Fabrication nano fiber optic by chemical etching for sensing application

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Abstract

Recently, emerged an easy way to fabricate to a nano fiber-optic sensor
(NFOS) using the inscription on the silica fiber. A simple etching reactor was
developing to obtain reproducible tapers of desired diameter and length. This
approach is reflected on-line monitoring of the etching using the transmitter and
receiver system. The experimental data indicate that the diameter of the optical fiber
decreases linearly with the time of survival of hydrofluoric acid, and etching was
used at room temperature. In this study, we were examined the best rate of etching
that used for the fabrication of such sensors. In the aforementioned technique, this
method aims to determine the diameter of the reduced core and show the evolution of
the two different processes from the nano fiber regime to the fixed regime in which
the mode was remote from the surrounding evanescent field, intensity can propagate
outside the segment fiber when the core diameter is less than 100 nm. To expire a
easy-going has developed the diameter of the fibre after etching to the process has
been done successfully.
Keywords: fiber optic sensor, chemical sensors, biosensors, nano-fiber optic.

تصنيع ليف ضوئي نانوي وبطريقة كيماوية لاستخدامه في تطبيقات التحسس

الخلاصة

برزت في السنوات الحديثة، طرق متعددة لتصنيع ليف بصري نانوي واستخدامه في
تطبيقات التحسس. لذا تم تطوير معدات تفاعل كيماوي بسيطة وسهلة الاستخدام للحصول على أقطار
وأطوال مختلفة من الليف النانوي. وتهدف الطريقة باستخدام المراقبة المباشرة لتغيير القدرة الخارجة
وباستخدام مرسلة ومستقبلة ألياف ضوئية بسيطة، مما تعطي دلالة إلى مقدار إبعاد الليف النانوي.
أضف إلى ذلك تم اعتماد الموديل الرياضي لتوضيح التفاعل الكيماوي مع الليف الضوئي ومقارنة
النتائج العملية بذلك السلوك الرياضي. وأعطت البيانات إن تصغير قطر الليف الضوئي يتغير خطيا مع
زمن التفاعل وإن التفاعل تم بدرجة حرارة الغرفة. يهدف البحث إلى تطوير وإيجاد أحسن حالة لمعدل
الإزالة ليصبح ما هو مناسب كعنصر متحسس ومن ناحية أخرى تم استخدام القلويات لإيقاف التفاعل
بنجاح.

INTRODUCTION

Numerous recent studies focused on advancements in fiber optic chemical sensors and biosensors [1]. Such sensors have numerous benefits compared with other types of sensors. The benefits include small size, immunity to electromagnetic and radio frequency interference, remote sensing, multiplicity of information from a large number of sensors into a single fiber, and in certain cases, low cost. The nano fiber optics (NFO) emerged in sensing applications as; biochemical, biomedical, and environmental sensors[1, 2]. Generally seek to increase the sensitivity of the tapered fiber and the use of tapered fiber geometries, such as in remote-sensing applications. Sub wavelength- diameter biconical fiber tapers (SBFTs) have divers of applications for the sensor for evanescent optical coupling and nonlinear optics [3]. Usually, the SBFTs are produce from a single-mode fiber and are composed of three contiguous regions: a waist radius of the minimum delivery areas in transition on both sides that taper in the nominal fiber diameter. Controlled heating and the withdrawal of single mode fiber (SMF) is the most common method of [4]. However, the main challenge of the acid with the existing drilling techniques is the high optical losses. So far, the lighting proved chemically etched biconical fiber tapers showed insertion losses of over 10 dB even in the micro-scale waist diameters. The high losses have been attributing to the formation of surface corrugations during the etching.

Fabricated fibers have decreased engraved thermal or chemical etching. These tapers can be fabricated starting with large multimode fibers. Heating can be accomplished with a flame, an electric arc, or a high-powered laser. Chemical etching may also produce tapers. The fundamental difference between chemically etched tapers and heat-pulled tapers is the index profile of the fabricated tapers [2].

Chemical etching may also produce tapers [3, 4]. The fundamental difference between chemically etched tapers and heat –pulled tapers lies in the index profile of the fabricated tapers. While the infrastructure core/ cladding interface and the province and the gradual decrease in heat pulled and chemically etched tapers tend to be only the core in the waist region. Thus, the tapers may have different optical characteristics. The heat-pulled taper contains a small core and a thick cladding; achieving light guidance through a core/cladding structure and the chemically etched taper has light guidance in the waist through a core/air structure.

Theoretical:

Through the process of etching of fiber-optic, and the diameter of it a drop in an area prone to HF[5].HF reacts with silica according to the following reactions, with the interaction domination in the second reaction at high HF concentration.

$$SiO_2 + 4HF \rightarrow SiF_4 + 2H_2O \qquad \qquad \qquad …(1)$$
$$SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H \qquad \qquad \qquad …(2)$$

The fiber is surrounding by 48% HF solution uniformly. Thus, we may assume that the concentration of the acid remains essentially constant during the etching reaction. Consequently, it is reasonable to assume that the etching rate is fixed. In addition, the reaction of HF with fiber limited surface area of contact. Diffusion of HF acid in to the silica lattice is also rather slow and need not be consider here. Thus, the change in mass of the fiber can be determined assuming a dissolution rate to depending on the dissolution constant, surface area of transfer and the concentration of the acid causing the dissolution .

Methodology

A deep penetration depth for the evanescent wave is important in current fiber optic evanescent field sensors (EFSs). An NFO is produced via the chemical etching method using HF. The penetration depth of an optical fiber stripped of its cladding is dependent on the wavelength of the incident light, the RI of the surrounding medium, and the incident angle. Chemical etching is a common and useful method for fabricating such element sensors [6]. The current study is proposed to examine and determine the etching time necessary to control the shape of the NFO during etching .

Etching reactor

One of the simplest ways to create an NFO is to use HF as an etching agent. During the etching process, once the optical fiber is etched, the conditions that govern the propagation of light inside the fiber change with the decrease in the fiber diameter. To monitor the improvements in etching, the fabrication method should incorporate a means by which to measure the light coming in at the output end of the fiber, which will allow for continuous control over the etching process and therefore produce a precise diameter of the NFO segment[7 .[

It is also necessary that the etching method should be limited to a specific length of the fiber to obtain the desired NFO length and desired interaction region. In addition, the etching segment should be available for easy addition or exclusion of etchant, washing fluid, and neutralizing agent. Additionally, the etching reactor should be designed such that the potential analytic can be conveniently loaded into the NFO region of the fiber to evaluate the transmission characteristics of the light. Finally, the success of this fabrication technique is achieved if low-cost materials are used to construct the etching reactor and if currently available optical instrumentation is used. The various steps in the design of the fabrication apparatus are described below.

A detailed schematic of the etching reactor is shown in Figure1-A. Two Plexiglas pieces (100 mm ×60 mm ×6 mm) are glued together to serve as the base. A 6.4-mm circular hole is drilled in the top piece to function as the etching chamber, Show in Figure 1-B, the experiment setup for fabricating NFOS .

Also known as acrylic, acrylic glass , and Plexiglas, polymethyl-methacrylate (PMMA) is an amorphous thermoplastic material with excellent optical properties[8]. This material is as transparent as glass and allows 92% of sunlight to pass. PMMA is characterized as solid or rigid, medium in strength, and simple to cut and scratch, yet easy to polish. This material is also weather and sunlight resistant, with no decline in its optical or mechanical performance outdoors, and it is used to construct the etching reactor. Additionally, the HF etchant does scratch glass, although it does not dissolve most plastic materials. Plexiglas, as the obvious choice for optical applications, facilitates examination from below and provides a mount for the bottom-view microscope required to measure the fiber waist diameter and taper.
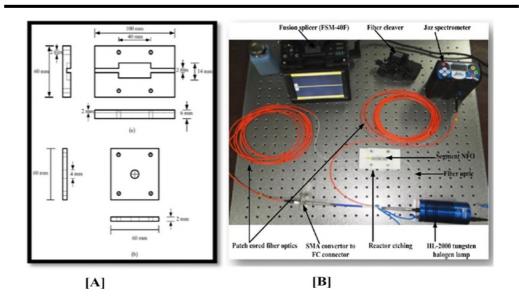
Figure (1), [A] the etching reactor design, and [B] The experiment setup for fabricating NFOS .

Etching experiments

The diameter of the fiber optic decreases in any area exposed to HF or HF-acid etching. The HF acts on the silica and becomes the dominant dissolving force in the second reaction at high HF concentrations according to the subsequent reactions with these interactions. As such, when the fiber is immersed in HF, it is surrounded by a constant 49% HF solution throughout the etching reaction. As a result, a two steps wet etch process were used consisting of etching with 28 M HF acid to remove cladding and 12M HF acid to reduce fiber core diameter. The rate of etching is believed to be fixed. Moreover, the reaction of HF with the fiber is limited to the surface area of contact. The HF also diffuses gradually in the silica pattern. Thus, the modification in the mass of the fiber can be selected by assuming a dissolution rate that depends on the dissolution constant, the surface area of transfer, and the concentration of the acid that causes the dissolution .

Removal of the plastic sheath is carried out by immersing the fiber in acetone (Fisher Scientific) for 15-20 min followed by the use of a mechanical removal tool, such as a fiber optic stripper (NO-NIK). A fiber optic cleaver (NO-NIK) is used for the clean-cut fiber tip to obtain better efficiency of the light inside the fiber assembly. The fiber is subsequently tested under a microscope to ensure a good entrance face. Once the fiber end is washed with 70% (v/v) ethanol solution, it is inserted inside the central 0.2 mm fiber groove to ensure a sufficient length of fiber. After the fiber optic segment is cleaned, the ends are cut and the fusion splice (Fujikura) is constructed using a patch cord fiber with an FC–FC connector. Details of this process will be presented in the following section.

Results and discussions

The NFO is distinguished by thin, long, and notably soft silica. Taper fibers are prepared using a simple and low-cost chemical technique. Etching is conducted with a 49% HF solution to first remove the fiber cladding, and other concentrations of HF

Solution is subsequently used to reduce the core diameter. Two different methods are employed for real-time monitoring of the fiber during this etching process, and both measure light differently in terms of input versus output. In the first approach, a laser diode is used as the source and is detected with an optical power meter that incorporates a photoreciver to detect light power. The second approach uses a Jaz spectrometer system, which uses white light as the source and CCD array detectors as to determine the percentage of transmission loss.

At the beginning of the process used to etch the fiber, HF solutions with different molarities (28, 12, 10, 8, and 6 M) are applied. This experiment delineates two methods that depend on the ease of monitoring the change in the power transmitted through the optical fiber. The fast etching rate at the core makes the etch depth difficult to manage, especially if using high-molarities acids, such as the 28 M HF solution. The rate of etching increases after reaching the core material. Therefore, it is advantageous to slow the etching rate with a weaker HF solution. However, performing the entire etching procedure with diluted HF solution would take longer than necessary because the cladding material above the core etches at a substantially slower rate than the core. The following tests show that if etching is performed, the HF hastens the rate of etching. The entire process is shown in Figure 2.
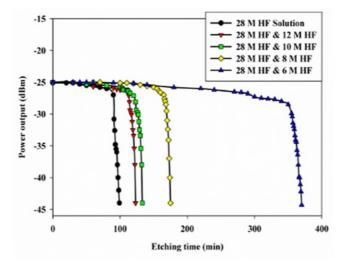

Figure (2) the power transmission (dBm) in fiber optics as etching time (min)
using 28M,12M, 10M, 8M and 6 HF solution.

Table 1 presents the different HF solution concentrations used to strip the thickly clad fiber with a total radius of 31.25 μm and a core radius of 31 μm. Hence, $\Delta t1$ is the etching time for the cladding segment, and $\Delta t2$ is the etching time for the core segment. Therefore, we can calculate and compare the etching velocity of the cladding, v1, with the etching velocity of the core, v2. This comparison is established using the data in Table 1, which illustrates that lower molarities of the HF solution decreases the speed of etching in the cladding and core segments. Therefore, the speed of etching in the core is faster than in the cladding.

Table (1) the variation of HF solution concentrations for fabricated NFO using Pin= -25.0 dBm when the initial etching time is t= 0 minute, and the Pout= -44.0 dBm when etching time stops.

HF molarities	Δt_1 (min)	Δt_2 (min)	$P_{out(t1)}$ (dBM)	v_1 µm/min	v_2 µm/min	$\Delta P_1/\Delta t_1$ dBm/min	$\Delta P_2/\Delta t_2$ dBm/min
28M	90	9	-27.0	0.695	3.44	-0.022	-1.888
12M	95	10	-26.7	0.657	3.10	-0.017	-1.730
10M	112	13	-26.4	0.558	2.38	-0.012	-1.354
8M	155	15	-26.8	0.403	2.06	-0.012	-1.133
6M	350	20	-27.0	0.1786	1.55	-0.005	-0.865

The second method in the experiment focuses on white light as a source and the Jaz spectrometer as a receiver. This method uses the same type of optical fiber and etchant with a 12 M HF solution to get slow interaction between the HF solution and the fiber optics. While monitoring the light transmittance levels during one iteration of the experiment, it was interesting to note that near t=200 min, just as the etching depth transitioned from removal of the last of the cladding to the start of reduction of the core, the meter level moved rather quickly until the end of the experiment near t = 250 min. The reaction was stopped with NaOH, and the fiber was washed with distilled water. This process is shown in Figures 3 A-B. After the experiment, the transmission power remained constant, and a value of 30% in the NFO was obtained. Hence, chosen maximum T% at λ =740 nm.
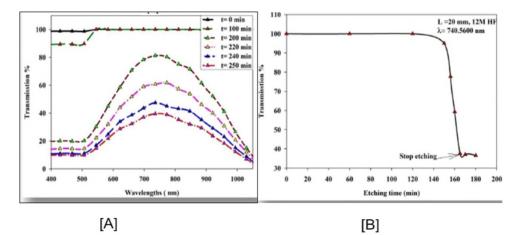

[A]   [B]

Figure (3) [A] the transmission percentage power in fiber optics segment vs. etching time (minute) using 12M HF solution, and [B] the transmission for λ= 740 nm vs. Etching time (minute.(

Figure 4 shows the shape of the tapering fiber after etching with HF solution and the SEM images for the NFO optical fibers. The smoothness of the core can be observed in the nano-diameter range after immersion for 30 min in 49% HF and immersion for 90 min in 10 M HF.
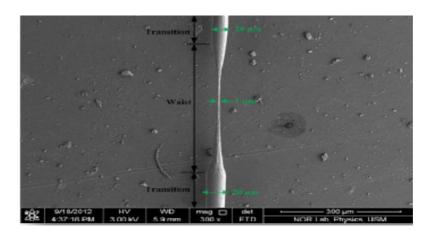
**Figure (4) FESEM image for taper a fiber in smoothing the NFO segments and
the shape-tapered fiber.**

The cross-sections of the optical fiber diameter at different etching times were
tested by FESEM and are shown in Figures 5A-F according to the NFO diameter as a
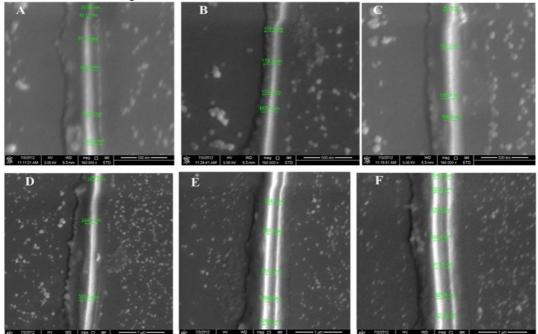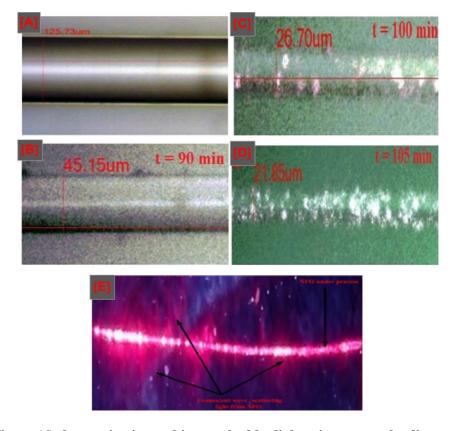function of the etching time and the use of 49% HF solution.

**Figure (5) the cross-sections of the optical fiber diameter at different etching
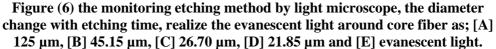times were tested by FESEM.**

NFO fiber images using a microscope

Light microscopy is one of the monitoring methods for etching fiber optics to
fabricate NFO. The normal fiber optics is without jacket under microscope before
etching time in air, obtained in Figure 6 [A]. The optical fiber diameter at different
etching times is displayed in Figures 6 [B-D] after full removal of the cladding and

reduction of the core, the diameter is approximately 20 μm, which enables the evanescent wave around the core. The reason for this observation in an optical interferometer is due to the number of different reflections inside the core of the optical fiber and the incident point at the core–cladding (HF solution) interface. When the area of the incident light rays refracts the light into the liquid cladding, more visibly shaped circles interface and the rays propagated inside the core as TIR. This situation indicates that the NFO segment becomes a good source light, and if another core fiber is simply placed beside it, the light is propagated inside the new fiber as well as shown in Figure 6[E].

Figure (6) the monitoring etching method by light microscope, the diameter change with etching time, realize the evanescent light around core fiber as; [A] 125 μm, [B] 45.15 μm, [C] 26.70 μm, [D] 21.85 μm and [E] evanescent light.

Conclusion:

A simple and low-cost technique has been suggested for the manufacture of NFO fiber optics and tapered fiber probes using HF chemical etching techniques. A two-step wet etching procedure is applied that consists of etching with 49% HF for 60 min to eliminate all cladding on the core, followed by another HF bath at a lower concentration to finish the etching and reduce the core diameter at a manageable etching rate of approximately 0.7 μm/min. This etching procedure ensures reproducible results, permits real-time monitoring of the etching method, and offers a method for determining the stop information needed to halt the etching at the precise diameter required. This technique produces a successful and effortless method with

which to gain moment-to-moment control of the fiber diameter to produce lengthy and smooth NFO that are highly sensitive to a broad range of sensing environments.

A two-step wet-etch method was useful, consisting of etching with 49% HF, followed by a slower etch process using a 12 M HF solution to reduce the core diameter. The real-time monitoring of etching ensures reproducible results, permits in-line monitoring of the etching method, and provides information used to halt the etching at the desired diameter. This technique provides a useful and simple method with accurate control of the fiber diameter for the fabrication of long, smooth NFO and the fabrication of probes with nanometric tips and high sensitivity with a wide range in bright and dark sensors.

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