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Photoimageable Thick-Film Components for a Ceramic Analytical Microsystem

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***Abstract*—This paper presents ceramic microfluidic components—a meander capillary column and a flame ionization detector—which have been designed and fabricated using photoimageable thick-film dielectric Fodel QM44F combined with low-temperature cofired ceramic tape. The components are integrated on a single piece of alumina substrate of dimensions 65 × 50 mm and have incorporated thick-film platinum heaters to ensure their temperature control. Details of the technological process as well as the application set-up for the tested components are presented. The samples of analyte were detected by a flame ionization detector after passage through a capillary column.**

***Keywords*—Flame ionization detector, microfluidics, LTCC, separation column, photoimageable thick-films**

Introduction

Miniaturization of chemical analyzers is a promising approach for realization of chemical instruments with lower cost, smaller size, lower power consumption, and increased portability for in-field use [1]. The functionality of such instruments can be improved when separation columns, heaters, sensors, valves, and detectors are incorporated on the same substrate and are resistant to chemicals and high temperature. Recently, low-temperature cofired ceramic (LTCC) technology has been used for manufacturing such mesoscale microsystems [2-5]. This paper presents two components of a ceramic analytical system—a capillary column and a flame ionization detector—that have been designed and fabricated using photoimageable thick-film dielectric Fodel QM44F combined with LTCC tape. The microfluidic components are integrated on a single piece of alumina substrate of dimensions 65 × 50 mm and have incorporated thick-film platinum heaters that ensure temperature control.

Application Setup

The application setup for the tested ceramic microfluidic components is shown in Fig. 1. The tiny quantity of the analyte is first stored in the sampling valve and then flushed out by a carrier gas into the separation column. The analyte, during transport through a micron-sized channel, undergoes separation into fractions. A thick-film platinum resistor deposited on the reverse side of the separation column can ramp the temperature of the separation column linearly up to 200°C. When the analyte reaches the combustion cavity of the flame ionization detector, it is burned there, producing ionized hydrocarbon radicals. The presence of hydrocarbon radicals is manifested by a change in ionic current between the anode and the cathode of the detector. Hydrogen and oxygen, as fuel and oxidant, respectively, are then delivered to the combustion cavity in order to promote stable and repeatable conditions for the burning process. A constant flow rate of hydrogen and oxygen is controlled by two flowmeters. The temperature of the combustion area can be controlled by a platinum thick-film resistor deposited on the reverse side of the anode of the flame ionization detector. This platinum resistor can operate in two modes: as a thermometer and as a heater that preheats the combustion area well above 200°C. A stable temperature of the combustion zone enables stabilization of the background current between the electrodes of the flame detector.

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Technology

A. Photoimageable Thick Films

The photoimageable thick-film process is a combination of conventional thick-film technology with some elements of thin-film technology. Photoimageable thick-film conductors and dielectrics incorporate a photosensitive polymer that allows the material to be directly imaged using mask ultraviolet (UV) photolithography. The UV light triggers a photo-initiator in the exposed regions of the layer that results in its selective polymerization. During the development process, the non-polymerized parts of the layer are removed; as a final step, the pattern is fired in a conventional furnace for thick films [6].

B. Exposure

In contrast to the LTCC technique, where a multilayer ceramic structure is formed in a single thermal operation, in a photoimageable process, the ceramic structure is built gradually by subsequent technological steps until the required thickness of dielectric is obtained. A precise alignment system for the exposure unit is absolutely necessary in this operation.

The exposure unit used in reported experiments contains a mercury lamp (500 W) that emits UV light of wavelength 365 nm, an optical system of lenses and mirrors that provides collimated light, and an alignment system with a split-field micro-

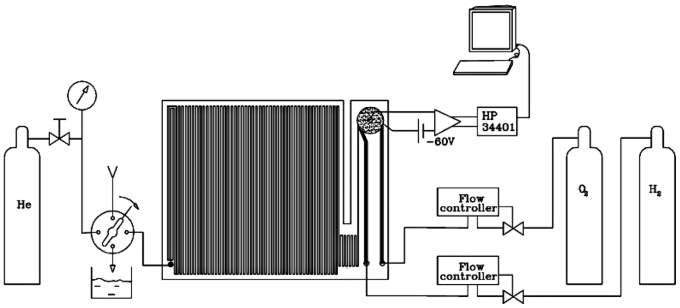

Fig. 1. Measurement setup for ceramic microfluidic components; a capillary column and a flame ionization detector are incorporated on a single piece of substrate.

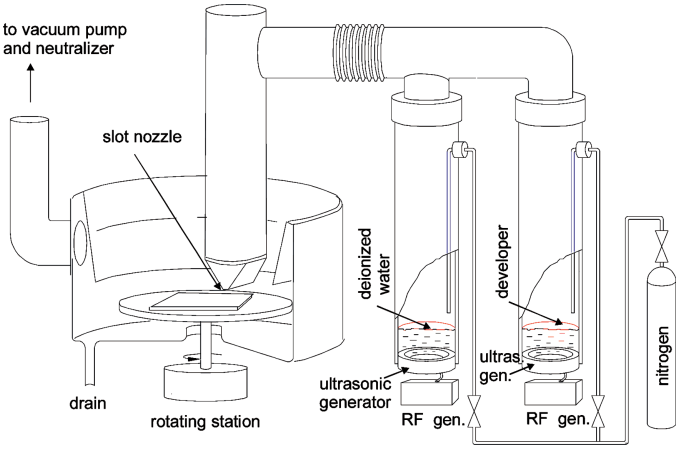

Fig. 2. Development unit.

scope that enables positioning of a photo mask in relation to the earlier deposited patterns.

C. Development in Mist

The development unit shown in Fig. 2 is described in detail in a patent application [7]. A fine mist of development solution and deionized water is created by the ultrasonic generators, blown out by the stream of nitrogen, and finally released through the flat nozzle on a surface of a slowly rotating sample. The mist reaches the substrate with some impact energy and enables removing soft, non-polymerized parts of the photoimaged thick-film layer. Development by fine mist allows formation of precise patterns without damaging them by dripping heavy drops of development solution, as happens when spray development is applied.

D. Drying in Vacuum

Processing of 3-D thick-film photoimageable structures reported herein requires the modification of drying the blank screen-printed photosensitive Fodel layers before exposure but after conventional oven drying at 80°C. Forming a ceramic structure of 100 μm height requires deposition of at least eight dielectric layers. Every deposition, the effective drying becomes more difficult and time-consuming. For every two drying cycles in the oven at 80°C for 30 min, an additional drying cycle in a vacuum (1×10^{-2} mbar) at 75°C for 15 min was applied. Vacuum-drying prevents the layer from sticking to the photomask during exposure. Fig. 3 shows an open microchannel photoformed in such a way in a Fodel dielectric on alumina substrate.

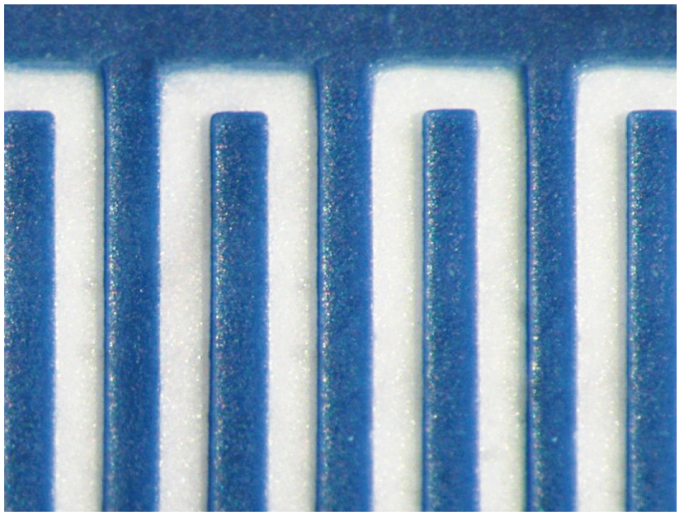

Fig. 3. Microchannel photoimaged in Fodel dielectric deposited on alumina substrate. Channel width 250 μm, depth 80 μm, total length 4.8 m (original magnification, 14×).

E. Lamination with LTCC

LTCC foil was used to cover the open photoimageable channel on top. Tests revealed that, for narrow channels up to 250 μm wide, the stretched LTCC tape does not break during firing. Before it is applied as a covering, the LTCC foil should be preconditioned with thinner; afterward, excess LTCC foil should be carefully removed by drying in vacuum. Drying in vacuum also prevents bubbling of the foil during firing because it removes air from the channels.

Fig. 4 shows the sequence of the technological process in which the microfluidic structure made of Fodel and LTCC was fabricated. The sequence includes additional drying steps in vacuum and lamination with LTCC.

Fig. 5 presents a cross-section of the capillary channel. Strong mutual diffusion between LTCC and Fodel can be observed.

Ceramic Microsystem

The ceramic microsystem consists of the separation column and the flame ionization detector.

The separation column is 4.8 m long, 250 μm wide, and 80 μm deep, and it was photoformed by additive deposition of eight Fodel QM44F dielectric layers. The flame ionization detector consists of a combustor cavity of 5 mm in diameter and three channels for supplying the analyte, the fuel (hydrogen), and the oxidant (oxygen). Both components—the separation column and the flame ionization detector—are combined on a single piece of 96% alumina of dimensions 65 mm × 50 mm. Fig. 6 shows the open structure of the microsystem. The photoformed microstructure is covered with DuPont 951AT LTCC foil. Fig. 7 (on the left) presents the top view of this structure laminated

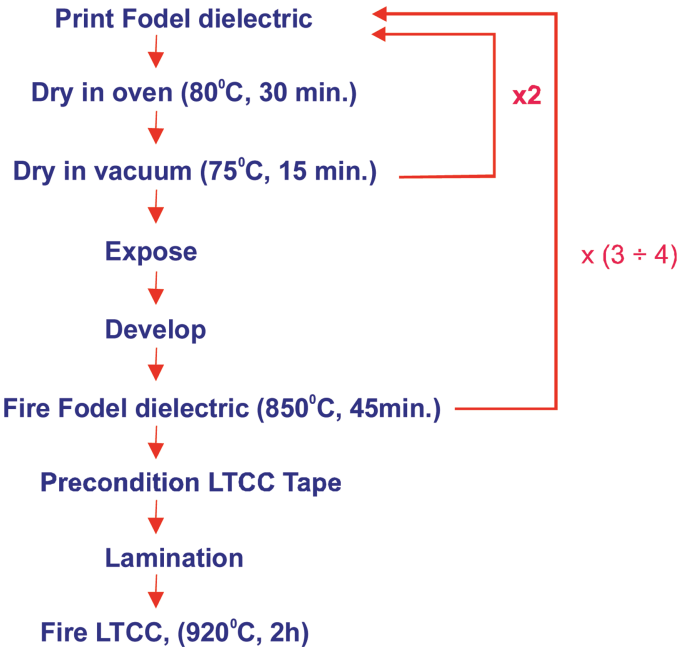

Fig. 4. Fabrication process of the microchannel photoformed in Fodel dielectric and covered with LTCC.

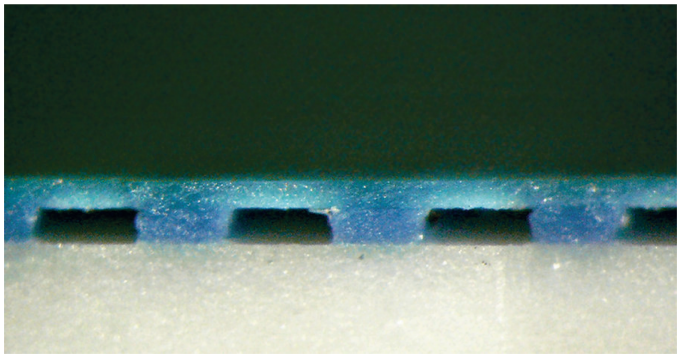

Fig. 5. Cross-section of the microchannel photoformed in Fodel dielectric and covered with LTCC.

with LTCC with an incorporated combustion cavity heater and the bottom view of the structure (on the right) where the platinum heater on the opposite side of the separation column can be seen.

The capillary column heater is a square platinum resistor of surface 2″ × 2″ printed with ESL D-5051 metallorganic composition. Three separately fired layers covered with ESL 4904 dielectric provide a resistance of 2 Ω. No trimming of the resistor was applied. The heater is located just under the separation column on the reverse side of the ceramic substrate.

The heater of the combustion cavity is a meander-shaped resistor printed with ESL 5545 platinum cermet composition. It is located on a small piece of alumina of dimensions 10 mm × 22 mm, the bottom side of which is covered with platinum and operates as an anode for the flame ionization detector. The arrangement of the separation column and the detector on the same substrate as well as heating facilities make the system especially useful when liquids are analyzed. Once a liquid analyte is vaporized in the separation column, it is not condensed on the pathway toward the detector. That is why precise temperature control along the entire pathway of the analyte is in this case absolutely necessary.

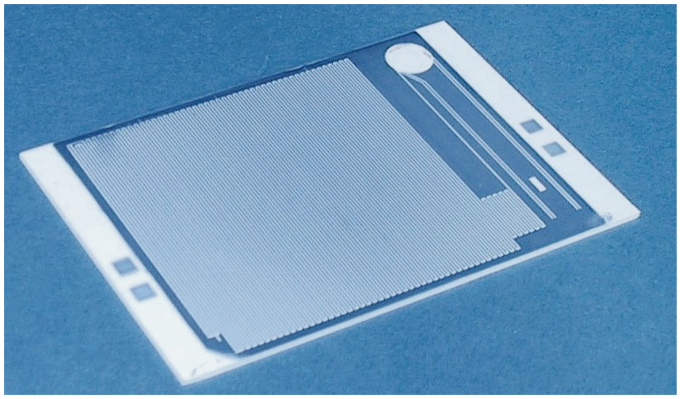

Fig. 6. Open microfluidic structure photoimaged in Fodel dielectric; the separation column and the flame ionization detector are incorporated on the same piece of alumina.

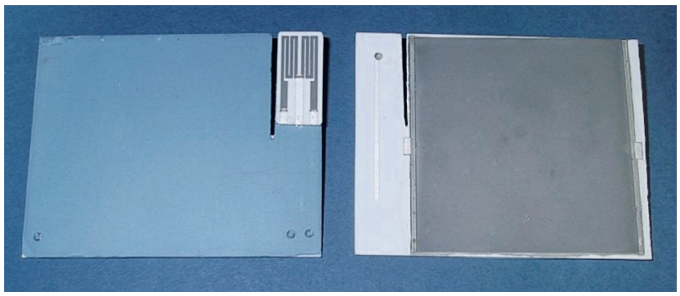

Fig. 7. Heaters of the microfluidic structure. (Left) Platinum meander heater of the combustion cavity. (Right) Platinum square heater of the separation column.

A slot of width 1.5 mm was cut by a diamond saw in order to separate the hot combustion zone of the ionization detector from the capillary column.

Measurements

Fig. 8 shows a test fixture for the microfluidic components. The fixture provides a sampling valve and a set of inlets and outlets for the media.

The ionic current between the electrodes of the flame ionization detector was measured after introducing a small portion of analyte into the separation column. At the beginning, the analyte was captured in the cavity of the sampling valve. After switching the valve, a small portion of analyte (approximately 1 μl) was pushed into the separation column. Helium at a pressure of 3 bar was used as a carrier gas. Flow controllers ensured hydrogen and oxygen flow rates of 30 and 20 sccm, respectively. The capillary column was heated up to 200°C with a platinum resistor on its reverse side.

Ethyl *tert*-butyl ether (ETBE) diluted in double-distilled water was used as the analyte. ETBE is commonly used as a gasoline additive to enhance gasoline performance.

Fig. 9 displays the ionic current for some samples of ETBE. Samples A, B, and C refer to ETBE diluted in water in ratios of

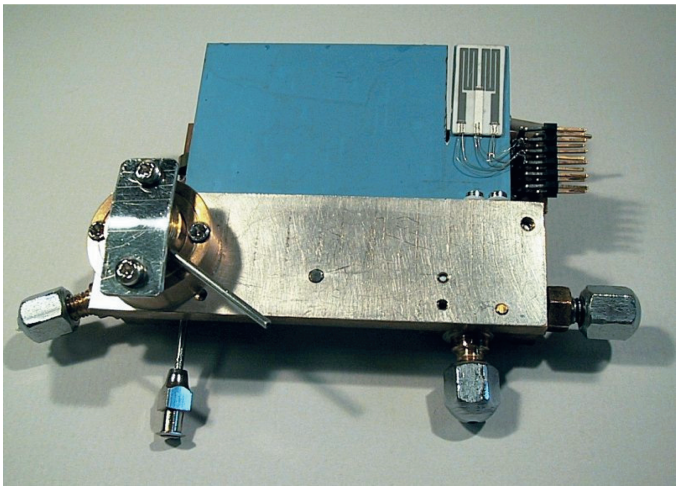

Fig. 8. Test fixture.

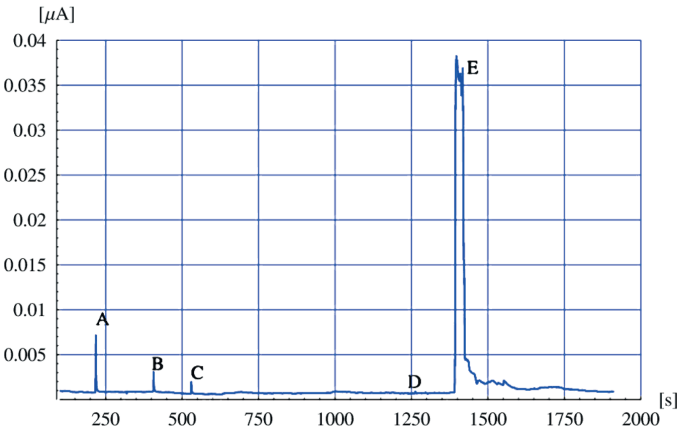

Fig. 9. Ionic current for the five samples of analyte Samples A, B, and C refer to ethyl *tert*-butyl ether (ETBE) diluted in water in ratios of 1:1000, 1:2000, and 1:4000, respectively. Sample D is pure water. Sample E is pure ETBE.

1:1000, 1:2000, and 1:4000, respectively. The samples were introduced to the column sequentially. Sample D was double-distilled water. Sample E was pure ETBE. Only a small pulse was recorded after sampling pure water into the capillary column.

Fig. 10 displays the stretched diagram from Fig. 9. In this figure, fluctuations of background current of the flame ionization detector can be observed.

At this stage of experiments, no stationary phase was applied for the ceramic capillary column.

Conclusions

The following conclusions have been reached:

Fine microchannel 250 μm wide, 80 μm deep, and 4.8 m long of rectangular cross-section was photoformed on the area 2″ × 2″ of alumina substrate with using thick-film photoimageable composition and LTCC.

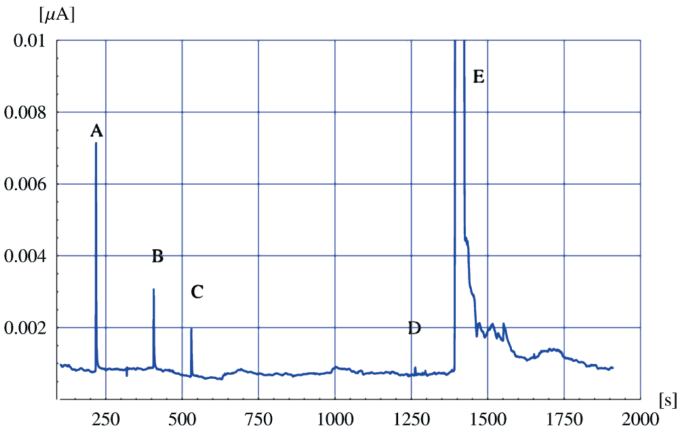

Fig. 10. Stretched diagram from Fig. 9. The small peak "D" indicates the presence of pure water.

Two basic microfluidic components manufactured in this technology—a separation column and a flame ionization detector—were combined on a single piece of ceramic.

Ionic current in the range of nA was detected by the flame ionization detector after introduction of a sample of the analyte into the separation column.

The design enables heating of the microfluidic components by platinum heaters.

If they become clogged, the ceramic microchannels can be easily reconditioned by refiring at 850°C.

It is possible to extend the capillary length by stacking ceramic substrates with capillary columns.

Further research is needed to choose the proper stationary phase for the ceramic capillary column.

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