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Dynamics Rate of Fiber Chemical Etching: New Partial Removal of Cladding Technique for Humidity Sensing Application

Muhammad Arif Riza[1], Yun Ii Go[2*] and Robert R.J. Maier[1,2]

[1] School Of Engineering and Physical Sciences, Heriot-Watt University, Jalan Venna P5/2, Precinct 5, 62200 Putrajaya, Malaysia
[2] Institute of Photonics and Quantum Sciences, Heriot-Watt University, Edinburgh, Scotland, EH14 4AS, United Kingdom
E-mail: y.go@hw.ac.uk

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Abstract

Sensor based optical fibers requires thinner cladding for enhanced interaction between the evanescence wave and the environment. Fiber Bragg grated fiber sensors relying on an axial strain requires chemical etching to weaken the fiber allowing more sensitivity in terms of strain. Ease of sensor multiplexing can be achieved through etching mid-section of fiber which are scarcely reported with custom-made holder. This paper proposes a technique for etching at the middle section of a fiber with custom fiber holder. Chemical etching of a single-mode silica fiber was done with 48% hydrofluoric acid. Etching proceeds until fiber was completely etched. Optical spectrum was recorded at an interval of 3 minutes. Microscope visuals indicated possible etching with the mount design until ~10µm at 45 minutes with the fiber intact compared to longer etching time. Longer etching duration than 45 minutes results in optical spectrum displaying null spectrum depicting core damage. Etching of fiber follows a linear trend and reaction rate was determined to be 1.699 µm/min experimentally. The proposed fiber holder and receptacle was capable of etching a fiber with minimal defects while maintaining ease of fiber handling.

Keywords: Cladding, Etching, Optics, Spectra, Wavelength

1. Introduction

The etching of an optical fibre is a common practice especially for fibres that will be utilised as an environmental sensor. The removal of cladding allows better exposure of the core to the environmental measurand [1]. A common etching technique widely used, involved etching the tip of the fiber [2-5]. Though viable in terms of handling, the fiber lose its capability of multiplexing. Multi-node sensing is highly regarded especially for environmental sensing. A multiplexed fiber sensor forms multi-node array of sensors can easily map out regions of the environment for the desired measurand. Though multiplexing is still possible with the aid of optical couplers or splitters, addition of extra modules could complicate sensor systems and are prone to errors.

Presently used techniques to etch middle section of a fiber involves bending the fiber into U-shaped form before dipping into an acid proof beaker [6]. Although simple, this technique can cause uneven etched due to part of the fiber is bent and stretched during etching. Mishandling can easily cause fiber damage as fibers lose their structural integrity upon etching. For fiber bragg gratings (FBG), bending of the mid-section where the gratings are imprinted can ruin its profiles,

Table 1: Related studies involving fiber etching process.

Section of fiber etched	**Custom Holder/ Receptacle**	**Etchant Conc. Used**	**Etching rate (**μm/min)	**Remarks**	**Ref.**
Fiber tip	None	N.A	N.A	Diluted HF, Unknown etchant concentration reported.	[7]
Fiber tip	None	40% NH_4F, 48% HF	N.A	Two different etchant used.	[1]
Fiber tip	None	46% HF	N.A	Repeated etching process (two times) until reached desired diameter of 84.74 μm.	[2]
Mid-section	None	48%	3.27	Fiber subjected to bend strain during etch. FESEM reveals slight morphological roughness on fiber surface.	[6]
Mid-section	None	30%	1.2	Fiber subjected to bend strain during etch.	[3]
Mid-section	Modular Holder with Tygon tubing	48%	N.A	Custom fiber holder is made up of several smaller pieces with may complicate attachment or removal of fibers if required.	[8]

introducing errors in bragg wavelength. A fiber should be at all times be at a state as free from any strain or bending on its segment that is to be etched as possible. This way, the defects to the fiber can be minimized.

Since the process of etching is utilized in other works, some literatures have been made in order to clarify the research gap of this study. Table 1 shows etching techniques used by other studies for sensing applications and their reported etching rates. Different techniques used affects the etching rate and the way the fiber can be positioned of handled. Based on collected literatures, most end-section etchings have not reported their etching rate. This could be due to the etching process was not the focus of the study. Another reason may be that it could be difficult to estimate the rate since it requires fibers to be etched and measured for every etch duration in order to find a trend in the diameter changes. Such methodology could be a hassle for studies that only see the etch process as a preliminary stage in their study.

Mid-section etched fibres are not uncommon and have been attempted by other studies. Different techniques yield their own perks and etching rates. For instance, Zaca-moran [6] etch middle section of the fiber without any holder attached and solely rely on proper placement of the fiber in a teflon petri dish. While this technique is straightforward, it produces fibers that are morphologically defect. This is due to the fibers are suffering strain in its etched portion. Another study [8] involves a v-cut acrylic holder for mid-section etching. The modular holder may pose a difficulty in handling, attach and detachment of the fibers. Furthermore, no custom receptacles were mentioned for containing the etchant solution during etching which could mean that the mentioned study only utilize teflon petri dish filled with etchant to allow full immersion of the fiber and its holder in substantial amounts of HF. In achieving a fiber free from defects and ease of handling, a technique with custom made fiber holder was proposed. This technique aims to allow etching of fiber with small amounts of HF used while minimizing present strain during etching. This paper reports on the performance of this technique which consist of fiber holder design and receptacle in undergoing etching process of a single mode silica fibers.

1.1 Etching Reaction

Chemical etching involves chemical reaction with the etchant and the cladding of the fibers. Silica glass are usually the material that the core and cladding are made out of. Most common etchant used typically for silica fiber etching is hydrofluoric acid (HF). This is because HF reacts with silica glass with reaction as follows [9]:

$$SiO_2 + 4HF \rightarrow SiF_4 + 2H_2O \quad (1)$$
$$SiO_2 + 6HF \rightarrow H_2SiF_6 + 2H_2O \quad (2)$$

Reaction shown in equation (2) is more prevailing when concentration of HF used is high, denoted with the higher empirical formula value than in equation (1) [10]. High concentrations of HF acid are frequently reported in various studies that involve chemical etching of silica fibers. With the established chemical reaction, mathematical expression can be followed as:

$$\frac{D_i}{D_f} = C - \left(\frac{2k[HF]}{D_i\rho}\right)t \quad (3)$$

Where Di and Df are initial and final diameters, C is a constant value, k is reaction constant, [HF] is the concentration of the etchant, ρ is fiber density and t is reaction time. Based on equation (3), the concentration and reaction constant affects the final diameter of the fiber. Therefore, in finding the etching rate experimentally, the concentration and volume of the etchant should be kept constant.

1.2 Effect of etching to sensor fibers

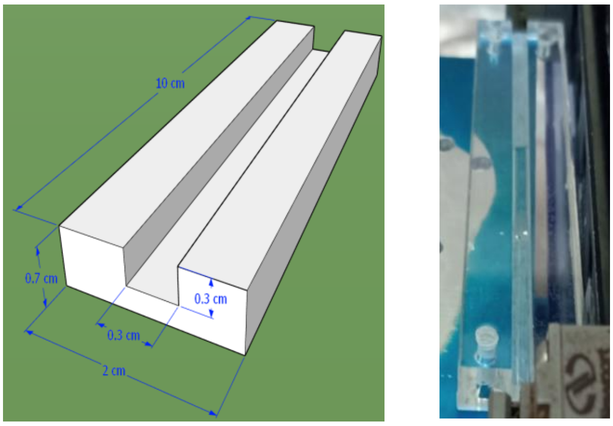
The removal of cladding is desirable for fibers modifed to be sensors. This is because thin cladding minimizes the axial strain required to induce spectral shift of light. Such phenomena renders the fiber to allow optical transducer to display clearer peak in the light spectrum. For core grated fibers that rely on hygroscopic material for environmental sensing, a thin cladded core allows the sensing material to easily induce strain when reacting with a measurand [11]. Wavelength shifts of power fluctuation can be determined in a much precise manner.

2. Experimental Design

Mount designs can vary from one study to another. As there are many possibilities to design a fiber mount, it is always recommended to allow a setup that minimize any mishandling during the etching and testing process. Furthermore, when dealing with HF acid, it is highly advisable to conduct the etching process in a fumehood complete with ventilation system. The mounting receptacle was designed in a way that the HF can be drained or remove with ease and care.

2.1 Fiber receptacle design

In this study, we propose the use of a simple fiber receptacle design in the form of rectangular cube. The fiber receptacle is made from Poly(methyl methacrylate) (PMMA) which is chemically resistant to HF [9]. A narrow half channel was carved along the longitudinal direction of the receptacle made of HF proof material. The channel is where the fiber will be placed and immersed with HF. Differing from other etching method involving the dipping of fibers while it is bent, The narrow channel allows small amounts of HF required to immerse the whole diameter of the fiber without having to bend the fiber. HF is a dangerous chemical and designs that allows small usage volume can be beneficial and safe. Maximum volume of HF suitable for this receptacle for this receptable is approximately 250µl. Higher volume of HF may cause overflowing of the HF outside the channel. Therefore, throughout this study, volume of the etchant used is kept constant at 250 µl. Additionally, during etching process, ventilation system was set to low to prevent loss of the HF volume due to becoming dried. Figure 1 shows the receptacle design used in this study.



a) b)

Figure. 1: Fiber rectangular, single-channel receptacle design. (a) Visual representation. (b) Prototype design.

2.2 Fiber holder design

Partial etching along the fiber may prove to be a challenging task. The fiber must be at all times secured and at an optimal tension. A tight fiber can cause elongation of the core that may perturb any gratings if they are present causing widening of the bragg wavelength. A loose fiber during etching can cause bragg spectrum to suffer from power loss, side lobe development and experience an irreversible shift from its default imprinted bragg wavelength [8]. As such, tension must be kept at a medium during the etching. Furthermore, it is not preferred to detach the fibre from the holder due to increasing probability of breakage. Post-etched fibers should be left at its holder before an attempt to coat or test for its performance especially for fibers with core gratings.

In this study, a custom fiber holder was made from a chemical resistant ABS material. Modelled via AUTOCAD software and printed using 3D Ultimaker 3, the fiber holder allows secure attachment for the fiber with minimal initial strain acting upon it. Figure 2(a) and 2(b) shows the design of the fiber holder and Figure 2(c) is the actual 3D printed version. The curved corners minimizes bending of the fiber during attachment and the small channel on the sides serves to further secure the fiber. Screws with washers on the sides are to fasten the fiber on to the mount. The fiber holder was used in conjunction with the fiber receptacle during etching in this study.

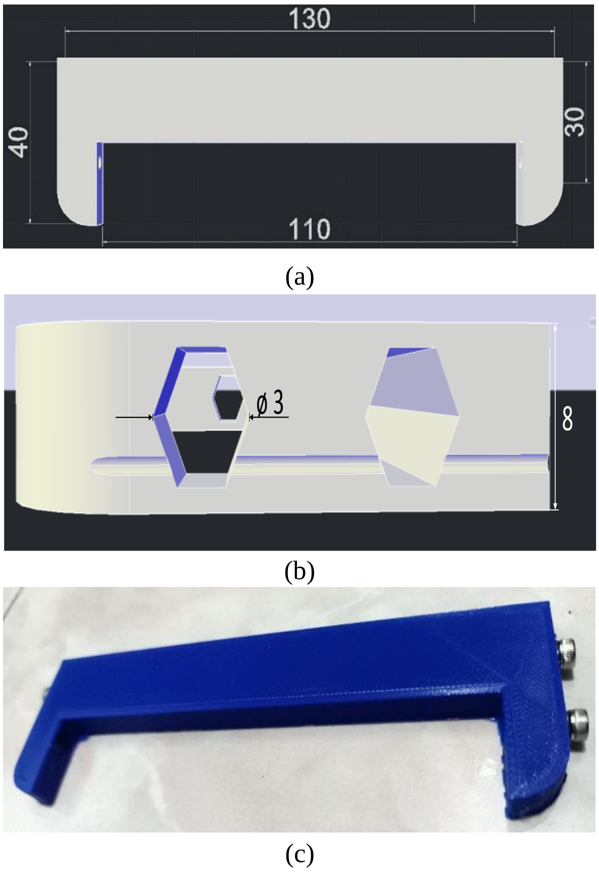


Figure 2: Fiber holder design. Measurement units are in (mm). (a) Top View, (b) Side view, (c) Printed Fibre holder

2.3 Chemical Etching Process

The stripping of the cladding in this study was done via the chemical etching method. The chemical etchant used is hydrofluoric acid (HF). Due to the dangers posed by this acid, the setup is assembled within a fume hood chamber for safety precautions. A controlled volume of 200 µl HF was used in this study. The overall setup is as shown in Figure 3. The connection was set in series and fiber mounted on the holder and in the channel of the receptacle. A timer was started as soon as the fixed volume of HF acid dropped in the middle of the fiber into the channelled receptacle with the aid of micropipette. The optical spectrum of the fiber was recorded by OSA and computer connected at the end of the fiber. Curves of the transmission spectrum was recorded by OSA at intervals of 3 minutes until the fiber completely breaks and showed null spectrum. For etching of fibers intended for thickness measurement, removal of fibers from receptacle requires the HF to be drained via micropipette and rinsed with deionized water. The fibers were left to dry before transferred to glass slides for microscopic observation.

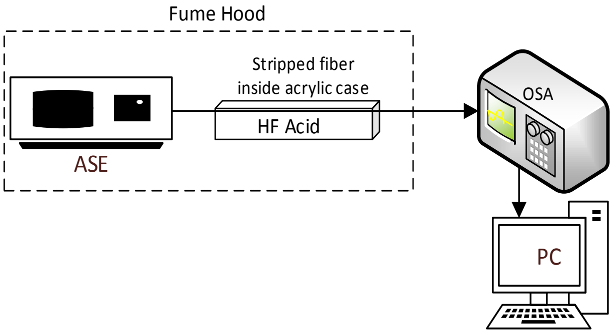


Figure 3: Etching process setup overall

3.0 RESULTS & DISCUSSION

Observations have been made on the fibre through microscope images with fixed magnification lens. This is to ensure that the etching have taken effect and successfully thinning the cladding of the fiber. Fibers are etched at different times and was monitored under a calibrated microscope at post etch.

3.1 Microscopic Visual of the Etched Fiber

The etched fibers are measured and observed via a microscope at 10x magnification. Based on Figure 4, unetched fibers that have their buffering outer layer stripped with a wire stripper have thickness of 86 µm. All fibers prior to etching were stripped with the same wire stripper, therefore it is a basis for all other fibers etched at different period of time. The stripping of fiber with wire stripper is for removal of outer buffer layer of the fiber that reinforces the mechanical integrity of the fiber. However, HF cannot react with the outermost layer as it is not made of silica. Upon exposure to HF of 15 minutes, thickness drops to 56 µm diameter. Further etching until 30 minutes to 45 minutes yields diameter of 30 µm and 10 µm respectively. At approximately 10 µm, the cladding is completely removed since the Corning PureMode HI 1060 fibre only have core diameter of ~9 µm according to its specifications. Etching of all of the fibers shown seems to be uniform visually.

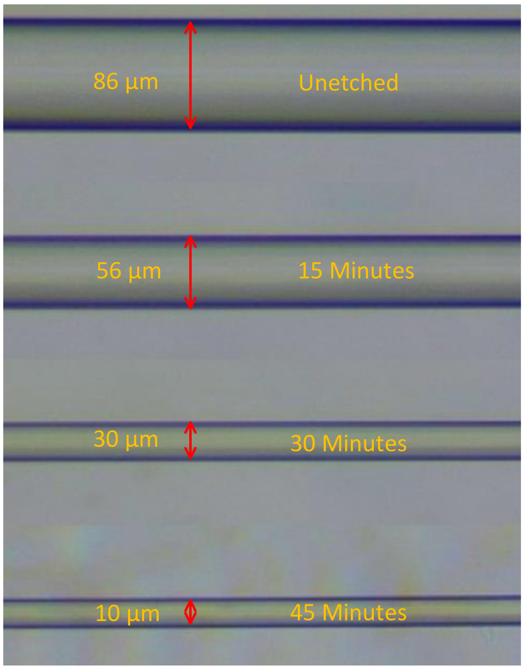


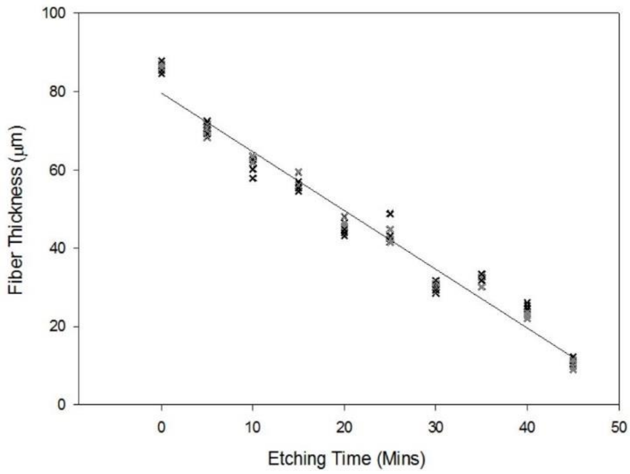
Figure 4: Microscope observations of etched fibers observed at 10x magnification

3.2 Thickness measurement of fibers

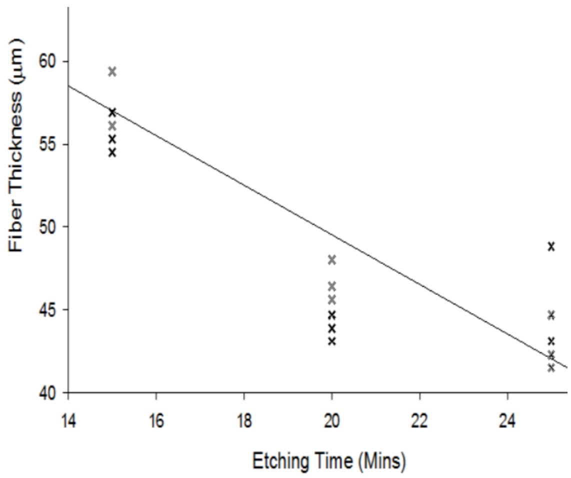
Thickness measurement of fibers were taken with several trials and an average value of thickness was calculated for fibers etched at different times. Fibers are etched at different etching intervals of 5 minutes up until 45 minutes maintaining all the controlled variables. The thickness of the fibers were measured via a microscope software calibrated with a 10x microscopic lens. Ten points of measurement taken throughout the etched part of the fibers and average values have been calculated. Figure 5 shows the plotted fiber thickness against time including all the trials. As expected, fibers etched with the use of the fiber holder follow this trend of etching and etching time to reach minimum but usable diameter of the fiber optic is 45 minutes as depicted in Table 2 which shows the average measured diameter of etched fibers. Since the graph in Figure 5 is linear in nature, determination of etching rate can be estimated from the gradient. The experimental etching rate obtained from this technique is approximately 1.699 µm/min. Longer etching times may not be advisable with this fiber etching setup especially when etching an FBG. Grated cores can be damaged and cause large light leakages and perturb the bragg wavelength of the fiber.

Table 2: Average thickness obtained for each fiber etched at different durations.

Etch Duration (Minutes)	Average thickness (µm)
0	86.48
5	70.39
10	60.53
15	55.79
20	45.39
25	43.51
30	30.18
35	32.21
40	23.45
45	10.02



(a)



(b)

Figure 5: Thickness of fibers. (a) All data. (b) Zoomed in at etching time 14-25 minutes

3.3 Optical spectra of fibre

Throughout the etching process of the fibre and post etch, the optical spectra of the fibre is monitored actively with an optical spectrum analyser (OSA). The optical spectrum are recorded every 3 minutes interval until the fiber finally breaks or dissolved completely in the etchant. As the cladding is thinning, the light propagation is affected and can cause shifts or disturbance to the optical spectra. Transmission power will decline due to evanescence leaking out of the etched region. Figure 6 shows the transmission spectra of several periods of etching time. At 0 to 6 minutes of etching time, spectra appears to be having minimal reduction or perturbation of transmission power. The power keeps increasing and peaks at 36 and 42 minutes before experiencing a decline at 45 minutes, rapid decline at 48 minutes and finally null transmission at 51 minutes (not shown in figure since no transmission was obtained, fiber destroyed completely). For ease of interpreting the optical spectrum and transmission spectra changes, Figure 7 graph of peak transmission drop was plotted. The rapid transmission power declines upon etching >42 minutes.

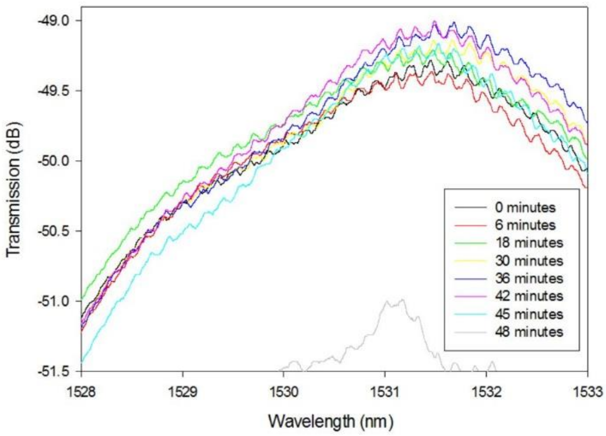


Figure 6: Optical spectrum of a fiber recorded at different etching durations

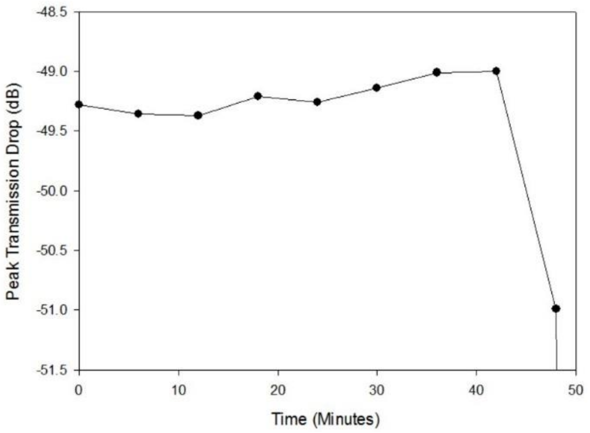


Figure 7: Peak transmission drop of etched fibers.

4.0 Conclusions

The proposed fibre etching technique has been applied and tested to etch standard SMF fibre. The designed fiber holder and receptacle have successfully allowed the etching of mid-section of the fiber with minimal defects. Etching of approximately 45 minutes duration with HF is possible with this setup to remove the cladding almost completely for use as an evanescent sensors. Etching at this duration results in the final thickness to be ~ 10μm. The experimental etching rate estimated to be 1.699 μm/min with this current setup. The study have successfully reported etching time and fiber thickness data with the proposed technique of etching. FBG type optical fibers are suitable for this technique as it exerts minimal tension to the fibers during etching, protecting the inscribed bragg gratings on the core from damage that can permanently alter the bragg wavelength on the optical spectrum.

Proper techniques are essential to be implemented in cases where a fiber is to be stripped of its cladding. While it is not necessary to etch a fiber, some sensor applications may require thin or absence of cladding especially if evanescence principle, interferometry or refractive index change monitoring is to be utilized. Post etched fibers, especially at sub-micron diameters are highly prone to damage and therefore requires a sort of holder for proper handling or transport. A fiber holder has to be simple and sufficient to secure the fiber during etching. Detaching and attaching the fiber must also be as effortless as possible because etching process is usually the earliest phase of an optical sensor preparation methodology. This study have succesfully reported a proposed technique of etching with a non-modular custom made fiber holder and single channel fiber receptacle design.

Acknowledgements

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