Devitrification in Annealed Optical Fiber

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Abstract— **The decrease in transmittance of annealed optical fiber has been measured versus temperature and time. The annealing loss is due to the devitrification of the glass and OH absorption in the 1200 to 1500 nm wavelength region. Both loss mechanisms propagate primarily from the surface into the core. However, to increase the OH absorption significantly, annealing times greater than 10 h are required. Fibers heated from 1000 to 1300 °C in an air atmosphere quickly devitrify and their transmittance approaches zero. Also, the current sensitivity of annealed fiber current sensors versus annealing time at 850 °C has been measured. A decrease in the current sensitivity is attributed to devitrification in the fiber.**

I. Introduction

In this paper, the effects of devitrification in annealed optical fibers are briefly discussed. Thermally processing optical fiber produces many useful components: splices, couplers, thermally expanded core fiber, fiber lenses, chirped Bragg gratings, and sensors that measure current, voltage, temperature, and strain [1]–[6]. However, the heat treatment can, under certain conditions, devitrify the glass fiber, i.e., nucleate and grow crystals within the glass matrix [7]–[10].

Devitrification in silica glass can occur through a nucleation and growth process at temperatures between about 200 and 800 °C, depending on the glass composition [11]. (Typical optical fiber with 4 mole % of GeO_2 in SiO_2, may have a higher nucleation temperature.) Crystal growth proceeds from the nuclei present for temperatures near 800 °C to a few hundred degrees Celsius below the melting point, which is about 1800 °C in fibers [11]–[13]. If the temperature of the glass remains low, nucleation will occur at a negligible rate because the nucleation and crystallization rates are exponential functions of temperature [10], [14]. However, further thermal processing into the crystal growth temperature range will promote growth of nuclei [8]–[14]. The phase cristobalite is the normal product of devitrification in silica glass [8], [13].

There are several ways to reduce devitrification in thermally processed fibers. One method is to rapidly heat and cool the fiber through the nucleation temperatures, minimizing the time to nucleate crystallization sites. Another is to heat the fiber near melting and cool rapidly. However, both of these methods produce thermally induced stress in the glass and, if the glass is cooled rapidly enough, a quenched state. Additives can inhibit (for example gallium oxide in borosilicate glass) or promote (for example titanium dioxide in alumina-silicate glass) crystal growth [10], [11], [15]. Devitrification can start at the glass-air surface and proceed inward. This can be due to the presence of surface flaws or to chemical reaction [8], [10], [16], [17]. Argon can be used to suppress surface reactions with oxygen, nitrogen, carbon dioxide, and water [1], [8], [16], [18], [19].

To produce fiber coils suitable for current sensing, all stress-induced birefringence must be removed. Coils annealed at 850 °C will be nearly isotropic, but require a slow cooling rate, approximately 0.2 °C/min [4]. The heating rate used is limited to about 5°C/min. These rates are slow enough to allow a significant amount of nucleation to occur during the annealing cycle. The maximum temperature, 850 °C, is high enough to promote crystal growth and slowly devitrify the fiber. Also, the fiber annealing is done in an atmosphere of ambient air where water, oxygen, carbon dioxide, and nitrogen can react with the glass surface to promote crystal growth [8], [11], [15], [16]. However, at this temperature the crystal growth rate is slow, and annealing times of several hours can be used without significantly affecting the performance of a current sensor [4], [8], [10], [20]–[22].

II. Experimental Measurements of Annealed Optical Fiber

A. Optical Fiber Current Sensors

1) Current Sensitivity: With advances in optical fiber annealing, current sensors with nearly zero linear birefringence (nearly perfect current response) can be made [23], [24]. This advance in annealing allows a systematic look at the current sensitivity of a given fiber versus annealing time. Fig. 1 shows the current sensitivity of coils, 50.9 turns on a diameter of 7 cm, versus the annealing time at 850 °C. The fiber used was single mode at 1300 nm, 125 μm diameter, step index fiber with a numerical aperture of 0.13. The fiber cladding is SiO_2 and the nominally 8 μm core is about 4 mole % doped GeO_2: SiO_2 glass. The fiber jacket is treated with acetone for a few minutes before the annealing and is burned off [4]. Annealing takes about four days. Current sensitivity is measured polarimetrically using a difference-over-sum method [4]. With a known current, the sensitivity is compared to theory to obtain a relative sensitivity [4]. Fig. 1 shows that as the annealing time increases the current sensitivity decreases linearly at a rate of about 0.02%/h. This decrease in current sensitivity can be due to several effects individually or in combination: a depolarization of the light in the fiber, a change in the dispersion $(dn/d\lambda)$ of the fiber, or an increase in the average linear birefringence.

The primary effect causing the sensitivity change seems to be scattering from crystal sites. Scattering would depolarize the light (or lower the degree of polarization) in the fiber

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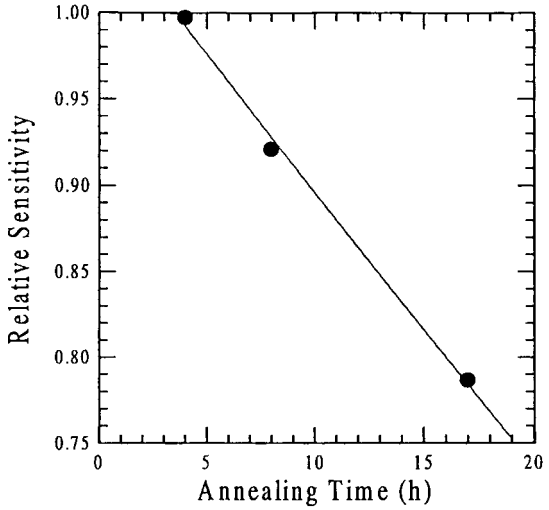

Fig. 1. Relative sensitivity of 50.9 turn, 7 cm diameter coils annealed at 850 °C for 4, 8, and 17 h.

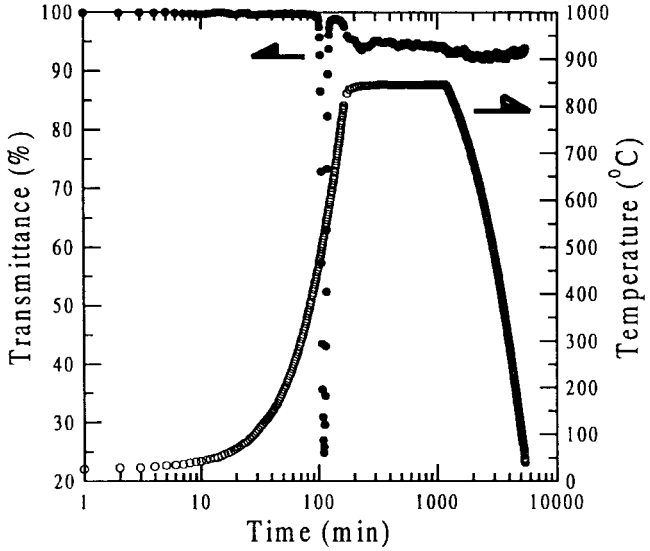

Fig. 2. The transmittance and thermal history of a 100.9 turn coil annealed at 850 °C for 17 h. The ∘'s mark the temperature and the •'s mark the transmittance.

and reduce the measurable effect of the Faraday rotation [25]. The degree of polarization was measure for the coils shown in Fig. 1 and the degree of polarization change, about 25%, would account for the change in sensitivity. Also, as discussed later, these annealed fibers have a significant amount of light scattered from the core.

The magnitude of the Faraday effect could be changed if $dn/d\lambda$ of the fiber in the 1300 nm wavelength region changed due to OH absorption [26]. In diamagnetic materials the magnitude of the Faraday effect is directly proportional to the glass dispersion. A 20% change in the dispersion would correspond to the change in current sensitivity seen in Fig. 1. However, for these modest annealing times the OH absorption does not increase enough to change the current sensitivity through $dn/d\lambda$. On the other hand, OH absorption does play a role in the loss of these annealed fiber coils.

Residual linear birefringence in the annealed fiber will reduce the measured Faraday rotation [4], [24], [25]. There are only two mechanisms to increase the linear birefringence in annealed fiber: deformation of the core and cladding [27], [28], and possibly devitrification. (Stress-induced birefringence and micro-bending birefringence is removed during anneal.) The core of the fiber waveguide will deform at high temperatures, above 1200 °C, producing birefringence [27]. At 850 °C the fiber will not deform in this way, because the fiber viscosity is high enough to hold the fiber's shape. Also, the measured birefringence of these coils does not increase with annealing time. Devitrification could increase the average linear birefringence of the fiber as the glass becomes polycrystalline. When the glass is cooled the index difference between the vitreous glass state and the micro-crystals present will produce a birefringence. The orientation of the birefringence axis will vary from crystal site to crystal site; however, with enough crystal growth the average birefringence could rise to affect the fiber coil's sensitivity. This distribution of birefringence, if present, could be measured with polarization mode dispersion (PMD) techniques [29]. I have measured the PMD of a coil, about 20 m long, annealed for over 100 h at 906 °C. No significant PMD increase could be measured in this annealed fiber coil. From this measurement, residual birefringence, possibly due to devitrification, does not appear to be the mechanism for reduced current sensitivity in these sensors.

2. Optical Loss: Devitrification in glass fiber can increase the optical loss of a coil when light is scattered from crystal sites [10]. Additional loss in Nd-doped fiber, bulk glass, and fluoride fiber has been attributed to devitrification or scattering from crystal sites [30]–[33]. Also, loss can come from OH absorption and possibly micro-bending or stress-induced waveguide loss. In some cases the loss in the fiber may be due to a combination of these effects. However, in the case of stress-induced loss or microbending, the waveguide is not severely deformed, all stress is removed from the glass during annealing, and the polycrystalline structures on the surface of the glass are small.

To measure the change in loss, the optical throughput was monitored as a coil was annealed. Fig. 2 shows a plot of the transmittance at 1287 nm for a 100.9 turn coil on a diameter of 7 cm annealed at 850 °C for about 17 h. Fig. 2 also has the temperature history for comparison. At temperatures between 500 and 600 °C the polymer jacket burns into a carbon ash. As the jacket swells on burning, it stresses the wound fiber coil, generating temporary microbending losses. If the coil is wound with too much tension the fiber will break during this portion of the annealing cycle. As the jacket burns into ash the stress is relieved and the transmittance returns to near preannealing conditions. Thus, micro-bending loss is not a dominate factor in this fiber. (Further visual inspection of the fiber surface after annealing shows only minor flaws.) However, as the fiber temperature reaches 800 °C the transmittance drops by about 7%. All annealed coils show some drop in transmittance as the temperature reaches 800 °C. Fig. 3 shows increased optical loss, in dB/km, due to annealing for this fiber type versus annealing time at 850 °C. The loss increases linearly with annealing time at a rate of about 0.41±0.05 dB/(km•h).

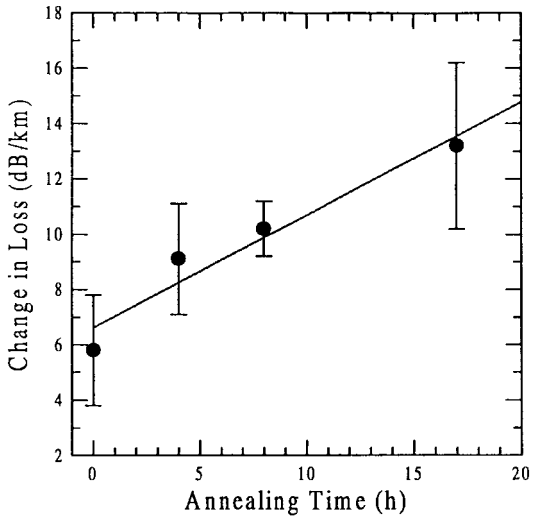

Fig. 3. Annealing-induced loss for 100.9 turn, 7 cm diameter coils annealed at 850 °C for 0.1, 4, 8, and 17 h.

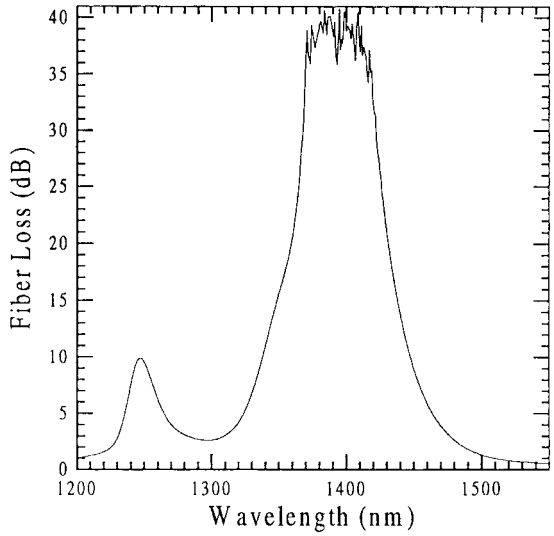

Fig. 4. Spectral loss of a 100.9 turn, 7 cm diameter coil annealed at 906 °C for over 100 h.

Annealing significantly increases the loss in this fiber from 0.34 dB/km to about 9 dB/km for modest annealing times.

The loss in these annealed fibers at 1300 nm can also be attributed to OH absorption. OH enters the fiber through a reaction with water from the air and hydrogen in the glass [34]–[37]. For annealed fiber, in an air atmosphere, the main reaction is with water. (The hydrogen concentration in this fiber is assumed low at high temperatures.) The glass-water reaction has been established as: $(H_2O)_{vapor} + (\equiv Si–O–Si\equiv)_{glass} \rightarrow 2(\equiv Si–OH)_{glass}$ [34]. The spectral loss of our fiber has been measured over the 1200–1550 nm range, and an increased absorption due to OH is measurable after annealing for 17 h. The loss at our laser wavelength, 1287 nm, is about 6 dB/km for a 17 h annealing and about half the measured loss in Fig. 3 for the same annealing time. To show OH absorption in an annealed fiber a coil about 20 m in length was heated for more than 100 h at 906 °C. The strong absorption spectra is shown in Fig. 4.

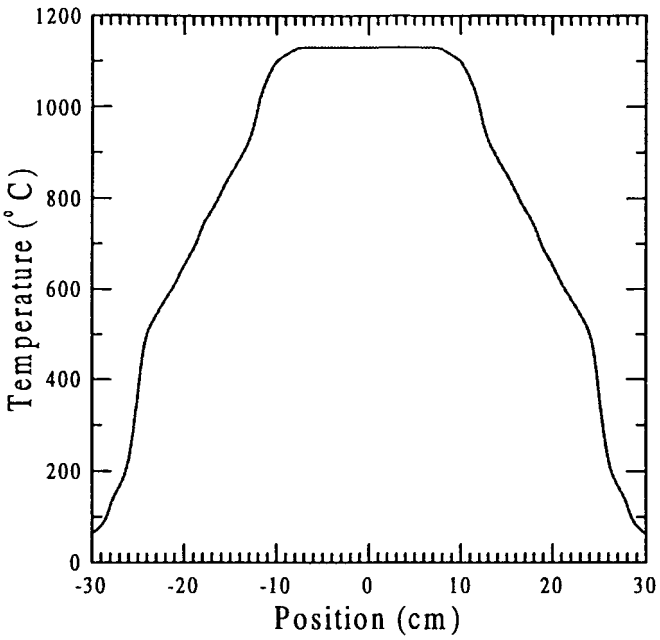

Fig. 5. Temperature profile of the tube furnace used for high temperature annealing.

The loss in fibers annealed at 850 °C appears to be dominated by OH absorption in the 1200 to 1500 nm wavelength region for annealing times greater than 10 h. At shorter times the OH loss is too small to measure with confidence. However, the loss due to scattering cannot be discounted because the coils radiate enough light to be viewed with an infrared viewer and localized radiation points can be seen in the fiber. At short annealing times, less than 10 h at 850 °C, the largest part of the optical loss appears to come from scattering. Microbending loss does not appear to significantly contribute to the loss of this fiber after the jacket is burned away, as seen in Fig. 2.

B. Optical Fiber Crystallization

To explore the devitrification of annealed fibers further, a high temperature tube furnace was constructed to anneal at temperatures from 850 to 1300 °C. To match the previous annealing cycle, the heating rate of the oven was set to 5 °C/min. The cooling rate was increased to 20 °C/min, to decrease the cycle time to about 16 h. Fig. 5 shows a typical temperature profile of the oven with a hot zone of about 19 cm. A fused quartz tube about 60 cm long with a 7 mm I.D. was used to anneal fibers at high temperatures. Fused quartz rings were used to suspend the fiber in the tube to avoid any anomalous diffusion between the tube and fiber at the higher temperatures. After each annealing cycle the fiber was examined and photographed under a microscope. The optical spectra of these fibers was taken to measure any OH absorption loss, however, no loss could be measured for the short annealing time used.

Figs. 6, 7, 9, and 11 show photographs of the surface of a fiber for annealing temperatures from 850 to about 1300 °C for 8 h. Fig. 6 shows a fiber annealed at 850 °C to compare with the annealed fiber coils discussed above. Only minor surface defects are present. (Care was used in handling the fiber to

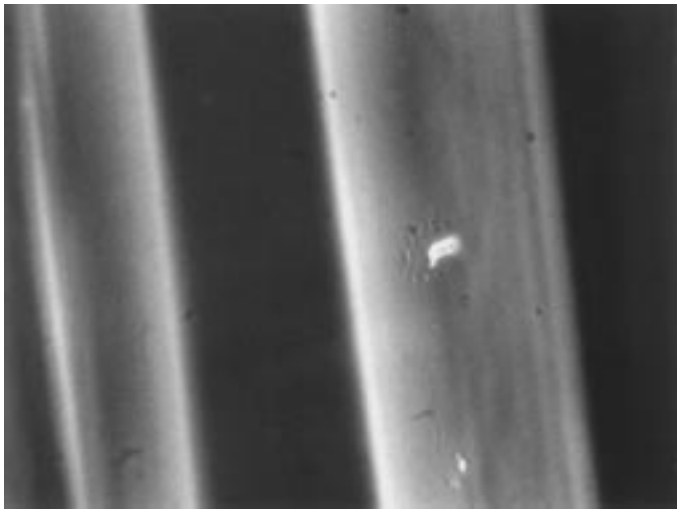

Fig. 6. 125-μm fiber annealed at about 850 °C for 8 h.

avoid scratching or other damage before photographing.) With further mechanical and environmental exposure, the formation shown in the figure will ultimately lead to a crack propagating into the fiber. However, with proper packaging, the fiber annealed at 850 °C can be preserved through many thermal cycles (−65 to +125 °C) and vibration (greater than 20 g or greater than 196 m/s^2) [38].

Fig. 7 shows a photograph of the surface of a fiber annealed at about 1100 °C. The surface of the glass is beginning to undergo a phase change as microcrystals cover significant portions of the surface. Fig. 8 shows the transmittance of the fiber and thermal history of the annealing cycle for the fiber shown in Fig. 7. The fiber transmittance falls with time as the fiber is held at about 1100 °C. This loss in transmittance can be attributed to the devitrification of the core glass, scattering light out of the fiber and possibly due to stress-induced loss from the crystal growth on the surface. (The spectra of fiber annealed at about 1040 and 1220 °C for 8 h showed no measurable increase in OH absorption.) When the oven starts to cool rapidly, the transmittance registers the thermal shock. This fiber's transmittance recovers to near its value before cooling. In other tests, the transmittance has been both lower and higher after the fiber cools. A fiber with this surface is slightly weaker than the fiber annealed at 850 °C due to surface defects.

Fig. 9 shows a photograph of the surface of a fiber annealed at about 1220°C. The fiber surface after annealing is fracturing as polycrystalline structures form. Fig. 10 shows the transmittance and thermal history of the annealing cycle. Again, the fiber transmittance falls with time as the fiber is held at about 1220 °C. As the oven cools rapidly, the different thermal expansions of polycrystalline cristobalite and glass may further fracture the fiber and increase the stress-induced loss to reduce the fiber transmittance [39]. This fiber is fragile and difficult to handle without breaking. Under mechanical movement the surface cracks quickly propagate through the fiber. The birefringence of fiber heated above 1220 °C increases due to waveguide deformation [27]. However, this would not contribute to a measurable loss in the short length of fiber used here.

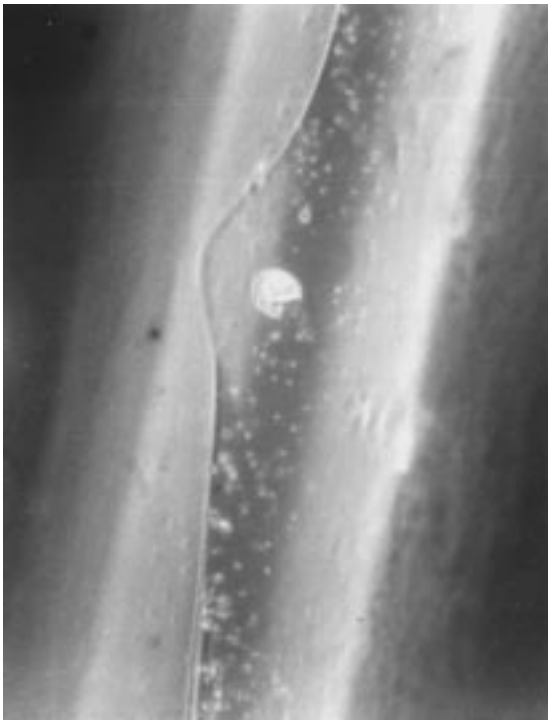

Fig. 7. 125-μm fiber annealed at about 1100 °C for 8 h.

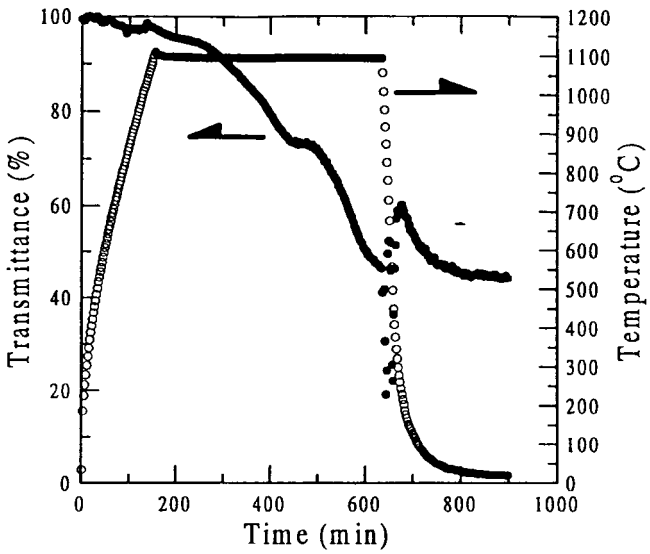

Fig. 8. The transmittance and thermal history of a fiber annealed at about 1100 °C for 8 h. The ∘'s mark the temperature and the •'s mark the transmittance.

Fig. 11 shows a photograph of the surface of a fiber annealed at about 1310 °C. The fiber surface after annealing is completely fractured by polycrystalline structures. Fig. 12 shows the transmittance and thermal history of the fiber during the annealing. As the temperature approaches 1300 °C the fiber no longer transmits. This is attributed to the nearly complete devitrification of the glass fiber, which breaks as a crystal fracture plane propagates through the fiber. Again, this fiber is extremely fragile. The fiber is no longer translucent and has a white appearance under normal laboratory lighting.

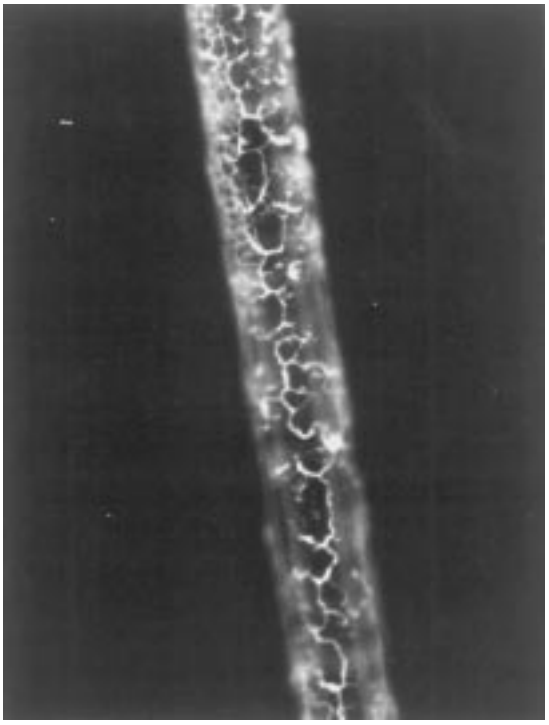

Fig. 9. 125 μm fiber annealed at about 1220 °C for 8 h.

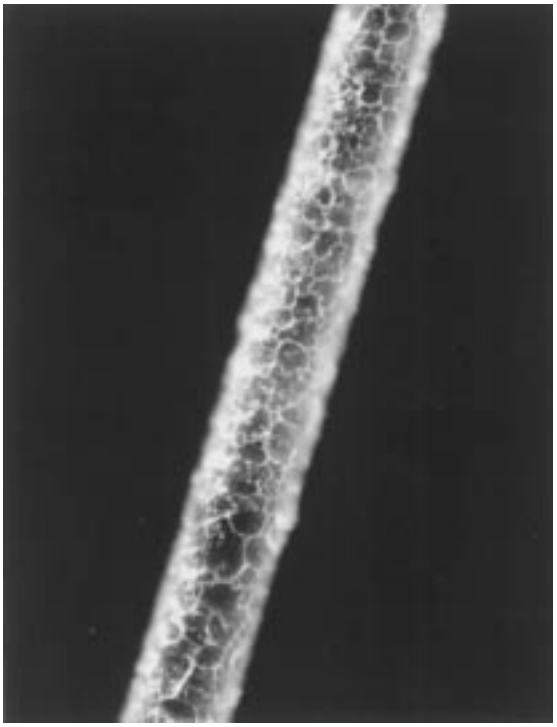

Fig. 11. 125 μm fiber annealed at about 1310 °C for 8 h.

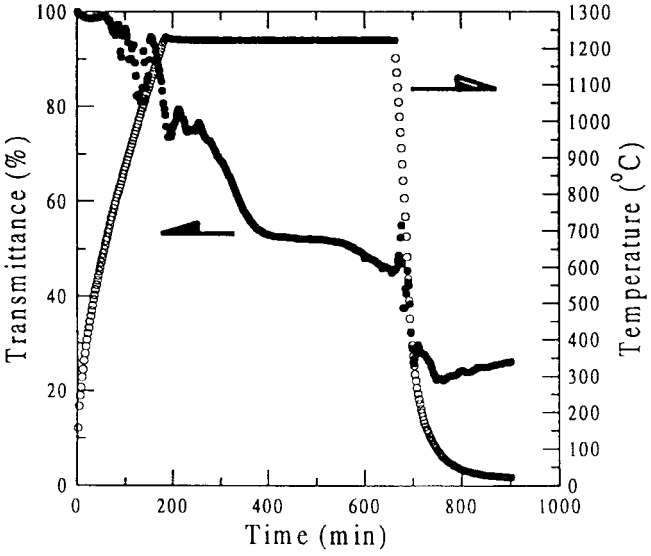

Fig. 10. The transmittance and thermal history of a fiber annealed at about 1220 °C for 8 h. The ∘'s mark the temperature and the •'s mark the transmittance.

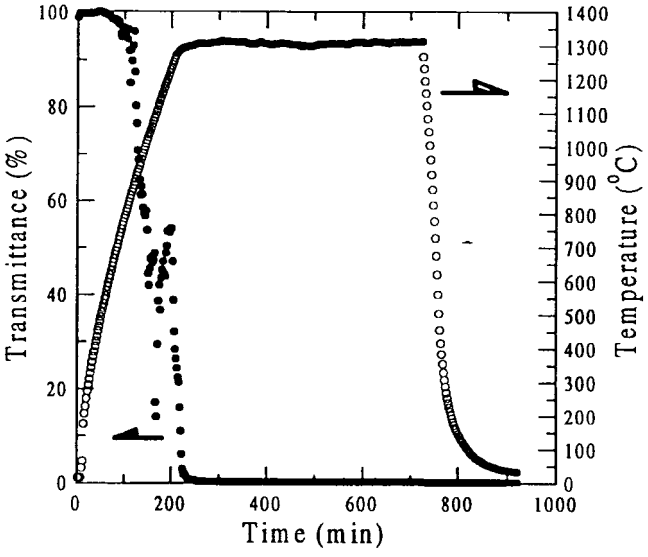

Fig. 12. The transmittance and thermal history of a fiber annealed at about 1310 °C for 8 h. The ∘'s mark the temperature and the •'s mark the transmittance.

Fig. 13 shows the transmittance change for many fibers annealed 8 h at temperatures from 850 to about 1300 °C. At annealing temperatures above 1000 °C, the loss due to devitrification significantly increases. This may be due to the temperature reaching the crystallization activation energy of the glass [10], [20]. The linear and spline fits are presented in Fig. 13 to aid in seeing the transition around 1000 °C and to note that the transmittance change in the fiber is linear with increasing temperature above 1000 °C. At these short annealing times OH absorption does not measurably contribute to the fiber's optical loss.

III. Summary and Conclusions

The current sensitivity and optical loss of annealed fiber coils are linearly dependent on the time of annealing at 850 °C. For optical fiber current sensing the most important fiber parameter is the elimination of linear birefringence [4], [23], [24]. Depolarization from scattering is a secondary consideration for optimum current sensitivity [25]. The optical loss in a fiber current sensor has much greater bounds than depolarization and linear birefringence. Many fiber current sensors can have up to 10 dB of loss before optical power budgets and photodetector noise limit performance. Therefore,

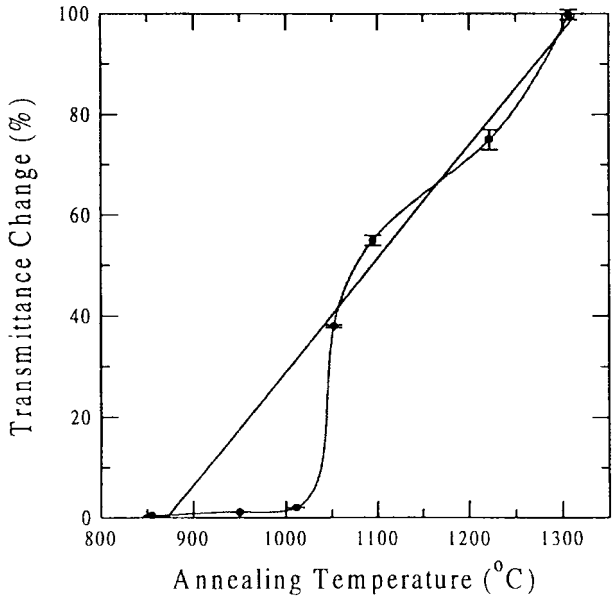

Fig. 13. The transmittance change for fibers annealed 8 h at different temperatures.

when optical fiber is annealed, the maximum temperature and time must be tailored to remove all linear birefringence and avoid significant depolarization. As discussed devitrification appears to effect a current sensor's response primarily from depolarization effects.

The loss in annealed fibers at 1300 nm has possibly three causes, scattering, absorption, and stress-induced. Scattering comes from the index difference between a crystal site and the vitreous glass. Absorption in the 1200 to 1500 nm range comes from OH. The stress-induced losses can arise when the surface of the fiber is severely damaged by crystal growth. At annealing temperatures, around 850 °C, the optical loss of the fiber is due to both OH absorption and scattering. Stress-induced loss is not considered a major source, because the surface is still visibly well preserved. OH and scattering loss is small, about 0.2 dB for a 20 m coil annealed at 850 °C for 4 h. For longer annealing times the optical loss is dominated by OH absorption.

For higher annealing temperatures above 1000 °C and times less than 10 h, the loss in the fiber is due to scattering, fiber fracture, and possibly stress-induced loss from surface crystal growth. At these high temperatures the optical loss and fiber fragility increase, making current sensing with this type of fiber impractical. Annealing at temperatures above 1000 °C promotes crystal growth starting primarily at the surface and propagating into the fiber to the core. This result agrees with the comments of Matusita and Sakka that nucleation and crystal growth will propagate from the surface inward for heating rates greater than 2 °C/min (Our heating rate was 5 °C/min.) [21].

Crystal growth at the surface is greatly affected by the treatment and type of jacket on the annealed fiber. As the jacket burns, the surface of the glass is altered to promote crystal growth through chemical attack [16]. Any residue left on the surface will also serve as nucleation sites, accelerating devitrification. Therefore, for fiber that endures fire or other high-temperature processing, the jacket material becomes critical to the fiber survivability and loss. From the data presented here, sensors that use silica fiber above 1000 °C are not practical for long term use.

The effects of optical fiber devitrification will limit the performance and lifetime of silica fiber sensors that operate at temperatures above 800 °C. Even at relatively low temperatures, OH absorption in the 1200–1500 nm wavelength band will alter the calibration of intensity-based sensors.

The method of monitoring the transmittance of the fiber during heat treatment could complement other methods of monitoring crystal growth in optical fiber for annealing times less than 10 h [10]. For annealing times greater than 10 h the loss measurement will be complicated by OH absorption in the 1200 to 1500 nm range. Longer annealing times could be used if wavelengths outside of the OH absorption bands were used.

For Verdet constant determinations in annealed optical fiber, the effects of depolarization must be considered as it directly effects the measured Faraday rotation. Also, Verdet constant measurements in the 1200–1500 nm region require annealing times less than 10 h, because OH absorption changes the dispersion of the fiber. (The Verdet constant in diamagnetic materials is directly proportional to $dn/d\lambda$ [26].)

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A. H. Rose, for a biography, see this issue, p. 807.