Alumina-based monopropellant microthruster with integrated heater, catalytic bed and temperature sensors

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Abstract. A liquid propellant alumina microthruster with an integrated heater, catalytic bed and two temperature sensors has been developed and tested using 30 wt. % hydrogen peroxide. The temperature sensors and the catalytic bed were screen-printed using platinum paste on tapes of alumina that was stacked and laminated before sintering. In order to increase the surface of the catalytic bed, the platinum paste was mixed with a sacrificial paste that disappeared during sintering, leaving behind a porous and rough layer. Complete evaporation and combustion, resulting in only gas coming from the outlet, was achieved with powers above 3.7 W for a propellant flow of 50 µl/min. At this power, the catalytic bed reached a maximum temperature of 147°C. The component was successfully operated up to a temperature of 307°C, where it cracked.

1. Introduction
For precise attitude and positioning control of small satellites, scale appropriate propulsion systems that can provide thrust in the micro to milli Newton range are needed. A number of different principles, including electric microthrusters, solid propellant microthrusters, cold gas microthrusters, vaporizing liquid microthrusters, and monopropellant as well as bipropellant microthrusters, have been explored [1]. Cold gas microthrusters made in silicon have already been tested in space missions controlling the positions of CubeSats and microsatellites [2-5]. However, the thrust from cold gas microthrusters is limited to about 10 mN to 55 mN [1, 6], and there is a risk of gas leakage of the highly pressurized gas, especially during long missions, where also very small leakages can be fatal.

An attractive alternative that can give thrusts in the range of 10 to 200 mN [1] is to use liquid propellants that can decompose, such as hydrogen peroxide or hydrazine. The continuous operation temperature of silicon and Pyrex is limited, and wall temperatures of liquid propellant microthrusters can be as high as 630°C or above [7]. In addition, chemically active liquid propellants are also highly corrosive. Silicon, e.g., is attacked by hydrazine [8], making it necessary to use more sustainable materials like ceramics.
Compared with silicon, the thermal conductivity of ceramics like alumina is approximately 5 times lower, resulting in reduced thermal losses. 3-D shapes can be made using ceramic green tapes, i.e., flexible sheets made from ceramic powder mixed with a polymer binder. Embossing, laser cutting or milling are used to shape the sheets, and conductors are screen printed with metallic paste before the sheets are stacked, laminated, and finally sintered, making it possible to create multilayered structures with integrated conductors. This technique has been used to make different types of ceramic microthrusters, like vaporizing liquid propellant microthrusters, where water has been used as a propellant [9, 10], for a gaseous bi-propellants microthruster [11], and a solid propellant microthruster [12].

Although the corrosion resistance and high-temperature endurance make ceramics suitable for liquid propellant microthrusters that use chemically active propellants, there are few published examples. By integrating a catalytic bed initiating the decomposition, the chemical energy stored in reactive propellants like hydrogen peroxide and hydrazine can be released. Although, not showing any results from a realized component, Plumlee et al. [13] present simulations showing that the temperature in the walls of a ceramic thruster can exceed 800°C, using hydrogen peroxide as the propellant. Wu et al. have made a low-temperature co-fired ceramic thruster, where the decomposition of a hydroxylammonium nitrate liquid propellant was used [14]. There have also been attempts to use hydrogen peroxide in a silicon-based microthruster, but there have been difficulties of achieving complete chemical decomposition of the propellant within the thruster, probably because the temperature was not high enough because of thermal losses [15]. To overcome the excessive heat losses on small scale, a mixture of 90% hydrogen peroxide with an addition of ethanol in a glass microthruster has been evaluated, and it was shown that the stability of the average thrust was improved using the blended propellant [16].

In this paper, a liquid propellant microthruster made in alumina with an integrated platinum catalytic bed to decompose hydrogen peroxide is presented. A resistive heater and two resistive temperature sensors have been integrated into the thruster, making it possible to control and monitor the firing.

2. Design and manufacturing

The microthruster is comprised of five integrated parts: a platinum heater, a resistive platinum temperature sensor close to the heater, a catalytic bed, a catalytic chamber with a micronozzele, and a second temperature sensor placed above the catalytic chamber, figure 1. A divergence half angle of 28° was used for the nozzle since literature suggests a value in the range of 25°-30° [15, 17]. The catalytic bed and the heater have the same size, 7.5 × 4.3 mm before sintering, and overlap fully.

The thruster was built by stacking four layers of 2.5 × 2.5 cm large alumina tapes (150 µm thick, ESL 44007-150, Electro Science Laboratories, USA) that had been structured individually, using milling, and screen printed with platinum paste (ESL 5571, Electro Science Laboratories, USA). For the catalytic bed, the platinum paste was mixed with a sacrificial paste (ESL 4441, Electro Science Laboratories, USA) contributing to 10-15% of the volume, in order to increase the surface area of the structure after sintering. A graphite sacrificial insert that forms volatile products during sintering (ESL 49000, Electro Science Laboratories, USA) was placed in the catalytic chamber. A detailed description of the manufacturing procedure used has been given in [18].

As a last step before sintering, the contacts pads from the heater and the temperature sensor on the bottom of the components were connected to pads on the top by depositing platinum paste over the edge of the component. Some components lacking the top layer, thus leaving the catalytic bed exposed, were also made.
Figure 1. Exploded schematic of full microthruster chip, showing, bottom up: floor layer with heater and temperature sensor on backside, catalytic layer, chamber wall layer, and ceiling layer with leads and temperature sensor on top.

Before characterisation, the electrical contacts of the chip were connected to copper wires (200 µm in diameter), using conductive epoxy (CW2400, Chemtronics, USA), and a coned Nanoport (10-32 IDEX Health & Science, USA), was glued to the inlet using epoxy glue (EPO-TEK 353 ND, Epoxy Technology, USA).

3. Characterization
The complete thrusters were X-rayed (XT V 130, Nikon, Japan) before their temperature sensors were calibrated in an oven using a K-element thermocouple as a reference. MATLAB code was used to control the power supply (TTi QL 355TP) connected to the heater, as well as the digital multimeters (Keithley 2010 and HP 34401) that monitored the resistance of the temperature sensors through 4-point measurements.

The open components were used to study the catalytic bed during initial decomposition tests where the beds were loaded with 10 µl large drops of propellant using a micropipette. These components were also operated dry.

Using 1/16" PEEK tubing, the nozzle inlet of the complete component was connected to a gas tight 5 ml glass microsyringe (VWR International) mounted in a syringe pump (PHD 2000, Harvard Apparatus), and the nozzle exit was monitored using a Nikon SMZ 800 microscope. The syringe was filled with a hydrogen peroxide solution, (30 wt. %, Sigma-Aldrich), and a flow rate of 50 µl/min was maintained. The voltage used to heat the thruster was ramped from 0 V in steps of 0.5 V/min with dwells of 5 minutes at 13, 15, 17 and 19 V.

4. Results
Figure 2 a shows an X-ray picture of the thruster after sintering. The catalytic bed, which has the same dimensions as the heater, exhibits a coarse, porous platinum structure, figure 2 b, increasing the surface area, as the result of mixing the platinum paste with a sacrificial paste before screen printing. When hydrogen peroxide was added as droplets to the open version of the thruster, immediate decomposition was observed already at room temperature. The thruster outlet of the complete nozzle after sintering is shown in figure 2 c.
Figure 2. X-ray image of the full thruster chip (a), with the loops meeting at the centre of the meandering heater, being the temperature sensors. To the left, 3 wider leads, making it possible to drive only half the heater at a time, are seen. The porous structure of the catalytic bed (b). The nozzle after sintering has preserved its well-defined rectangular cross section (c).

For a thruster having the catalytic bed open and without feeding with any propellant, a voltage of 10 V, corresponding to a power of 2.7 W, resulted in a temperature of 230°C of the catalytic bed. Tests both with and without propellant flowing through thrusters showed that the temperature detected by the sensors on top and bottom of the thruster differ from each other with less than a few degrees. In order to reduce the wiring needed, only one of the sensors was therefore used for the actual firing experiments.

When the applied voltage was ramped from 0 to 19 V at a propellant flow of 50 µl/min, the temperature in the component first rose slowly, and then, when the temperature was around 110°C, it reached a plateau corresponding to the phase transition from liquid to gas in the thruster, figure 3 a. At this stage, the component started to exhaust a mixture of liquid and gas, and the component spat fiercely, being calmer and less prone to spit as the temperature was further increased. During the dwell at 13 V, the temperature rose to 147°C, and only vapor came out from the nozzle. The temperature of the component rose during the dwells, but to less extent on increasing voltage. By holding a glass slide a couple of centimeter from the thruster, the condensation of the vapor could be detected, figure 3 b. The sample cracked during the dwell at 19 V, at 307°C. The crack cut through the center of the component, along the direction of the flow, splitting the component in halves. This was a failure mode seen for several components, usually occurring in the region of 250 to 307°C.

Figure 3. During the dwells at 13, 15, 17, and 19 V, the temperature increased (a). With the thruster activated using a heater voltage of 15 V, corresponding to a temperature of approximately 225 °C, no vapour is seen near the nozzle exit but condensation is observed at the glass slide. The arrow points in the direction of the ejected gas, toward the condensate at a glass slide (b).
During the startup of the thruster, where the decomposition of the hydrogen peroxide is not yet complete, it showed to be important to avoid letting the propellant come in contact with the silver epoxy that covered electrical pads since they then were chemically attacked. Also, the gluing of the Nanoport showed to be a weak point.

5. Discussion and Conclusions
Successful firing of a liquid propellant hydrogen peroxide driven microthruster, having a catalytic bed made of porous platinum, has been demonstrated at temperatures up to 307°C, where the component finally failed by cracking. This temperature is lower than the 409°C that the zirconia microthruster designed by Cheah et al. [10] survived in dry stage. However, and more important, this temperature is much higher than the temperature of 80°C the same authors reported as being the temperature where their component cracked when it was operated with liquid. This shows the potential in the design presented here.

When a ceramic microthruster is fed with liquid propellants, the large temperature difference between the liquid and the component itself is a source of large thermal stresses. This can cause failure also at temperatures far from what the ceramic material in itself can tolerate. Failure caused by thermal stresses in ceramic microthrusters has previously been reported by several authors [10, 14, 19] and is something that has to be addressed already on the design stage.

By integrating temperature sensors, precise control of the decomposition can be achieved, and here the temperature was showed to be more or less equal on both sides of the component. A heater voltage of 10 V corresponded to a component temperature of 230°C in the dry state for a component having the catalytic bed open, but during firing of a complete thruster with hydrogen peroxide, energy is consumed for the phase transition, and the temperature is only slightly above 100°C at the same driving voltage. Since heat is actually generated when the hydrogen peroxide is decomposed, the reaction can be self-sustained once started, if the thermal losses are low enough and high concentrations of hydrogen peroxide is used [13].

Although not being the limiting factor in this work, it is clear that in order to reach higher operating temperature, there is a need to use more robust ways to interface the component since temperatures close to functional limit of the epoxy glue used both for the Nanoport and the electrical wires have already been reached. It is not only the temperature that puts strain on the glue, but also the fact that hot hydrogen peroxide is extremely aggressive.

Future work will focus on reaching higher operational temperature of the component and improve the interfacing. Part of this, entails using a higher concentration of hydrogen peroxide.

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