

Effects of sterilization methods on key properties of specialty optical fibers used in medical devices

Andrei A. Stolov*, Brian E. Slyman, David T. Burgess, Adam S. Hokansson, Jie Li and R. Steve Allen

OFS, Specialty Photonics Division, 55 Darling Drive, Avon, CT 06001

ABSTRACT

Optical fibers with different types of polymer coatings were exposed to three sterilization conditions: multiple autoclaving, treatment with ethylene oxide and treatment with gamma rays. Effects of different sterilization techniques on key optical and mechanical properties of the fibers are reported. The primary attention is given to behavior of the coatings in harsh sterilization environments. The following four coating/buffer types were investigated: (i) dual acrylate, (ii) polyimide, (iii) silicone/PEEK and (iv) fluoroacrylate hard cladding/ETFE.

Keywords: Optical fiber, sterilization, autoclave, ethylene oxide, gamma radiation, polyimide coating, silicone coating, PEEK, polymer cladding

1. INTRODUCTION

Optical fibers are successfully used in various areas of medicine, including urology, general surgery, ophthalmology, cardiology, endoscopy, dentistry and medical sensing.¹⁻⁴ Prior to use inside a human body the fiber must be sterilized to ensure it is free of microorganisms such as fungi, bacteria, and virus or spore forms. Sterilization can generally be defined as any process that effectively kills or eliminates all microorganisms from a surface, contained in a fluid, equipment, food, medication or biological culture medium.⁵ Many types of physical or chemical treatments are known as effective sterilization techniques. Roughly, the methodologies can be subdivided into three groups: (i) use of elevated temperatures, (ii) chemical treatment, and (iii) exposure to radiation. The first group includes flaming, exposures to dry heat and hot steam (autoclaving) and boiling in water. Chemicals such as ethylene oxide (EtO), formaldehyde, ozone, hydrogen peroxide, phthalaldehyde and peracetic acid in the gas phase and/or solutions are used for chemical sterilization. Finally, microorganisms can be effectively killed by UV light, X-rays, gamma- and e-beam radiation.

Generally speaking, sterilization is a “harsh” process that may represent a challenge to the performance of treated objects. Thus, exposure of optical fibers to harsh conditions may significantly affect their properties.^{6,7} Ideally, sterilization of optical fibers should be such that it eliminates all the microorganisms but does not affect their optical attenuation and mechanical strength. It should be noted however, that no systematic study of possible effects of sterilization on optical fiber properties has been reported thus far.

In this work we investigate effects of different sterilization methods on performance of several optical fibers designed for medical applications. Three common sterilization methods were selected: (i) steam sterilization (autoclaving), (ii) treatment with ethylene oxide and (iii) gamma radiation.

Each of the selected sterilization methods has certain advantages and disadvantages. Steam sterilization is widely used because of its short processing time, non-toxicity and safety. On the other hand, items sensitive to heat and moisture cannot be sterilized by this method. EtO sterilization is preferable for

* stolov@ofsoptics.com; phone 1 860 678-6629; fax 1 860 674-8818; www.specialtyphotonics.com

materials that are sensitive to heat. However, EtO gas is toxic, carcinogenic and explosive. Another disadvantage of using EtO is relatively long sterilization and ventilation times. Gamma radiation is a cold method with which very high sterility assurance levels can be achieved. It produces minimal waste byproducts and does not require quarantine for outgassing. It has been known, however that this technique can lead to significant alterations in the materials being treated. High-energy radiation produces ionization and excitation of polymer molecules which may result in crosslinking and/or chain scission.

Of particular interest were effects of sterilization on specialty fibers employed with different polymer coatings. In this paper we investigated fibers with the following coatings: dual acrylate, hard polymer clad, silicone, and polyimide. The results of our study are reported herein.

2. FIBER DESIGN

All fibers selected for the study were OFS Fitel (OFS) products. The fibers had 200 μm silica glass cores (Table 2.1). Four of the five fibers had 220 μm doped silica claddings with a numerical aperture (NA) of 0.22. The fifth fiber (200/HCS/ETFE) used an HCS[®] fluoroacrylate polymer cladding (NA = 0.37). It is important to note that in 220/HCS/ETFE and 200/HCS/ETFE fibers, HCS[®] fluoropolymer plays roles of a cladding and a coating simultaneously. Thus, 220/HCS/ETFE fiber comprises two consecutive claddings with numerical apertures of 0.22 and 0.37, respectively. The coatings on the rest of the fibers were dual acrylate, polyimide and silicone. The refractive index of the silicone material used in Silicone/PEEK fiber is such that it also guides light as the secondary cladding with NA of 0.37.

Three fibers were further up-buffered with either poly(ethylene tetrafluoroethylene) (ETFE) or polyether ether ketone (PEEK). The coating and buffer dimensions are given in Table 2.1.

Table 2.1. Fibers selected for the study

Fiber ID	Core OD (μm)	Glass cladding OD (μm)	Coating Material	Coating OD (μm)	Buffer material	Buffer OD (μm)
Acrylate	200	220	Dual acrylate	500	-	-
Polyimide	200	220	Polyimide	250	-	-
Silicone/PEEK	200	220	Silicone	350	PEEK	600
220/HCS/ETFE	200	220	HCS [®]	250	ETFE	400
200/HCS/ETFE	200	-	HCS [®]	230	ETFE	500

3. STERILIZATION PROCEDURES AND TEST TECHNIQUES

Loose 500-meter coils about 12'' in diameter were prepared from each fiber. Prior to sterilization exposures, the fiber attenuation and strength was determined as described below.

The autoclaving treatment was performed by MycoScience, Inc. using a Consolidated Sterilizer, model SSR-3A-ADVPB. The samples were exposed to total twenty gravity autoclave cycles. Each cycle consisted of 8 minutes at 132°C and 30 Psi. After every 5 cycles were completed, three meters of each sample were cut and removed from the coil. Those 3-meter samples were further tested for their possible strength degradation.

EtO sterilization was performed by Geotec, Inc. The fibers were packaged in standard Tyvek pouches. One side of each bag was made of 1073B Tyvek while the other side was a polyester/polyethylene laminate. The bags were placed in a hermetic chamber which was evacuated prior to filling with 100% EtO. The gas temperature and pressure were 60°C and 0.5 atmosphere, respectively. From start to finish the sterilization treatment took 7.5 hours.

Gamma radiation sterilization was performed by Steris Isomedix Services. The fibers were packaged in Tyvek pouches, same as being used for EtO treatment. The pouches were placed in a chamber where they were exposed to radiation of a ^{60}Co source. The radiation power was measured via an Alanine Pellet Dosimeter. The exposure time was 331 minutes. The overall radiation dose was in the range 40 – 50 kGy.

The fiber attenuation was evaluated by two independent approaches. Optical Time-Domain Reflectometry (OTDR) measurements were performed at 850 nm using a PK 8000 Production and Laboratory OTDR instrument. In addition, spectral attenuation in the region 600 – 1100 nm was determined using a custom-made spectral bench.

The OTDR approach is insensitive to quality of the fiber end faces, so it is an accurate way of evaluating the fiber attenuation at a single wavelength. In its turn, spectral bench provides important information on chemical changes in the fiber core and the cladding.

The fiber strength was evaluated using two-point bend technique. The tests were performed with a Fiber Sigma 2 Point Bend Apparatus.⁸ All strength testing was conducted at controlled humidity and temperature in accordance with a Telcordia GR-20 condition ($\text{RH} = 50 \pm 5\%$, $T = 23 \pm 2^\circ\text{C}$).⁹ The samples were kept at least 12 hours at this condition before the testing, which is also required by the GR-20 standard.⁹ Most of the tests were performed at a strain rate of 4%/min, and the median fracture stress, σ_m , was determined from the data. In addition, the dynamic fatigue parameter, n_d , was evaluated for the as-drawn and autoclaved fibers. For this, the testing was performed at strain rates of 0.08, 0.57, 4 and 28%/min using the same two-point bend technique. The number of data points taken for the aforementioned strain rates are 5, 7, 20 and 15, respectively.

Chemical changes in coating and buffer materials were analyzed using Fourier-Transform Infrared spectroscopy (FTIR). The spectra were collected using a Nexus 670 spectrometer employed with a slide-on micro-attenuated total reflection (ATR) accessory. A germanium internal reflection element was utilized. With this setup, the IR beam probed about 1 μm layer of the analyzed samples (i.e., coatings and buffers).

4. RESULTS AND DISCUSSION

4.1. Effects of Autoclaving Cycles on Fiber Strength and Fatigue Parameters

The glass fiber strength can be described well by Weibull statistics.¹⁰ This approach uses two parameters for characterizing the strength: the median strength value (equivalent to the median fracture stress, σ_m) and the Weibull slope, m . The latter parameter is a measure of the variability in the strength and is inversely proportional to the standard deviation. A broad distribution of strength (and hence a low Weibull slope) may indicate an out-of-control process of fiber manufacturing or a damage developed during the service.

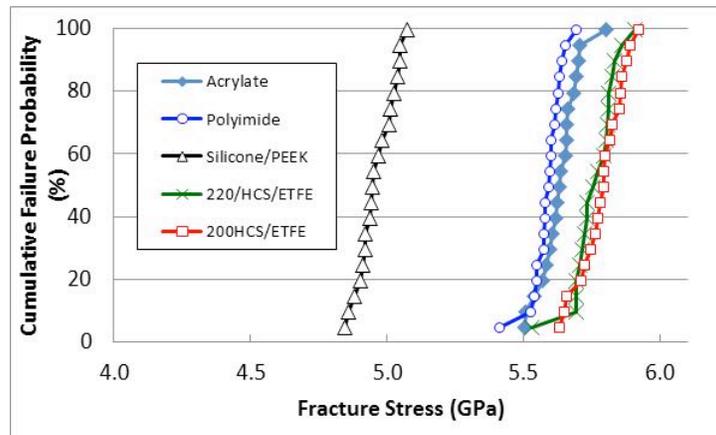


Figure 4.1.1. Weibull plots for “as-drawn” fibers determined via the two-point bend approach at 4%/min strain rate.

Figure 4.1.1 shows the two-point bend test results obtained for the “as drawn fibers”. The strength of the silicone-coated fiber is around 5.0 GPa while the rest of the fibers exhibit the strength magnitude around 5.6 GPa.

It is known that if stress is applied to silica-based optical fibers, their strength becomes time-dependent due to crack growth that is enhanced by moisture. The degradation of fiber over time is known as fatigue and is characterized by the stress corrosion parameter (n_d). Higher values of n_d correspond to lower rates of crack growth, i.e. to higher mechanical reliability of the optical fiber.¹¹ The data on median strength, Weibull slopes and n_d values are summarized in Table 4.1.1.

Table 4.1.1. Strength and fatigue parameters of optical fibers before and after sterilization.

Fiber ID	As drawn			Autoclaved			EtO		Gamma	
	σ_m (GPa)	m	n_d	σ_m (GPa)	m	n_d	σ_m (GPa)	m	σ_m (GPa)	m
Acrylate	5.61	91	30.7	5.61	103	25.1	5.55	82	5.52	64
Polyimide	5.58	109	25.1	5.6	49	25.6	5.59	121	5.43	48
Silicone/PEEK	4.94	87	19.4	4.91	67	21.2	4.87	74	5.03	84
220/HCS/ETFE	5.73	80	28.9	5.87	31	27.5	5.63	123	1.27	5
200/HCS/ETFE	5.77	83	28.1	5.79	112	27.3	5.69	142	1.99	8

In an autoclave, the fibers are exposed to relatively high temperature and highly concentrated water vapor. This combination may cause cracking of polymer coatings and/or deterioration of the glass cladding surface. Both factors may result in strength degradation of the fiber, which changes are typically cumulative.¹² In most medical applications, the maximum number of autoclave cycles does not exceed 20, so in our study the maximum number of cycles was selected to be 20.

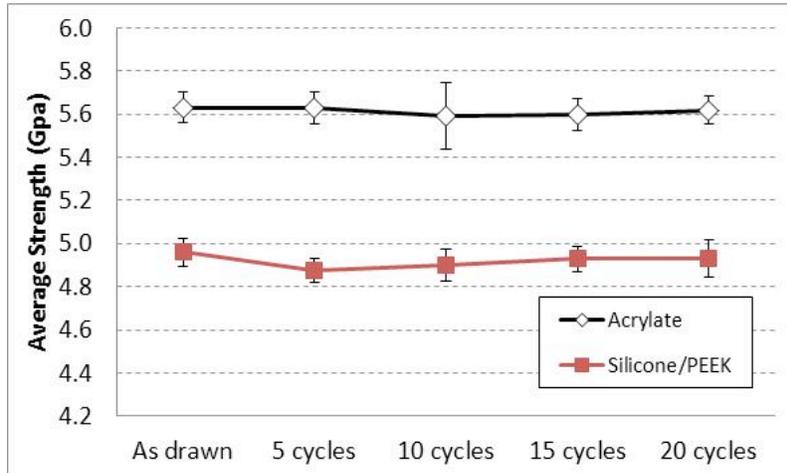


Figure 4.1.2. Strength of Acrylate and Silicone/PEEK fibers exposed to consecutive autoclaving cycles. The error bars correspond to the standard deviation.

After every 5 cycles were completed, three meters of each sample were removed from the fiber coils and tested for strength. Figure 4.1.2 shows the results obtained for two of the studied fibers. Notwithstanding the difference in the coating chemistry, the strength of Acrylate and Silicone/ETFE fibers was found not to be affected by autoclaving, at least within the 20 consecutive cycles. The same conclusion can be made for the strength of fibers with polyimide and HCS/ETFE coatings (Table 4.1.1).

In addition to the fiber median strength, we also determined the stress corrosion parameter for the fibers exposed to autoclaving. The obtained results are displayed in Figure 4.1.3. The error bars correspond to 95%-confidence limits for the n_d values. The only statistically significant change was observed for the

Acrylate fiber, where n_d value decreased from 30.7 ± 1.1 to 25.1 ± 1.1 . For the rest of the fibers, the n_d values did not change upon the autoclaving within the measurement accuracy.

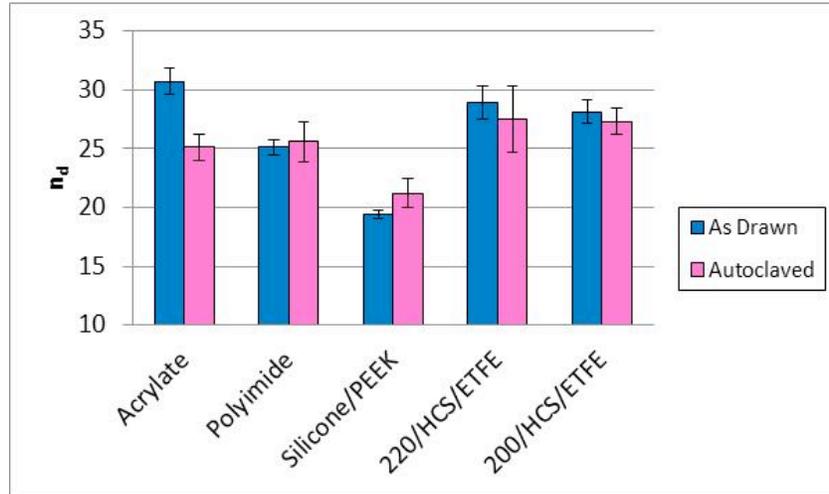


Figure 4.1.3. n_d values for as drawn and autoclaved (20 cycles) fibers.

Thus, a gentle reduction of n_d value observed for the acrylate-coated fiber is the only revealed effect of autoclaving on the fiber mechanical properties.

4.2. Effects of EtO and Gamma Radiation on Fiber Strength

EtO treatment conditions applied in this study (0.5 atmospheres of 100% EtO, 60°C, 7.5 hours dwell time) were same as commonly used for various medical devices. It can be seen from Figure 4.2.1 and Table 4.1.1 that exposure to ethylene oxide did not produce any effects on the fiber strength. It follows that EtO molecules did not cause any chemical degradation of the silica and polymer coatings.

Gamma radiation dose applied in this study (40 – 50 kGy) was typical for medical sterilization. It is known that 25 kGy dose is 40% above the minimum to kill the most resistant microorganisms.¹³ Whereas a minimum dose of 25 kGy is desired for microbial control, the actual applied dose is often in the 25 – 50 kGy range. It can be seen from Figure 4.2.1 and Table 4.1.1 that exposure to gamma radiation lead to a significant strength degradation of fibers with HCS[®]/ETFE coating/buffer layers. At the same time, mechanical strength of fibers with acrylate, polyimide and silicone/PEEK coatings was not affected.

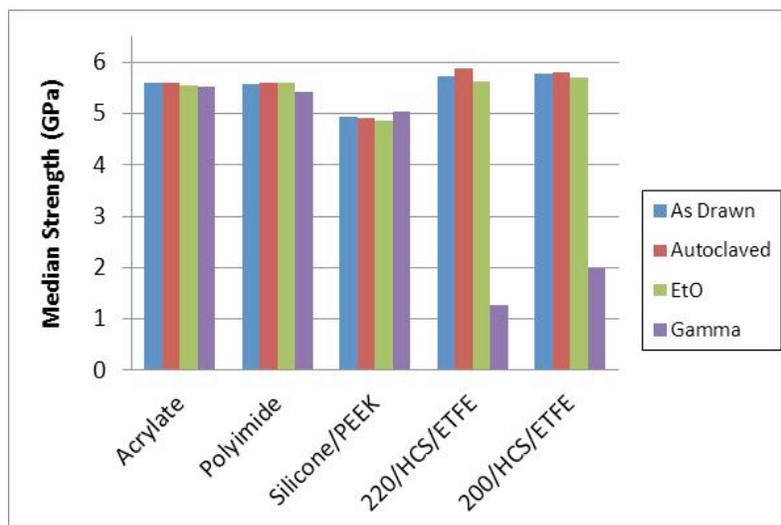


Figure 4.2.1. Effects of sterilization conditions on median strength (σ_m) of optical fibers.

Gamma radiation normally affects polymers in two basic manners, both resulting from excitation or ionization of atoms. The two mechanisms are chain scission, a random rupturing of bonds, which reduces the molecular weight (i.e., strength) of the polymer, and cross-linking of polymer molecules, which results in the formation of large three-dimensional molecular networks. For polymers with carbon-carbon chains (backbones), it has been observed that cross-linking generally will occur if the carbons have one or more hydrogen atoms attached, whereas scission occurs at tetra-substituted carbons. Polymers containing aromatic molecules generally are much more resistant to radiation degradation than are aliphatic polymers; this is true whether or not the aromatic group is directly in the chain backbone or not.¹⁴

Since the strength degradation was observed only for the fibers with HCS[®] coating up-buffered with ETFE, it was obvious that the issue is due to specifics of the coating or buffer chemistry. It should be noted that both polymer materials are the only fluorinated compounds. Moreover the HCS[®] material contains a perfluorinated monomer. It is known that fluorine-containing polymers, like polytetrafluoroethylene are highly sensitive to gamma radiation ($\sim 10^3$ times more sensitive than polystyrene or polyimide).¹⁴ It seems most likely that interactions with gamma radiation generate hydrofluoric acid (HF) as one of the reaction products. HF can easily diffuse through the coating and deteriorate the glass surface which results in significant strength degradation.

Generally speaking, chemical changes in the materials can be followed by FTIR spectroscopy. Figure 4.2.2 displays FTIR spectra of HCS[®] coating of the as-drawn and sterilized fibers. The spectra look almost identical, indicating that gamma radiation did not cause major changes in the coating chemistry. Spectra of ETFE, PEEK, silicone, polyimide and acrylates (primary and secondary) also did not show any significant effects of any sterilization technique. Thus it should be concluded that gamma radiation caused very minor changes in polymer chemistry of HCS[®] and/or ETFE which are undetectable by FTIR but fatal for the fibers. Obviously, since the observed strength degradation is related to the fluorine content in the coating/buffer layers, similar behavior is expected for other types of commercially available fluorinated polymer-coated fibers.

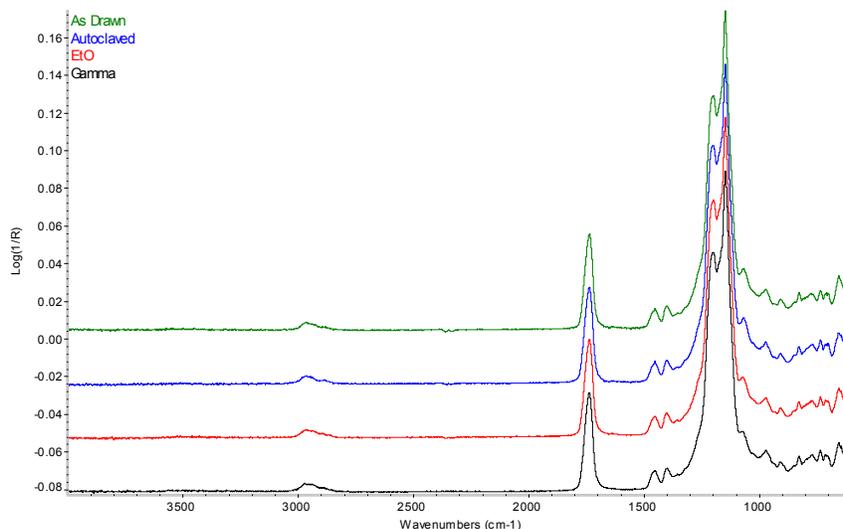


Figure 4.2.2. FTIR spectra of HCS[®] coating before and after sterilization. The spectra are shown with an arbitrary offset.

4.3. Effects of Sterilization on Fiber Attenuation

Table 4.3.1 summarizes the 850 nm attenuation data obtained using OTDR and spectral bench. Figure 4.3.1 displays the data obtained by OTDR only. In a nutshell, the results can be interpreted as

follows: (i) EtO treatment did not affect the attenuation at 850 nm; (ii) multiple autoclaving cycles caused slight changes for Silicone/PEEK and 200/HCS/ETFE fibers only; (iii) in contrast, gamma radiation affected the fiber attenuation significantly. Furthermore, we were not able to register optical signal transmitted through 220/HCS/ETFE and 200/HCS/ETFE fibers after they were treated by gamma radiation. We assume that these fibers became so weak after being exposed to gamma rays that further handling lead to multiple disruptions throughout the fiber length.

Table 4.3.1. Attenuation at 850 nm as determined by OTDR and spectral bench.

Fiber ID	As drawn		Autoclaved		EtO		Gamma	
	OTDR	Bench	OTDR	Bench	OTDR	Bench	OTDR	Bench
Acrylate	2.36	3.37	2.42	2.76	2.01	3.12	20.1	57.1
Polyimide	2.04	2.76	2.19	2.69	2.02	1.93	22.0	24.1
Silicone/PEEK	2.69	2.49	3.48	3.39	2.10	2.57	19.1	19.5
220/HCS/ETFE	2.69	3.31	2.34	2.86	2.51	2.34	No signal	
200/HCS/ETFE	4.09	3.98	6.34	6.61	4.03	4.71	No signal	

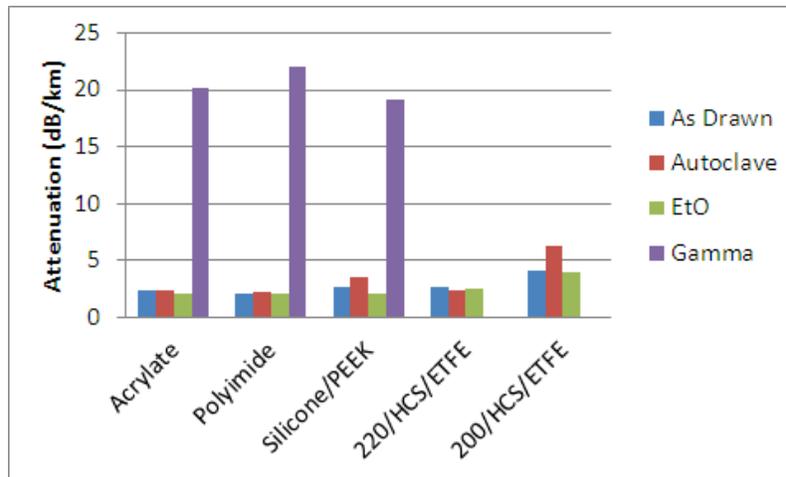


Figure 4.3.1. Attenuation at 850 nm (OTDR data) for as-drawn and sterilized optical fibers.

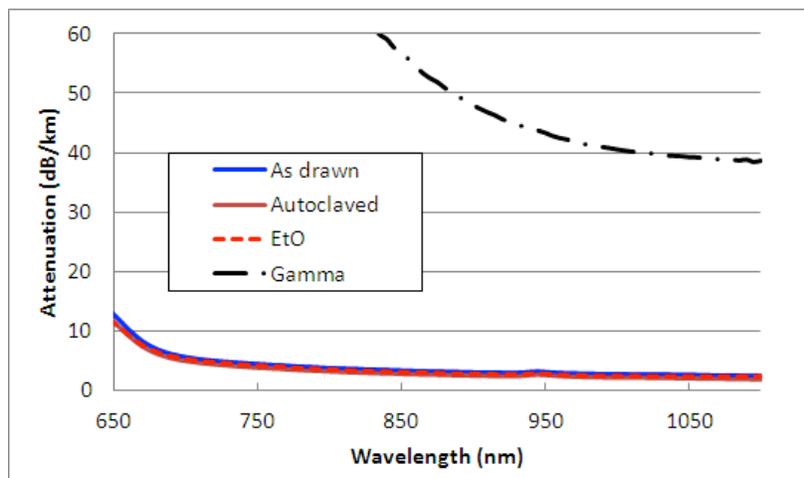


Figure 4.3.2. Spectral attenuation observed for the as-drawn and sterilized Acrylate fiber.

In more details, effects of sterilization on the fibers can be analyzed using the attenuation spectra. Figure 4.3.2 shows the data obtained for the fiber with dual acrylate coating. It can be seen that the spectra of the as-drawn, autoclaved and EtO treated fibers are nearly identical. The gamma radiation resulted in additional losses which gradually increase at shorter wavelengths.

Effects of gamma radiation on silica-based glasses have been studied extensively, including effects on optical fibers.¹⁵⁻¹⁸ Gamma radiation and X-rays are able to travel many inches in most materials and are sometimes called "penetrating" radiation. Radiation may cause the displacement of lattice atoms or electron defects that involve changes in the valence state of lattice or impurity atoms. The ionizing radiation produces electron-hole pairs in the glass structure. In general, these absorptions are associated with either oxygen deficiency or oxygen excess in the glass network. The most fundamental radiation-induced defects in glass are the non-bridging oxygen hole center ($\text{^{\wedge}Si-O}^*$), the E' center ($\text{^{\wedge}Si}^*$), the peroxy radical (POR: $\text{^{\wedge}Si-O-O}^*$), and the trapped electrons, where the notation " $\text{^{\wedge}}$ " represents three bonds with other oxygen in the glass network and " $\text{^{\ast}}$ " denotes an unpaired electron.¹⁵ All those defects result in development of absorption bands in the range below 800 nm. The lower frequency wings of those bands spread throughout the whole visible and near-infrared spectral regions and are responsible for the changes shown in Figure 4.3.2. Thus it follows that gamma radiation is not the best method of sterilization even for fibers with non-fluorinated coatings.

It is important to note that adsorption of radiation is cumulative, so using lower radiation doses (≤ 25 kGy) may still be appropriate, especially for the wavelength above 1000 nm.

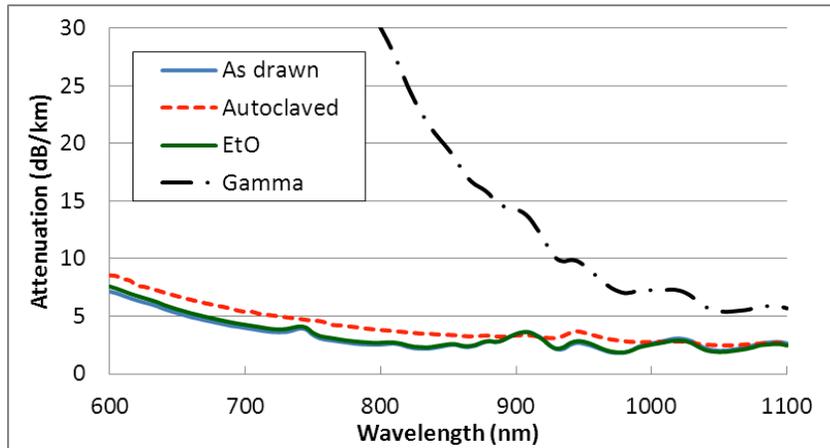


Figure 4.3.3. Spectral attenuation observed for the as-drawn and sterilized Silicone/PEEK fiber.

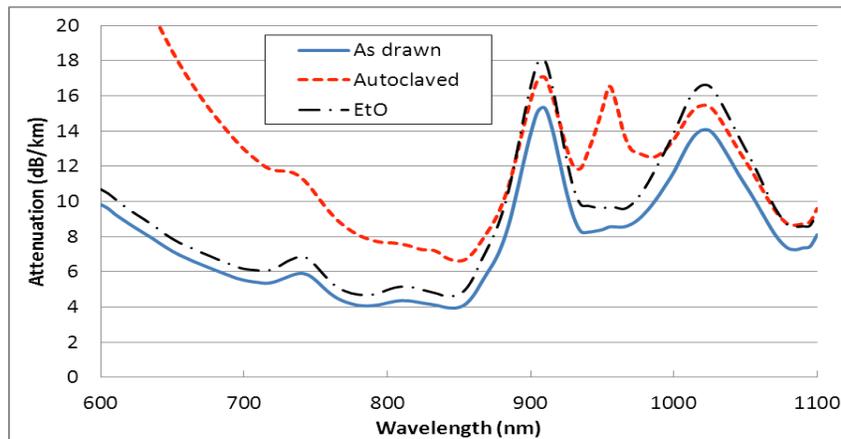


Figure 4.3.4. Spectral attenuation for the 200/HCS/ETFE fiber exposed to different sterilization conditions.

Spectral attenuation behavior of the polyimide-coated fiber was found to be identical to the one displayed in Figure 4.3.2. Two of the studied fibers (Silicone/PEEK fiber and 220/HCS/ETFE) have dual cladding, so their attenuation spectra displayed additional features due to the light absorption by the coatings. Figure 4.3.3 shows the attenuation spectra of Silicone/PEEK fiber. The observed small peaks around 750 and in the range 810 – 1100 nm should be attributed to the absorption of silicone. It is worth noting that all those peaks disappeared after multiple autoclaving cycles.

Effects of autoclaving on 200/HCS/ETFE fiber were found to be more significant as shown in Figure 4.3.4. Two new features developed in the spectrum: a peak at ~950 nm and also a shoulder at shorter wavelengths. Those features are typical for O-H groups bonded with silicon.¹⁹ Among the studied fibers, 200/HCS/ETFE is the only one employed with a single polymer cladding. Indeed, water molecules can penetrate through the polymer cladding and react with the glass surface forming Si-O-H groups.

5. CONCLUSIVE REMARKS

Different methods can be applied for sterilizing optical fibers. The three of the most common sterilization techniques were trialed in this work: steam sterilization (autoclaving), treatment with ethylene oxide and gamma radiation. The fibers under study were employed with different coatings: dual acrylate, hard polymer clad, silicone and polyimide.

It has been found that EtO treatment did not cause any harm to mechanical and optical properties of the fibers. Multiple autoclaving cycles caused minor influence on the fiber strength. The only found effect was a gentle decrease of n_d observed for the fiber with dual acrylate coating. For most fibers, the attenuation was not or very weakly affected by the autoclaving. The only exception was the polymer-clad fiber where O-H absorption peaks developed in the spectra upon the autoclaving treatment.

In contrast, gamma radiation was found to strongly affect the fiber attenuation. Furthermore, mechanical strength of fibers with fluorine-containing coatings rapidly degraded upon the exposure to gamma radiation. It follows that gamma radiation should be generally avoided for optical fibers. Still moderate radiation doses (≤ 25 kGy) can be applied to fibers with non-fluorinated coatings, assuming that the fibers are utilized at wavelengths above 1000 nm.

Disclaimer. Some of the results herein are based on the latest techniques of accelerated testing. Although as such, they may provide an indication of the useful service performance of materials, they do not constitute or imply any warranty on behalf of OFS. No warranties other than OFS' normal contractual warranties should be inferred.

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