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Development of a micro-flame ionization detector using a diffusion flame

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ABSTRACT

A micro-flame ionization detector (micro-FID) design is presented that is targeted for use in a portable gas sensor. Our micro-FID is based on a diffusion flame and features a folded flame structure that is more sensitive than a counter-flow flame designs. Unlike conventional FIDs that use a premixed or open diffusion flame, an air-hydrogen diffusion flame is employed and tested in an encapsulated structure of Quartz–Macor–Quartz layers. Diffusion flames are generally known to be more controllable and stable than premixed flames, where the stability of the micro-FID plays an important role for portable gas sensors. Various channel designs for oxidant and fuel flows meeting with different angles at the burner cavity are tested to obtain a stable flame and high output sensitivity over methane test samples. To verify the empirically designed microchannel, we simulate the temperature distribution in the microchannel by using computational fluid dynamics (CFD) software. To gauge the sensitivity of the device, the collected electric charges per mole (C/mol) is calculated and taken as a reference value of ionization efficiency. The result of the folded flame design is 1.959×10^{-2} C/mol for methane that is about 34 times higher than the result using a counter-flow flame, which is 5.73×10^{-4} C/mol for methane, while one of the commercial macro FIDs' is 10^{-1} C/mol. This result shows that the micro-FID using the folded flame structure has higher ionization efficiency with less leakage of the analytes than of the classical counter-flow flame design.

1. Introduction

A Flame Ionization Detector (FID) is a sensitive detector for hydrocarbons. The measurement is performed due to the chemical ionization of the hydrocarbons in an oxy-hydrogen or air-hydrogen flame. The hydrocarbons can be detected by measuring the resulting ion current [1].

The FID has been extensively used in research and industrial labs to analyze gas samples in tandem with gas chromatographs (GC). An increasing interest in the portability and miniaturization of GC leads to the need for development of sensitive detectors compatible with these devices. A portable micro-FID device will be useful if it has a comparable sensitivity to a conventional FID. This micro-FID can be used to identify chemical threats, spills, and for environmental monitoring [2,3].

In conventional FIDs, micro-catalytic combustors have been employed [1,4]. The micro-combustor was made up of a catalytic

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film deposited on the surface of a micro-scale hot plate. Although catalytic combustion could sustain a flame with a relatively low fuel consumption rate at the micro scale, the presence of catalysts within the combustion regime will strongly affect the generation of ions from hydrocarbon. Fewer ions will reduce the FID signal making catalytic combustion unsuitable for the micro-FID. Kuipers and Müller proposed a micro-FID using a premixed flame with oxygen and hydrogen [5]. The oxy-hydrogen flame burns in a silicon channel encapsulated inside a glass-silicon-glass sandwich structure, which was proposed by Zimmermann et al. [5–7]. In conventional FIDs either a premixed or an open diffusion flame has been used. For pre-mixed flames the controllability of the fuel and oxidizer flow rate is relatively limited due to the intrinsic flame speed. On the other hand, diffusion flames are generally known to be more controllable and stable than premixed flames, which may be suitable to micro-FID.

As a first step to implement a diffusion flame in a micro-FID, a stable flame with the thickness below 1 mm was developed in our previous effort [8]. This paper focuses on the design of the channel to create a stable and highly sensitive diffusion flame within the specified thickness. We start with channel designs that can create a stable diffusion flame for a micro-FID. Various geometries of micro-channels that have different angles for oxidant and fuel flows to meet at the burner cavity are tested in order to increase

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Fig. 1. (a) Configuration of the micro-FID that is composed of a silicon channel (750 μ m thickness) and two Quartz plates (1 in. × 1 in. × 0.062 in., Technical Glass Products, Painesville, OH, USA). Two quartz plates are used to sandwich the Macor or silicon channel whose thickness is 750 μ m and make an encapsulated structure to enhance the stability of the diffusion flame. The flame burns inside the enclosed space by the two quartz plates on which electrodes are patterned. The electrodes are composed of two pairs of sputtered Cr/Au pattern that the electric field is established across and resultant current is measured when gas samples burns off. The top quartz plate contains the exhaust hole while the bottom quartz plate has holes into which the hydrogen, air, and sample gas are separately supplied. (b) The size of the Macor channel compared to a U.S. penny. (c) Assembled stack of the micro-FID. (d) A Vespel package where the FID stack is housed with connections for tubing and supplying the gases. The Vespel package also has holes for pogo pins to serve as electrical contacts.

the sensitivity of the FID output over methane test samples. The increase in sensitivity is attributed to an increase in the percentage of the ionization of the injected sample analytes. In other words, the channel is designed to create a flame that minimizes the loss of the sample analytes.

2. Design of the micro-FID

Fig. 1(a) shows the configuration of the micro-FID that is composed of a silicon channel (750 μ m thick) and two Quartz plates (1 in. × 1 in. × 0.062 in., Technical Glass Products, Painsville, OH,

USA). The two quartz plates are used to sandwich the channel and make an encapsulated structure to enhance the stability of the diffusion flame. The flame burns inside the enclosed space of the channel, created between the quartz plates. The top quartz plate contains the exhaust hole while the bottom quartz plate has inlet holes to supply hydrogen, air, and the sample gas. Two pairs of Cr/Au electrodes are sputtered on the quartz plates along with the flame footprint. These electrodes create an electric field across the flame that measures the resultant ion current.

Initial experiments are performed in order to observe the flame in silicon channels with different shapes. Silicon channels are used to employ the benefit of fabricating different channel structures at a relatively inexpensive cost using a batch process. Flame shapes are observed by changing the thickness of the silicon channel as well as the angle of the flow direction of the incoming air and hydrogen gas. An initial goal is to create a stable diffusion flame that can minimize the loss of analytes. The loss of analytes can occur between the flame and the wall of the channels, or at the discontinued regime in the flame.

Different thicknesses of the silicon channels are used to verify the critical dimensions that will locate the flame at the burner cavity. The same Si channel pattern but with different thickness of $500 \,\mu$ m, $750 \,\mu$ m, and $1000 \,\mu$ m are tested. The silicon channel with the thickness of $750 \,\mu$ m is the smallest dimension that allows the flame to stay stable inside the channel at the burner cavity. Channels with thinner thickness prevent the flame from sustaining into the burner cavity.

The channel length from each inlet port to the burner cavity needs to be long enough to keep each flow laminar at the burner cavity. Also, the length of the exhaust channel from the burner cavity to the exhaust hole is set long enough to avoid any splitting of the flame. As shown in Fig. 2, when the channel length of the exhaust channel is not long enough, the flame splits at the end of the channel at the exhaust hole that can lead to loss of the analytes. Furthermore, various channel angles between the air and hydrogen flow are tested to create a stable diffusion flame and high efficient flame. More detail is provided in Section 2 regarding the investigation of the effect of the channel angle on the flame shape and its stability.

After the desired channel shape and the flame are developed, Macor is used to machine the designed channel. Macor has low thermal expansion $(93 \times 10^{-7} \text{ m/(mK)})$ and good stability up to temperature about 1000 °C. Fig. 1(b) shows the size of the Macor channel compared to a U.S. penny. Fig. 1(c) shows the assembled

(a) (b)

Fig. 2. The flame splits at the end of the channel at the exhaust hole when the length of the exhaust channel is not long enough, indicated with the dotted line circle. The presence of an exhaust flame can lead to loss of analytes and needs to be avoided. (These pictures are taken when a small amount of methane is injected to visualize the hydrogen-air flame.)

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