was annealed at 1000 °C for 33 h. As can be seen in Fig. 4, despite a faster degradation of the fibre that may be due to different humidity conditions, the overall behaviour is similar to that observed in Fig. 1b: an increase in the background loss and in the OH− band near 1.4 μm and also the appearance of the irregular oscillation pattern with an average wavelength separation between the dips of about 10 nm (which is approximately one half of the value obtained in Fig. 1b). Therefore, in what concerns the apparent irregular pattern, it cannot be attributed to a cavity interference effect resulting from the 18 cm long fibre heat treated in the oven since that would result in a wavelength separation of the order of 4 pm. Furthermore, since it also appears in the fibre without grating it cannot be related to coupling between different core modes and the cladding modes as a result of core dopant diffusion (the fibre becomes multimode). We believe also that the irregular oscillations pattern in the transmission spectrum is not related to temperature fluctuation inside the oven, because this pattern appears only after tenths of hours of treatment at stabilized temperature and grows continuously with time. On the other hand, the crystals form an irregular structure that can be considered as an irregular grating. If the reflection surfaces form a Fabry–Perot interferometer then \( \Delta \lambda = \lambda^2 / 2 R_{\text{fib}} L \). Indeed, the wavelength separation points towards “obstacles” of the order of tens of micrometers (for \( L = 40 \mu m \), \( \Delta \lambda = 20 \) nm), such as crystals formation. However at this stage, it is not clear the origins of such apparent interference pattern.

3. Conclusions

We presented results on the long-term heat treatment (15 days) of arc-induced gratings at 1000 °C. It was observed a shift of the grating spectrum to longer wavelengths and a decrease of the resonances depth as a result of structural relaxations. Following 200 h of treatment, it was found an increase of the background loss in the transmission spectrum and the appearance of the irregular wavelength dependence, which may be due to crystallization. This investigation showed that the long-term use of transmission gratings induced in silica based fibres is compromised by devitrification. Therefore, despite the possibility of using some techniques that may slow down this degradation process (for example by inserting the fibre inside a sealed silica capillary), the implementation of high temperature optical fibre sensors requires more stable materials such as sapphire. Nevertheless, it should be emphasized that these sensors can be continuously used, with a proper heat treatment, at temperatures up to 800 °C and for short time periods at temperatures of the order of 1000 °C.

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