Fabrication and Characterization of the Fiber Optical Taper for a Surface Plasmon Resonance Sensor

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ABSTRACT— For a fiber optical surface plasmon resonance (SPR) sensor a short part of its cladding should be removed to coat a thin layer of a metal. Usually this is problematic when an optical fiber with small core diameter is used. In this paper, a new method using µliter droplet of the HF acid for short fiber optical taper fabrication is reported. Using this method in a multi-mode optical fiber with the core/cladding size of 50/125 µm a 2 mm long taper with 40 µm diameter is fabricated. Roughness of its surface is investigated using an atomic force microscopy. The measured mean value of the roughness is about 8 nm. A 60 nm thin layer of pure silver is coated on the taper surface in order to investigate its performance using a fiber optical SPR sensor. Using this SPR fiber sensor measurement of the lead concentrations in water ranging from 0.1 to 10 part per million (ppm) is reported.

KEYWORDS: chemical etching, fiber optical taper, optical fiber, surface plasmon resonance, sensors.

I. INTRODUCTION

Fiber optical tapers have different applications in optics especially in the fiber optical sensors. Fiber optical tapers can be used in biosensors [1], devices for coupling light into the waveguides [2] and micro-resonators [3]. Since the fiber optical tapers proved the possibility of leakage of guided optical power, they can be used in the fiber optical surface plasmon resonance (SPR) sensors [4]. In a fiber optical taper guided-modes have to be guided in a narrower area. Therefore, its surface quality would results in the amount of light losses.

Thermal stretching and chemical etching are two usual methods to make a fiber optical taper. One can reach to a high quality fiber optical taper with low optical losses using thermal stretching [5]. But its complex setup to heat the fiber via CO_2 laser and stretch it via motorized setup cause the fabrication of the fiber taper to be expensive [6]. In addition, in this type of tapers, fiber clad won't be removed and it stretches with core. Consequently, less optical power would be leaked out. This leads to reduction of the sensitivity in the sensors using this type of the tapers. In contrast to this method, chemical etching method is simple and inexpensive. But it attracted less attention mainly due to its high optical losses. Recently, an etching method based on surface tension driven flows of hydrofluoric acid micro droplets is reported [7]. With such a method low loss fiber tapers are developed. The advantage of this method is ability to make short tapers for various applications such as fiber optical SPR sensors [4], waveguide tapers [8] and etc. Fabrication of mm sized fiber tapers by chemical etching is problematic. Here in this paper fabrication of a fiber taper with about 2 mm long and its application in a double-pass fiber SPR sensor is reported. This is achieved via an optimized method based on surface tension driven flows of hydrofluoric acid micro droplets. Instead of using a droplet with about 100 µliters of acid, here much less volume of about 10 µliters is used to etch the fiber. In order to fabricate

such a tiny fiber taper, micro droplets of acid is added frequently to the main droplet to prevent the evaporation of the main droplet in the one hand and to keep the volume of it less enough to cover small area of the fiber in the other hand. Direct qualitative measurements such as atomic force microscopy (AFM) indicate high surface quality of the fiber after etching. The optimal etching parameters of the mentioned method are discussed in detail. In addition, indirectly measurements are done when the taper is used in a SPR fiber sensor to measure the lead concentration in water.

II. Chemical Etching of the Fiber

Since the method reported here is based on the surface tension driven flows of hydrofluoric acid micro droplets, its brief description is presented here. In this method, as shown in Fig. 1, a droplet of HF acid with specific size is placed on a hydrophobic plastic container and a short section of the fiber float in the droplet and stretched from both sides [7].

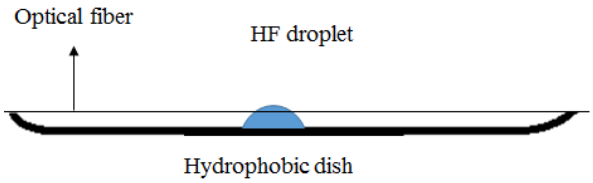

Fig. 1: Schematic of the etching method based on surface tension driven flows of hydrofluoric acid micro droplets to fabricate fiber optical taper.

Stretching from both sides causes a surface tension along the fiber. This leads to the formation of micro-droplets of HF acid which is distributed equally in both sides of the main HF droplet. Figure 2 presents the formation of the micro-droplets along the fiber. This phenomenon can be described using the Marangoni effect [9].

Fig. 2: the picture shows the micro-droplets distribution along the fiber caused by surface tension via stretching of the fiber [7].

The surface tension (γ) can be expressed as Equation (1).

$$\frac{\partial \gamma}{\partial x} = \left(\frac{\partial \gamma}{\partial C_{HF}}\right)\left(\frac{\partial C_{HF}}{\partial x}\right) \quad (1)$$

where C_{HF} is the HF acid concentration and x is its traversal direction. Since the HF acid used here has 49% purity, according to Marangoni effect [9], the HF traversed along the transition and its concentration decreased. This leads to formation of a graded diameter profile of HF concentration which is elongated in the transition region. According to equation (1) this results to a cone shape etching of the fiber. The acidic corrosion rate of about 1.6 μm/s is reported in Ref. [7] when the volume of the HF droplet is about 100 μliters. Due to the relatively large volume of the droplet, a taper with length longer than 5 mm is achievable.

Here in this work in order to make a short fiber optical taper, the HF acid droplet with volume about 10 μliters is used. To prevent the evaporation of such a tiny droplet, about 2 μliters of the HF acid is added to the main droplet each 15 minutes. To keep the form of the droplet and prevent from its spreading, it is placed on the plastic hydrophobic dish. Figure 3, shows the etching setup explained above. The green U-shaped piece of an optical fiber with its polymer jacket (overall diameter 250 μm) is placed to hold the main fiber slightly above to have an efficient reaction with acid droplet.

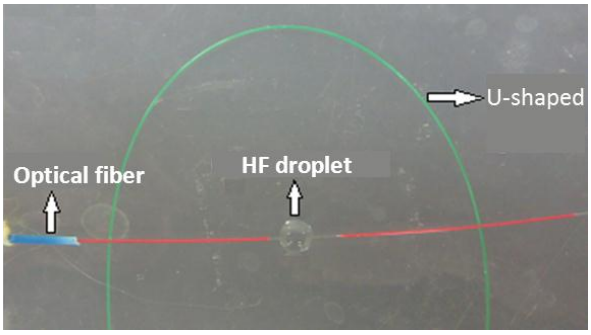

Fig. 3: Picture of the etching setup to make about 2 mm long fiber taper. The green U-shaped piece of an optical fiber is used to maintain the efficient reaction of the etching area with acid droplet.

The quality of the fabricated fiber taper is investigated first under an optical microscope. Figure 4 shows the image of both sides of the taper when a 4X objective of the optical microscope is used. The cone shape of the taper with minimum waist of 40 µm with a smooth surface can be seen in this image.

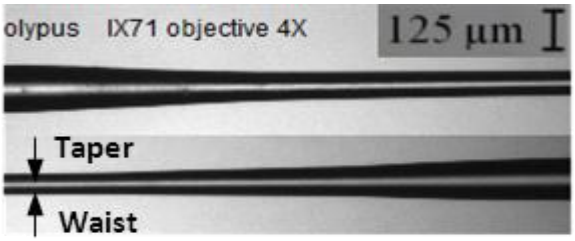

Fig. 4: The image of the fabricated taper under an optical microscope with a 4X objective. The minimum waist of the cone shape taper is about 40 µm.

As it is mentioned, the aim of the work reported here was to develop a fiber taper with a length shorter than 2 mm. Therefore the initial acid droplet volume used here was about 10 µliters. Consequently the etching rate obtained here differs to what is reported in the Ref. [7]. Results of the fiber etching rate is presented in Fig. 5. This rate is obtained for both clad and core of the fiber. Results indicate the acid corrosion rate of about 0.9 µm/minute for the fiber clad and about 2.5 µm/minute for its core. The corrosion rate is obtained via step by step measurement of the fiber diameter using an optical microscope. The measured points are shown in the graph presented in Fig 5.

As it is reported frequently, the main drawback of the fiber optical tapers made with chemical etching is its high transmission loss. This is mainly due to the high surface roughness caused during chemical etching. Here using the surface tension driven flows of hydrofluoric acid micro droplets with little amount of the acid droplets features a slow etching process and results in a very smooth etched surface. The optimized parameters obtained after etching of the fiber in different temperatures and acid purities. Fig. 6 shows the image from the etched surface of the fiber using an AFM when a 49% HF acid in room temperature is used. Although the maximum roughness of the surface showed in the Fig. 7 is about 42 nm, but the mean roughness of a selected area of the fiber taper measured to be about only 8 nm. This clearly shows a step toward to prepare a fiber taper with less scattering loss using a chemical etching method. During the etching and after it only negligible power loss is observed.

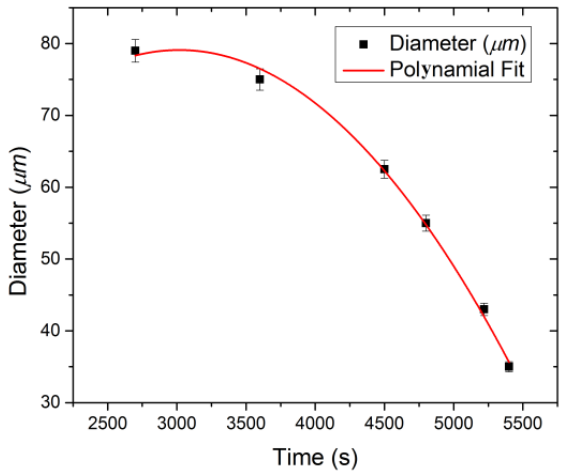

Fig. 5: Acidic etching rate of a multimode glass fiber (core/cladding-50/125 µm) using HF acid with 49% purity when a 10 µliters droplet is used.

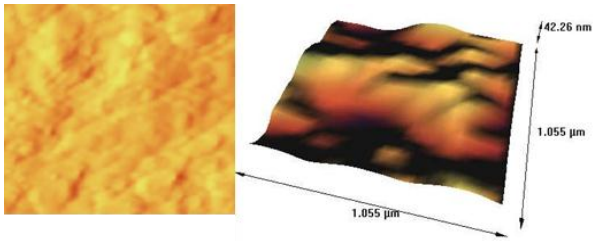

Fig. 6: Recorded 3D (right) and 2D (left) images of an area of about 1 μm^2 of the taper by AFM.

In order to have a qualitative test of fabricated fiber taper it is used to act as a SPR sensor probe. A SPR sensor consists of a thin layer of a metal on a dielectric surface. Here the dielectric surface is the surrounding of the fabricated fiber taper. To prepare Hence the fiber optical SPR sensor probe, a 60 nm thick layer of pure silver is coated on taper using ion sputtering deposition. Figure 7 illustrates the AFM image of a selected area of the coated taper. The maximum roughness of the surface is about 50 nm whereas the mean roughness is about 10 nm.

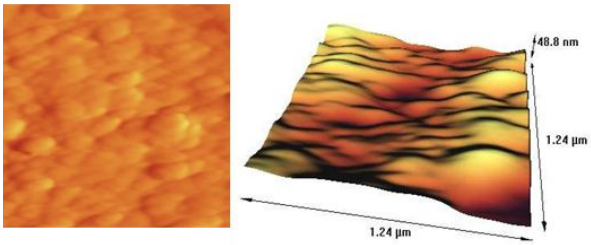

Fig. 7: AFM 3D (right) and 2D (left) images of an area of about 1.5 μm^2 of the taper after silver deposition.

III. Fabricated Taper Used in a SPR Fiber Sensor

In a fiber SPR sensor, a TM (transverse magnetic) or p-polarized light causes the excitation of surface plasmon wave (SPW) at the metal-fiber interface. When the energy and the momentum of the guided mode and SPW are matched, a resonance occurs which results in a dip in the transmitted/reflected spectrum of the sensor. Therefore, if the wavelength of the injected light varies, then resonance condition accrues for a wavelength in which phase matching condition is satisfied. This wavelength is called SPR wavelength. The SPR wavelength depends on the refractive index of the dielectric medium. Change in refractive index changes the value of the SPR wavelength [10]. Here the silver coated taper is used as a probe of a double-pass fiber SPR sensor. The fiber taper is located in the one end of a fiber and its end face is coated in order to reflect the guided mode back to the taper. In this way a double-pass sensor can be performed. The sensor is operated according to the setup sketched in Fig. 8 to measure the lead concentration in water.

The output light of a 3 W output LED used in the experiment is unpolarized. Its polarization is adjusted using a homemade fiber polarization controller and splits by a fiber 3dB coupler. One port of the 3dB coupler is connected to the fabricated fiber SPR sensor. The sensor is inside a solution of lead in water. The back reflected light from the fiber optical SPR sensor is connected to the monochromator using another port of the 3dB coupler in its opposite side. Finally the output optical power from the monochromator is detected using a photomultiplier tube (PMT). It is necessary to mention that, effective operation of the SPR sensor depends on the polarization of the coupled LED light to the sensor. Since the SPR wavelength depends on the phase matching between guided and surface plasmon waves, the polarized guided wave can satisfy the phase matching condition efficiently. In this way more phase matched light either with TE or TM polarization can be exited to guide in the taper part. The fact that special polarization gives rise to higher efficiency can be addressed to the deviation of the fiber taper from its perfect circular cross section during the fabrication.

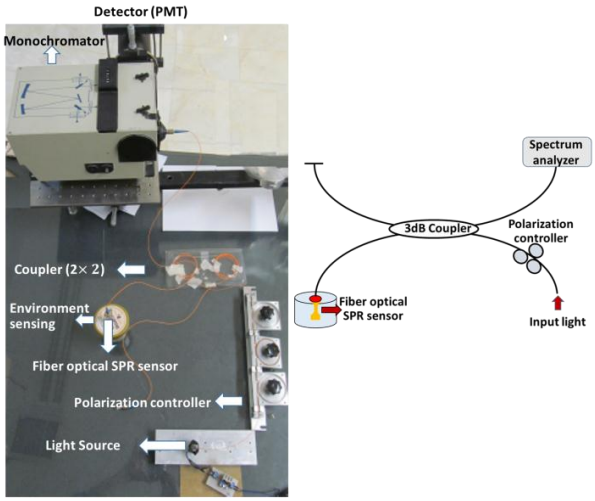

Fig. 8: the picture (left) of the sketched (right) experimental setup used to measure the lead concentration in water by fabricated fiber SPR sensor.

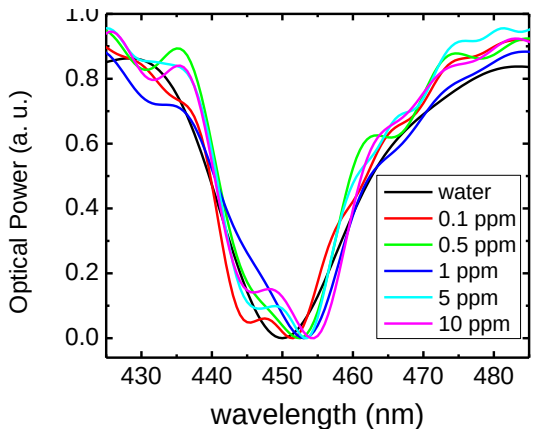

Fig. 9: measured spectra of the fiber SPR sensor for different concentrations of lead in water.

As it is mentioned above, the SPR wavelength is strongly depends on the phase matching between guided mode in the taper and the surface plasmon wave. Therefore, any change in the refractive index of the surrounding medium of a SPR sensor results in the SPR wavelength shift to compensate induced phase difference [10]. In the other word, a SPR sensor is capable to measure the changes of the refractive index of its surrounding by measuring the SPR wavelength shift. Here the standard solutions of lead in water ranging from 0.1 to 10 part per million (ppm) was prepared by dissolving lead nitrate in distilled water. Then each solution was investigated using the fabricated fiber SPR sensor by means of the SPR wavelength. The SPR wavelength of the sensor for distilled water measured to be about 450 nm. Figure 9 shows the spectra obtained from the sensor for different concentrations of lead in water. The increase in the lead concentration results to a red shift of the SPR wavelength.

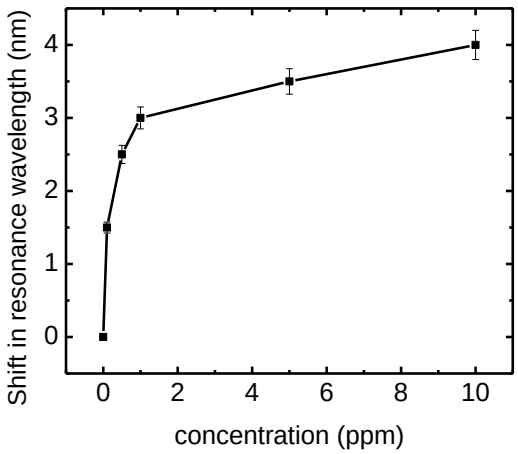

Fig. 10: The SPR wavelength shift versus concentration of lead in water.

As it can be seen in the Fig. 9, the fiber SPR sensor has less sensitivity when the concentration of the lead is increased. This is visualized in the graph presented in the Fig. 10 where the SPR wavelength shift is plotted versus the concentration of the lead in water. It seems a kind of saturation happens for the sensor when the change in the refractive index is large. In addition the graph presented in Fig. 10 can be used to determine an unknown concentration of the lead in water when a SPR wavelength measurement is done.

IV. Conclusion

In this paper a simple method to fabricate fiber optical taper with high quality is reported. The main objective of the work presented here was to develop tiny sized fiber optical tapers to use in the fiber SPR sensor. To achieve tiny sized fiber taper, much less volume of the acid droplet (about 10 μliters) is used in comparison with Ref. [7]. Therefore fiber tapers with length shorter than 2 mm are developed. To prevent the evaporation of such a tiny droplet, about 2 μliters of the HF acid is added to the main droplet frequently each 15 minutes. Although, the main drawback of the tapers made by chemical etching is there high optical losses, here with reported method fiber optical tapers of negligible losses are obtained. Direct characterization results obtained either in μm scale by optical microscope or in the nm scale using an atomic force microscope indicate a good surface quality after the chemical etching of the fiber. Measured mean value of about 8 nm is achieved for etched surface when 49% HF acid in room temperature is used. The fabricated fiber taper is used in a fiber SPR sensor for its qualitative test. Results of the measurements of lead concentration indicate the good quality of the fabricated fiber taper to distinguish even down to index of refractive changes caused by only 0.1 ppm lead solution in water.

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