TNT Vapor Detection Based on a Lab-on-a-Fiber: Achieving a Millimeter-Scale Sensing Element on Fiber

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***Abstract*—In this paper, we discuss in detail the lab-on-a-fiber (LOF) concept that we proposed earlier (Ma *et al.*, *Proc. 4th Eur. Workshop Opt. Fiber Sens.*, 2010, vol. 7653, PP. 76531E-76531E-4), and present a specific example of this platform intended for trace vapor TNT detection. We show how we arrive at the current LOF design by examining the performance, technical difficulties, and complexities of various possible architectures. In the design solution adopted in our example, the sensing section occupies a space measuring only $1.6 \times 1.6 \times 0.8$ mm, but contains several optical and chemical components/functions including a transmission/reflection mirror and TNT sensory film coated on a segment of the side wall of the fiber core. The fiber itself thus serves as the film substrate and when coated with the film becomes an evanescent-wave form fluorescent emission power collector. Its tiny dimensions notwithstanding, the LOF performs several simultaneous functions, including power density tuning and stray excitation light blocking. In addition, this LOF platform features ease of system construction and polymer film application. We demonstrate its fast response to TNT vapor, in the form of a 29% signal drop after 10 s of exposure.**

***Index Terms*—Fluorescence spectroscopy, optical fiber transducers, thin films, TNT explosive detection.**

I. Introduction

IN A PREVIOUS PAPER [1], we introduced the concept of the lab-on-a-fiber (LOF), a term we use to refer to fiber-optic sensing platforms featuring a millimeter-scale sensing section. In this connection, we wish to put forward some further points for discussion. By definition, an LOF occupies a tiny space yet it contains one or more regular laboratory functions or components that have been downsized or modified in order to fit into such a small space. Although fiber-optic sensors (FOSs) of this kind have existed for many years, they are treated as if they had the same significance as other platforms containing bulky components that are separate from the fibers and contribute to the large overall size of the sensing element. The most obvious evidence of this tendency is that they share the same name, "fiber-optic sensors."

It is commonly accepted that a FOS platform with a mini sensing element directly attached to the fiber has much more to offer in terms of real applications than its bulkier counterparts. The LOF concept is proposed in recognition of this fact, the intention being to promote the idea of a specific category restricted to FOS platforms featuring tiny sensing elements. This has become even more necessary since this whole class of sensors has grown into a very large family. The proposed new category will place this new type of fiber-based sensing platform squarely in the limelight, and shine the spotlight on efforts devoted to its further development.

Although inspired by the lab-on-a-chip (LOC) concept, the word "lab" in our LOF concept does not narrowly refer to chemical lab processes and microfluidic systems as in the case of the traditional LOC. The scope of functions or components is much broader, running across many disciplines, which might range from chemistry and physics to optics, as long as they can be integrated into the small space of an LOF for sensing purposes. We expect this LOF proposition will encourage research efforts that use fiber as the substrate to build lab functions or components to form sensing elements on a millimeter scale.

A representative example of an LOF application is TNT explosive detection. This involves the use of a specific material, amplifying fluorescent polymer (AFP) [2], whose fluorescent emission can be quenched when exposed to TNT vapor. We developed some modified TNT sensory polymers [3], which provide a fast fluorescent quenching response to the target vapor. Technically, a very thin layer of film on a substrate is sufficient to resolve the target vapor with a concentration as low as parts per trillion. This is the critical starting point in our search for a proper LOF platform for trace vapor TNT explosive detection. The desirability of an LOF-style platform for explosive detection cannot be overstated for either a handheld device or a remote-operation system, even though tradeoffs may be required based on the comprehensive analysis of relevant factors. These considerations are reflected in the design of our LOF in order to achieve fast response, robustness, tiny size, and low cost.

In the following sections, we will detail how the equivalent bulky individual lab functions and components can be downsized, modified, and simplified to fit into a $1.6 \times 1.6 \times 0.8$ mm

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cubic space, just the size of two fiber diameters. The response to TNT vapor of the resulting platform is verified experimentally. An overview of the LOF concept is given in the conclusion, as well as a summary of our work applying this concept for TNT explosive detection.

II. Choosing an Appropriate LOF Architecture

A. *Technical Issues for Platforms With Sensory Film on a Planar Substrate*

At first sight, depositing TNT-sensitive polymer thin film onto a planar substrate appears to be a straightforward procedure. This method offers certain advantages including the use of a series of commercially mature tools such as the spin-coater, the ellipsometer, and the high-end lab-based spectrometer. However, when it comes to connecting the substrate with the fiber, the question of packaging arises immediately. As illustrated in Fig. 1(a), some suitable fixture or technique has to be adopted to bridge the gap between the substrate and the fiber(s), making the tiny size of an LOF a hard-to-reach goal. It is also doubtful that an efficient enough use of excitation (Ex) light and collection of fluorescent emission (Em) power could be achieved without a complex and costly design, and that would further complicate the packaging problems.

From the point-of-view of achieving the miniature size of an LOF with the highest possible Em power collection, the architecture in Fig. 1(b) seems more sensible. Adopting the fiber itself as the substrate for the sensory film immediately reduces the size of the sensing element to approximately the diameter of the fiber. However, this approach leads to a fundamental change of the mechanism for Em power collection.

The architecture in Fig. 1(b) uses the evanescent-wave (EW) tail of the core mode to collect the Em power rather than using the fiber end as in Fig. 1(a), resulting in a lower Em power level due to the fact that EW tails have a much weaker light-collecting capability. A tradeoff proves to be the solution, i.e., utilizing the EW principle, while boosting Em power to the highest possible level. All discussions in the next several sections are devoted to this goal.

B. *Cylindrical Fiber Side Wall: Very Weak Ex Power in Ew Form*

The architecture in Fig. 1(c) demonstrates the weakest collectable level of Em light due to two factors: first, substantial Ex and Em losses occur when light passes through the beam splitter (BS) and second, only weak Ex power in EW form is available for film excitation, which stems from the fact that the cylindrical shape of the core side wall allows only a small percentage of the EW tails of core modes to extend into the film.

C. *Fiber Taper: How Good is it for the Increase of Ex Power in Ew Form?*

An incomplete but still elegant cure for the shortcomings of Fig. 1(c) is a taper at the fiber tip, as illustrated in Fig. 1(d). The tapered fiber tip, under study for many years, has been commercialized [4], [5]. It has three advantages. First, when the Ex light rays encounter the taper, their incident angles become steeper. In terms of wave-optics, this enables the EW tails of the core modes to extend deeply into the cladding, resulting in

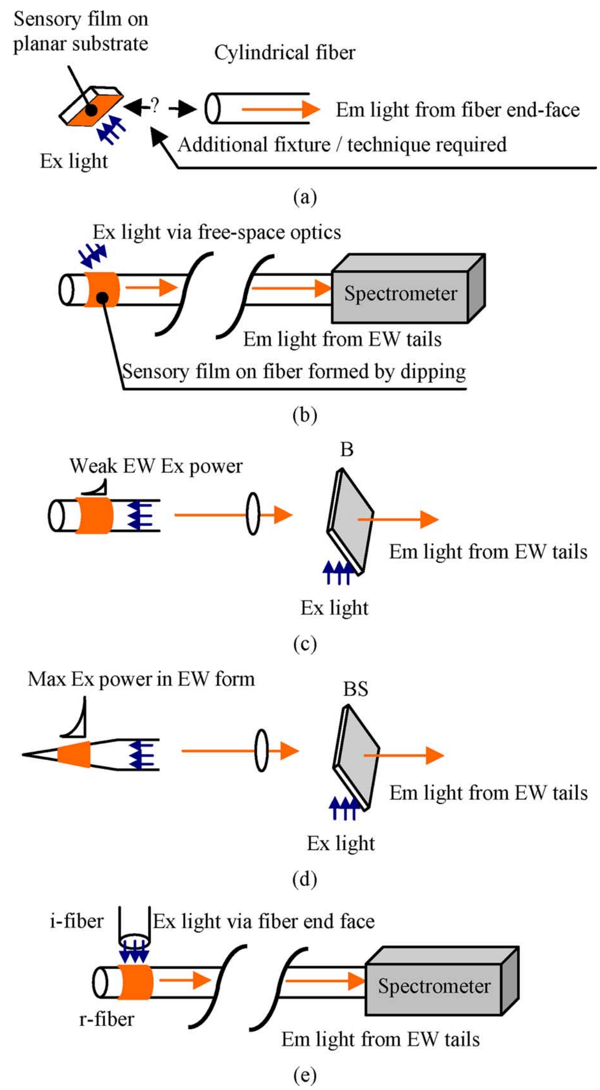

Fig. 1. Five approaches to sensing element architecture for LOF-type TNT explosive detection, where i-fiber is the illuminating fiber for Ex light delivery and r-fiber is the receiving fiber for the Em signal. (a) Packaging difficulties are encountered when connecting the fiber with the planar substrate. (b) Using the fiber itself as substrate for the sensory film immediately creates an LOF-size sensing element. Ex power can be efficiently used with free-space optics, but at the cost of a bulky sensing element. (c) One-fiber LOF architecture requires a beam splitter (BS) to separate Ex and Em light, complicating the packaging. Coating the polymer sensory film on the unmodified cylindrical fiber segment leads to low Ex/Em power. (d) A tapered fiber tip results in stronger Ex/Em power than (c) but still offers limited signal level increase. The BS still has to be used. (e) A second fiber guides the Ex light to the thin film coated on the original fiber, enabling the best use of Ex power and enhancing Em power collection. The BS component is omitted, enabling the simplest form of connection to the spectrometer. An additional small fixture is needed to hold the two fibers in position, slightly complicating the design of the sensing element.

stronger Ex power. Second, because of their deep penetration, these EW tails possess better Em power collection capability, allowing much stronger Em power to be sent to the detector. Third, both Ex and Em power in the cylindrical segment of the fiber can be concentrated in lower-order modes, thanks to the mode-converting ability of the taper. This permits stable transmission along the fiber. However, we must realize that the performance improvement through the taper is limited, determined by its EW-based nature for both Ex and Em power. Besides, the

BS itself introduces not only insertion loss to both Ex and Em power, but also alignment difficulties among the fiber, the Ex light source and the detector/spectrometer.

D. Beam Splitter: Is it Always Beneficial?

We notice that many sensing platforms proposed in the literature and even existing commercial products prefer the BS component to separate Ex/Em light. The most important reason for its popularity may be its elegant one-fiber architecture. This fiber serves as a medium for both sensing and light transmission. In addition, both Ex/Em forms of light are trapped within the fiber and will not be affected by what happens outside the fiber core, which is particularly useful when lossy samples are encountered where light from outside the fiber can interfere with the sample. Furthermore, the one-fiber sensor can be inserted into a bulky sample with an arbitrary volume, as long as the taper is completely immersed. This greatly simplifies the sampling procedure and is a desirable feature for field use.

However, the advantages of the BS are much less significant if we consider the sensory film exposed to the vapor-form sample, where the vapor, in fact, causes zero loss to the Ex/Em light and the sampling area is naturally limited by the film itself. However, the drawbacks of BS summarized above become significant, encouraging us to seek a more feasible alternative that offers highly efficient use of Ex power, combined with zero insertion loss, simplified packaging techniques, robust overall structure, and easy detector/spectrometer connection. From this point-of-view, we found that Fig. 1(b) has all these merits except that the use of free-space optics for Ex power delivery tends to increase the sensing element size. This difficulty can be overcome simply by introducing the second fiber as the replacement, as illustrated in Fig. 1(e). This architecture is the same as the one we investigated before [9], [10], the only difference being that the sample is a solid thin film instead of being a liquid droplet.

E. An All-Fiber Architecture Dealing With Many Issues So Far

Fig. 1(e) illustrates an elegant all-fiber architecture. There is one remaining problem: how to connect these two fibers securely with a designated air gap. In comparison to Fig. 1(a)–(d), the packaging techniques are significantly simpler, and the i-fiber may be bent to enable a smaller overall sensing section, but a bulky fixture is likely still needed. We believe it is possible to further simplify the packaging technique and reduce the size of this section.

F. An All-Fiber LOF Platform Dealing With All Critical Issues

After a thorough consideration of the architectures from Fig. 1(a)–(e), we now arrive at the final solution, an all-fiber LOF platform illustrated in Fig. 2, where the i-fiber is arranged in parallel with the r-fiber. The Ex light is directed to the sensory film area via a slanted end-face created at the tip of the i-fiber, which is achieved by a simple polishing process.

The packaging technique required by this change in architecture is as simple as a mere gluing process. The overall dimensions of the sensing section allow it to fit in a space as small as $1.6 \times 1.6 \times 0.8$ mm, as shown in the inset of Fig. 2 when large-core fibers with core/cladding/jacket/NA of 800μm/ 830μm/ 1400μm/0.37 are adopted.

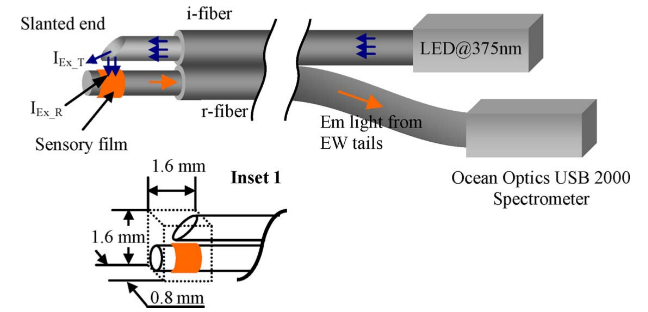

Fig. 2. Final sensing element architecture for TNT explosive detection, where I_{Ex_T} is the Ex power transmitted from the slanted end-face, I_{Ex_R} is the Ex power reflected from the slanted end-face, and the sensing section is of LOF dimensions. Inset 1 illustrates that such a sensing section can fit into a $1.6 \times 1.6 \times 0.8$ mm cube.

III. Several Mechanisms in Combination: Tuning Ex Power With Large Dynamic Range

An obvious difference between Figs. 2 and 1(e) is that the i-fiber in Fig. 1(e) appears to be more efficient and straightforward in delivering the Ex power to a small area of the sensory film. The slanted end-face in Fig. 2 without coating will, however, spread the Ex power over a larger area, especially the transmitted power, represented by I_{Ex_T}. The value of I_{Ex_T} as a percentage of the total incoming Ex power is determined by the slant itself and by the incident angles formed by the incoming Ex rays on the slanted end-face. These two factors, along with the reflection coating on this end-face, in fact, offer a combination of mechanisms for tuning the overall Ex power sent to the film. These are elegant mechanisms since they provide a wide dynamic tuning range but maintain the overall sensing section at LOF size. A similar Ex power density and film area to that in Fig. 1(e) can be achieved by depositing highly reflective coating on the slanted end-face of the i-fiber in Fig. 2. Given an extreme scenario – such as could occur under very high-power density – where the film presents signs of photo degradation, these continuously adjustable tuning mechanisms can play a role in achieving the highest possible Em signal level, while avoiding the degradation effect. A fourth tuning mechanism is afforded by the possibility of tuning the separation between the two fibers, a technique that could prove to be simple and straightforward since the power density drops rapidly with even a slight increase in this separation.

IV. Comprehensive Assessment of the LOF Platform for Trace Vapor TNT Detection

A. Impact of the Performance of the Sensory Polymer Film

The success of TNT vapor detection with the architecture in Fig. 2 strongly depends on the performance of the polymer sensory film for several reasons. First, since the film for the proposed LOF platform extends for only a very short distance, from one to several millimeters, signal accumulation through a long fiber segment [6], [7] is out of the question. A sufficiently high quantum yield is thus fundamental, and will also assist in reducing photo-bleaching of the polymer caused by high Ex power density. Second, as thin a film as possible is required for a high quenching percentage, which poses a challenge for Em power collection. Third, we found that the uniformity of the film will greatly affect the quenching performance as well. If the sensory film were being coated onto a planar substrate, both the thickness and the uniformity could be precisely controlled by employing the readily available spin-coater and ellipsometer. This equipment is not practical, however, for a film applied to the fiber wall, even when the fiber has a large core diameter (800 μm in this work). Selecting an alternative coating technique is thus crucial.

B. Solutions to the Technical Challenges in Creating a Thin and Uniform Sensory Film on the Curved Fiber Side Wall

Dip-coating can be used to form the sensory film on the curved outer wall of the fiber. Two steps are involved:

First, the fiber must be pretreated before being coated. Direct dipping of the fiber into the polymer solution will not generate a uniform film distribution. Proper pretreatment of the fiber core is mandatory to provide strong bonds between the film and the glass fiber core surface. An adhesion promoter is used for this purpose. We found that an excellent choice was 3-aminopropyltrimethoxysilane or $H_2N(CH_2)_3Si(OCH_3)_3$.

Second, the right polymer solution concentration must be found to form the appropriate film thickness. While it has been claimed that nanometer-scale film thickness is necessary to ensure a fast response to explosive vapor and to guarantee the reversibility of the film, in reality these characteristics will be limited by the Em power collection and the signal extraction capability of the employed platform. When a decision is made to adopt an LOF-type platform – not to compete with its lab-based counterpart but rather to offer a portable and cost-effective method with acceptable performance for identifying the presence of TNT explosive – tradeoff measures have to be seriously considered.

Under such a guideline, polymer 4b [3], with a quantum yield of 0.16, is chosen for performance assessment. First, the jackets and claddings of two identical large-core fibers with a core diameter of 800 μm and a length of 1.5 m are stripped off at one end for a distance of approximately 3 cm. The end of one fiber is angle-polished to form a 45° slanted end-face. This serves as the i-fiber shown in Fig. 2. The exposed core of the second fiber, the r-fiber, is cleaned with chloroform to remove any contaminant residues. This core segment is pretreated by dipping it into an adhesion promoter. Immediately afterwards, it is dipped into water to provide moisture for better adhesion and then into polymer solution of a known concentration to form a coating of film. The coated r-fiber is securely attached to the i-fiber with temporary glue and connected to the system (Fig. 2).

To generate the TNT explosive vapor, DNT powder (the decayed form of TNT) is sealed in a bottle measuring $\phi 45\times$ 50 mm. A $\phi 10$ mm hole is drilled in the middle of the cap to provide access for the sensing head. DNT evaporation occurs within the bottle until a dynamic equilibrium is reached, stabilizing the DNT vapor concentration. Although TNT vapor is not collected from the surrounding air, this is a good simulation of a preconcentration unit typically equipped with an explosive detector [8]. More importantly, this is a low-cost but effective approach for the quality evaluation of our film as well as for performance assessment of the entire system.

With this system, the optimized film thickness is identified by trial and error for several polymer solutions from high to low concentrations. Both the highest possible quenching percentage within a short time and the acceptable Em power level are used as the criteria for this assessment.

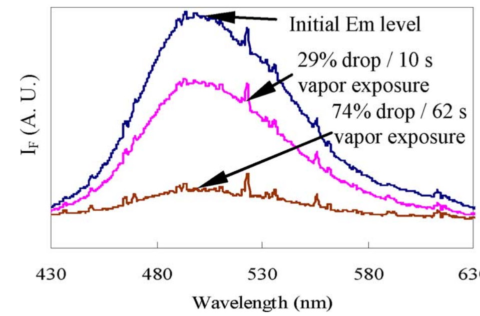

Fig. 3. Explosive vapor quenching response of the proposed LOF platform in Fig. 2 when coated with polymer 4b [3]. The film thickness of ~ 90 nm from 0.03% mass polymer concentration generates the fastest response, which is a 29% Em level drop for 10 s vapor exposure and 74% after 62 s.

For this LOF platform and film, an optimized film thickness of around 90 nm is achieved with a mass polymer concentration of 0.03%. The results illustrated in Fig. 3 indicate a fast quenching response of 29% after 10 s of exposure, from the time when the entire sensing head is immersed into the vapor container. A dramatic 74% quenching is observed after 62 s of continuous exposure. This result is comparable to $75\% \pm 5\%$ for a 200 Å thick film after 60 s exposure reported in the literature [11], where the development of pentiptycene-containing polymer was described. A better response such as $95\% \pm 2\%$ for a film 25 Å thick [11] after 60 s is not out of the question if we consider that our system (in Fig. 2) uses a low-end spectrometer that requires a thicker film. Higher sensitivity of the system is expected for an analytical grade spectrometer and thinner film.

V. Further Discussion

In a real-life scenario, the vapor sample likely contains other substances that may potentially interfere with the quenching response of the system and even trigger a false response. For example, Nitroglycerin tablets are used to treat artery disease and could be carried by patients entering crucial infrastructures such as railway networks, airports and harbors. However, Nitroglycerin is also an active ingredient of dynamite explosives. Insensitivity to the Nitroglycerin tablets is thus crucial to avoid false alarms. When we replaced the DNT with Nitroglycerin tablet powder, the preliminary test of our platform showed no quenching response. Here, we do not intend to test other species for two reasons: First, the selective response in such a test reflects the property of the sensory polymer itself rather than the instrumentation system, the major topic of this paper. Second, the selective response capability relying solely on polymer itself is not an easy-to-reach target since contaminants in the vapor can vary depending on the environment. The selective response to certain interferents does not mean that the system is immune to all contaminants that might be encountered. In reality, incorporating a preconcentration unit not only is a reasonable option but also is proved to be a determining factor in excluding the unwanted vapors [8]. The preconcentration unit can easily include many mature technologies such as adsorption/desorption and gas chromatography with synchronized cooling/heating cycles to effectively separate the target particles from others. With the capability of collecting a large volume of surrounding air, filtering or trapping the target particles into a miniaturized space and delivering them to the LOF sensing device, the preconcentration unit plays an equally important role in the entire system along with the sensory polymer material and the LOF device for the successful detection of the trace vapor TNT explosive.

VI. Conclusion

Our purpose in advancing the LOF concept is to further the development of this new class of fiber-optic sensing platforms whose functions are built on the fiber itself. Under this definition, an LOF is a specific category of FOS with millimeter-scale sensing sections as distinguished from platforms that have bulky functions separated from the fibers. Our motivation is a desire to encourage continuing R&D efforts into these tiny individual functions that can match the performance of bulky sensing platforms. The LOF approach could also eliminate the cumbersome fixtures needed to hold the various parts together, or at least reduce their number. A much broader scope of applications will no doubt be found, given our results demonstrating that an LOF is an elegant, compact, robust, portable, high-performance and cost-effective sensing platform.

In this paper, we presented an example of an LOF platform, specifically designed for TNT vapor detection. We compared several possible architectures available within the large family of optical fiber sensors. We analyzed how the separation of bulky lab functions/components from the fiber can lead to a range of issues in packaging, system complexity, size and overall cost. The final LOF design solution we reached after addressing these issues is based on the fact that we are dealing with thin-film fluorescence that is extinguished by a sparse vapor quencher. This LOF platform, which will fit into a space as small as $1.6 \times 1.6 \times 0.8$ mm, contains a reflection/transmission mirror for efficient Ex power delivery, a layer of polymer sensory film and the fiber section that serves as the substrate. Among the functions operating in this small space are mechanisms to collect Em signal power via the EW field, inhibit the guiding-mode formation of Ex light and tune the Ex power density on the film surface.

Techniques for quality control of the film are also summarized. Verification of this LOF platform by exposing it to TNT explosive vapor demonstrates that it delivers excellent performance. From the perspective of a real-life scenario, the pre-concentration is examined with the emphasis on its significant impact on the selective response of the system and the trace vapor TNT detection capability.

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