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Dynamic behaviour and conditioning time of a zirconia flow sensor for high-temperature applications

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ABSTRACT

The temperature dependent ion conductivity of yttria stabilized zirconia (YSZ) can be used to create a miniaturized flow sensor using a calorimetric measurement scheme. Such a sensor is compatible with harsh environments, and can sustain temperatures of up to 1000 °C, although thermal crosstalk will limit its performance as the temperature rises. This paper investigates if the integration of thermal isolation in the form of sealed cavities can mitigate the detrimental effect of the thermal crosstalk, particularly by studying the conditioning time of the sensor to temperature changes. To this end, high temperature co-fired ceramic (HTCC) sensors were fabricated from tapes of 8 mol-% YSZ that were screen printed with platinum paste. Definition of channels and structures were made by milling the green tapes, and sacrificial inserts were placed in all cavities to give mechanical support during lamination and sintering. Cavities with widths of 240 μm, 400 μm and 560 μm were investigated, and sensors without cavities were also made to serve as references. Additionally, two different positions of the sensor element with respect to the edge of the cavity (560 or 800 μm) were investigated. The results showed that it was possible to improve the conditioning time of the sensor by up to five times by the use of isolating cavities, and that this improvement is translated into a reduction in rate-dependent hysteresis for measurements with long elapse times. The latter effect is most pronounced for the sensors with the largest cavities.

1. Introduction

Microsystems made from high-temperature co-fired ceramics (HTCC) have recently started to find application in sensors [1–5], actuators [6–8] and complete systems [9–11] intended for use in what is often somewhat crudely summarized as harsh environments [12]. For example, these encompass surroundings with highly elevated temperatures, pressures, radiation levels, chemical and mechanical stresses, etc., but environments with, e.g., highly reduced temperatures are similarly regarded as harsh. Hence, more detailed examples entail everything from the nozzle in a jet engine [13] to space [14]. It should also be pointed out that the harsh environment must not be external, but could be created inside the system itself, as is the case in highly integrated micro-rocket engines [7,10].

Material systems used in such applications include HTCC alumina (Al_2O_3) and zirconia (ZrO_2) combined with conductive materials that can be co-sintered, most often in the form of platinum [6,10,11,13] or tungsten [1]. Metals such as silver [2,3,15] and copper [11] are also used, but they have to be integrated after sintering of the ceramic. For zirconia, however, the combination of dielectric ceramics with electrical conductors has been shown to cause problems at highly elevated temperatures (>700 °C), partly due to thermal crosstalk, or rather spurious influence of parasitic elements of the sensor construction, at high levels of integration [16], but primarily due to the ion conductive properties which become increasingly apparent as the temperature rises [17]. An increase in electrical conductivity at elevated temperatures is found in a number of different engineering ceramics including SiC [18] and AlN [19].

The ion conductivity of engineering grade zirconia is caused by doping of yttria (Yr_2O_3) that is used to improve the mechanical properties of the material over large temperature intervals [20]. The resulting ceramic is often referred to as yttria stabilized zirconia (YSZ). The surface ion conductivity of YSZ is largely dependent on the partial oxygen pressure of the surroundings, wherefore it is frequently used in miniaturized gas sensors [21,22]. Moreover,

Abbreviations: HTCC, high-temperature co-fired ceramics; YSZ, yttria stabilized zirconia; DAQ, data acquisition; SEM, scanning electron microscopy.

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we have previously demonstrated that the exponential temperature dependence of the ion conductivity of YSZ can be used to create high-temperature flow sensors using a calorimetric measurement scheme [23]. The temperature dependence of the ion conductivity of this particular configuration is described in earlier work [17]. The resulting flow sensors were shown to be highly sensitive (>3 MΩ/sccm) and linear ($R^2 > 0.999$), but suffered from high power consumption (>1.5 W) and substantial spurious heat loss to the bulk of the devices.

The most common thermal flow sensors use thermoresistive conductors to measure the asymmetry of the temperature profile caused by the flow [24]. The calorimetric sensor in this paper has, like these sensors, an electrical heater on the bottom of a miniature gas channel, through which the flow is to be measured. Two sensor elements are positioned upstream and downstream of the heater, which measured the ion conductivity of the surrounding YSZ, giving a much more temperature sensitive signal compared to the thermoresistive change in the metal conductor. At zero flow, the conductivity upstream and downstream of the heater is identical, but when a flow is applied, the heat distribution of the gas in the channel is shifted towards the downstream sensor element, making its conductivity increase whereas the upstream conductivity decreases [23]. The response of the sensor corresponds to the difference in conductivity, or rather in resistance, between the upstream and downstream sensor elements, using the heater as a common ground. This response is linearly dependent on the flow as long as the heat transport by the gas dominated the change of the ion conductivity. However, if the sensor is operated at high flow rates (>10 sccm) or over long periods of time (>120 s), the flow not only affects the temperature distribution locally, between the sensor elements, but globally, by heating or cooling in the entire device. This effect causes the above mentioned thermal crosstalk making the sensor response for varying flow rates both less predictable and linear [23].

In this paper, we investigate if improved thermal decoupling of the active part of the calorimetric flow sensor can reduce the observed problems with thermal crosstalk, and simultaneously improve the sensors conditioning time. Therefore, the heater and sensor elements were placed on zirconia membranes of different sizes, and the resulting sensor responses were evaluated in terms of power consumption, conditioning time and rate-dependent hysteresis. In order to achieve this, a new fabrication process was developed and evaluated using scanning electron microscopy (SEM), X-ray, and surface profilometry.

2. Experimental

The design of the flow sensors used in this study bore great resemblance with those presented in references [17] and [23], although with one major difference: a sealed cavity was created beneath the active part of the sensor to supply thermal and electrical isolation from the bulk. Hence, the sensor was effectively confined on top of an YSZ membrane.

The active part of the sensor can be seen in Fig. 1. It consisted of four platinum conductors, 80 μm wide with a pitch of 160 μm, where the centre two were used as heaters and the outer two were used to study the ion conductivity by measuring the resistance of the surrounding YSZ, while using the heaters as common ground, Fig. 1. A 400 μm wide, 100 μm deep and 12.5 mm long channel was aligned perpendicular to the heaters, through which a gas flow could be conducted.

The cavity was aligned with the channel, Fig. 1. It had the same depth as the channel, and was made in three versions with widths, *w*, of 560 μm, 400 μm and 240 μm, all with a length of 2.1 mm. Furthermore, the position of the sensor elements along the cavity

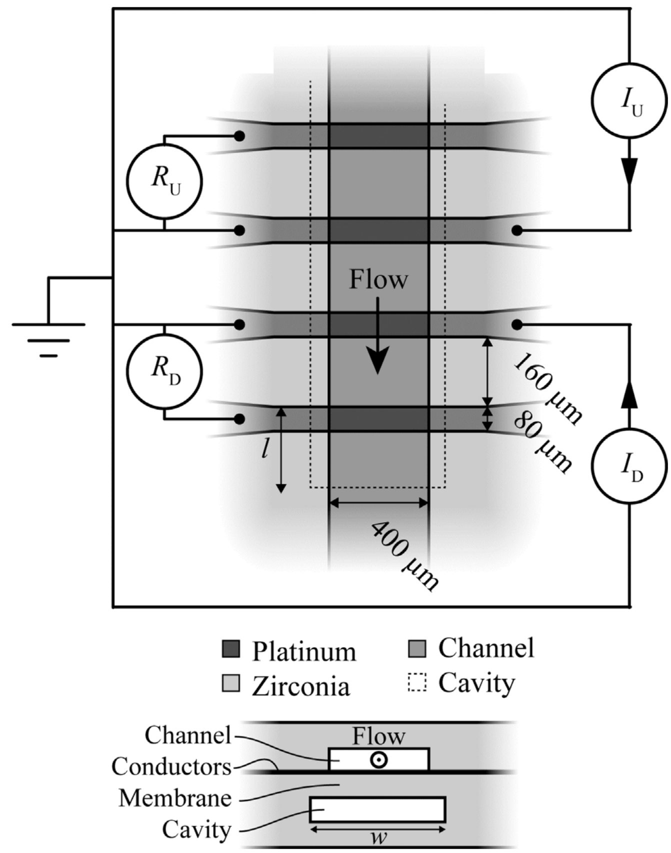

Fig. 1. Top view of the active part of the flow sensor (top) showing the channel, cavity and platinum conductors, as well as the configuration of the electrical connections to the DAQ and power supply. The bottom shows a cross section of the sensor perpendicular to the direction of the flow.

could be varied, where distances between the element and the edge of the cavity, *l*, of 560 μm and 800 μm were studied, Fig. 1. Finally, flow sensors without cavities were made to be used as references. A cross section of the sensor showing the channel and the cavity can be seen at the bottom of Fig. 1. All dimensions given above refer to actual lengths, i.e., after sintering during which the green YSZ shrunk by about 20%.

The flow sensors were made from 125 μm thick HTCC ceramic green tapes, consisting of 8 mol% YSZ (ESL 42401-G, ElectroScience Laboratories, USA). The sensors consisted of five tape layers: a bottom layer for mechanical support (I), the cavity layer (II), the membrane layer that also carried the platinum conductors (III), the channel layer (IV) and a top layer in which the gas inlet, outlet and electrical vias are made (V), Fig. 2(a).

The fabrication of the sensors was somewhat different from earlier versions, [17,23], where, e.g., the YSZ tapes were patterned by milling instead of embossing, lamination was performed under isostatic conditions, and the alignment of the layers was greatly improved by the use of alignment pins. However, like before, the heaters and conductors were made by manually screen printing of platinum paste (ESL 5571-G, ElectroScience Laboratories, USA) with a 325-mesh screen (Laser Technical Services A/S, Denmark). The paste was dried for 15 min at 50 °C before further handling. 5571-G paste is specifically designed for use as heaters in planar sensors and therefore it was used for this sensor instead of ESL 5542 used in earlier versions. The screen was designed so that 4 samples could be printed in each run. The milling of the cavities, channels, gas inlets and outlets, electrical vias and alignment holes were done on the green tapes using a PCB plotter (Protomat S100, LPKF, Germany). Fugitive inserts with the same shape and size as