

# CERMIC MICROCOMPONENTS FOR HIGH-TEMPERATURE FLUIDICS

Ville Lekholm\*, Fredric Ericson, Kristoffer Palmer, Greger Thornell

Ångström Space Technology Centre,  
Department of Engineering Sciences, Uppsala University, Uppsala, Sweden

\*Presenting Author: ville.lekholm@angstrom.uu.se

**Abstract:** For aggressive environments, the material properties of silicon become a limitation. Macroscopically, ceramics are as abundant for high-temperature applications as is silicon in miniaturized systems, but this group of materials has been little exploited for MEMS components. A major reason is the lack of means for high-resolution structuring. This paper describes the application of silicon-based manufacturing processes in the fabrication of ceramic yet truly micromechanical structures and devices for very high-temperature applications, and demonstrates the technique's implementation in, and significance for, high-temperature microfluidics. Embossing of structures down to 2  $\mu\text{m}$  wide is demonstrated, as well as deep embossing (50  $\mu\text{m}$ ), punching through 15  $\mu\text{m}$  tape, and lamination of structured layers. The resulting samples survive temperatures of 1400°C.

**Keywords:** microfluidics, high-temperature, ceramics, processing, HTCC

## INTRODUCTION

With MEMS devices seeing a tremendous increase of use, both in everyday consumer electronics as well as specialty applications, the components are subjected to a wide variety of operating environments, few of which are really aggressive, however. Also in the case where no respect is paid to corrosion, i.e. when the surroundings are chemically neutral and dry, the use of most MEMS is limited to rather low temperatures. One reason for this is the frequent bundling, or even integration, of electronics which take offence already at a few hundred degrees centigrade. However, even bulk passive, purely mechanical silicon devices are seldom reliable further than another few hundred degrees above this, as the material starts to soften and accommodate to external loads and residual stresses, hence losing its tolerances and integrity.

There are many examples of applications (automotive, aerospace, process industry, ...) which should benefit from microdevices made from a more creep-resistant material than silicon. So called cold-gas microthrusters are attitude-controlling rocket engines beginning to appear on small satellites, offer an interesting example. Despite their name, they often rely on heaters in the stagnation chamber to increase the specific impulse, ISP, i.e. the efficiency of the thruster [1]. Higher temperature in the heaters would decrease fuel consumption and reduce the size and mass, and thereby the launch cost, of the spacecraft.

With applications like this in mind, and the intention to profit from the well-developed toolbox of silicon technology, the potential of the high-temperature 8 mol% yttria-stabilized zirconium oxide (YSZ8) ceramic is investigated in this work.

Unsintered, in the form of green tape, it is embossed and punched with dry-etched silicon tools, but also laminated and metallized, in order to explore its suitability for high-temperature microfluidics. A number of structures and patterns were designed, fabricated and tested in order to extensively, yet

preliminary, evaluate the suitability of the material, both as a substrate and as an active part of the component.

## MATERIALS AND METHODS

The ceramic was worked on in its green tape form, and fired as a last step in the fabrication. The tapes used were ElectroScience Laboratories (ESL) 8 mol-% yttria stabilized zirconia of thicknesses 15 and 125  $\mu\text{m}$ , (product 42400 and 42401, respectively). An Entech ECF 20/18 furnace was used for firing in air at 1450°C. The silicon tools used for structuring of these tapes, were fabricated using standard UV lithography followed by deep reactive ion etching (DRIE) of polished silicon. This allowed for high-resolution structuring of the wafer into sharp and elaborate contours, which could be pressed to the tape with a Bungard RMP 210 multilayer press. The same equipment was used for laminating layers of YSZ8-tape.

Embossing was accomplished by imprinting structures of different depths (10  $\mu\text{m}$  and 50  $\mu\text{m}$ ), widths (ranging from 5  $\mu\text{m}$  to 5 mm) and geometries, with a pressure on the tools of 10 MPa at room temperature on both tapes. Using the same technique, the possibility of punching holes by patterning tall and narrow ridges was studied on the 15- $\mu\text{m}$  tape. The same temperature and pressure was used, but with 50  $\mu\text{m}$  deep and 100  $\mu\text{m}$  wide structures.

In order to evaluate the mechanical properties of the fired ceramic, 5 mm diameter membranes and 1 and 2 mm long, 0.5 mm wide cantilever beams were fabricated. These could represent structures used in pressure and force sensors. For maximum pliability, these structures were fabricated from the thinner tape.

As channels are essential elements in microfluidics, 5 mm long, 200  $\mu\text{m}$  wide and 50  $\mu\text{m}$  deep ones were made from 125  $\mu\text{m}$  thick tape, and sealed with 15  $\mu\text{m}$  thick tape – both in their green state. These channels were verified by pressing ink

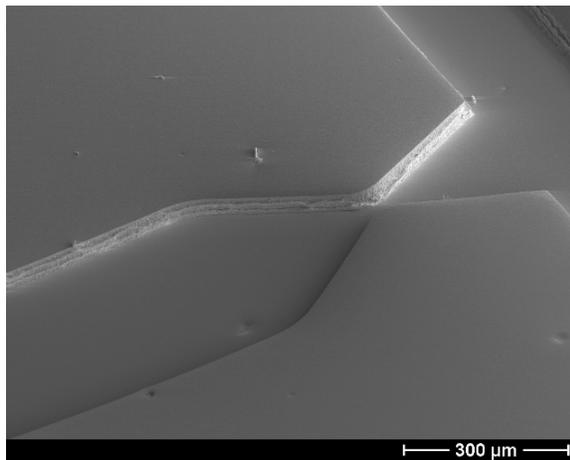
through the inlet. More specific to the application described above, nozzles for microthrusters were made. All lamination was performed with 21 MPa pressure at 70°C.

For many basic functions, e.g., conductors, strain gauges, vias, heaters, metallization of the material is necessary. As a first step, screen printing of platinum paste (ESL 5574) was tested on 125 μm tape. The paste was cofired with the tape.

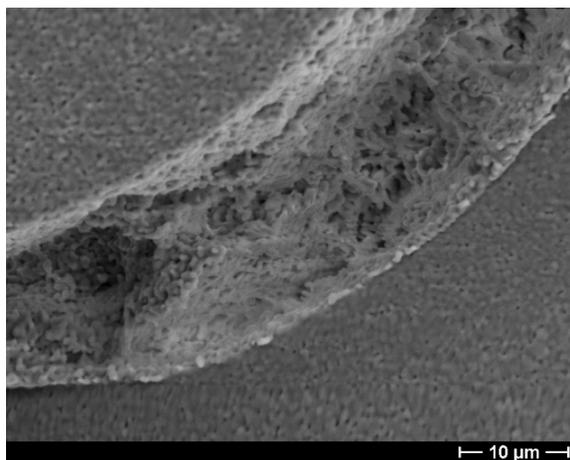
The samples were studied using SEM imaging and optical microscopy. The embossed beams were studied using a micromanipulator [2], to examine

## RESULTS

Figure 1 shows one half of a micronozzle embossed to a depth of approximately 30 μm after sintering, and a close-up of the side wall in the nozzle throat. For comparison, Figure 2 (a) shows the side walls after shallow embossing (7 μm) of a similar structure.

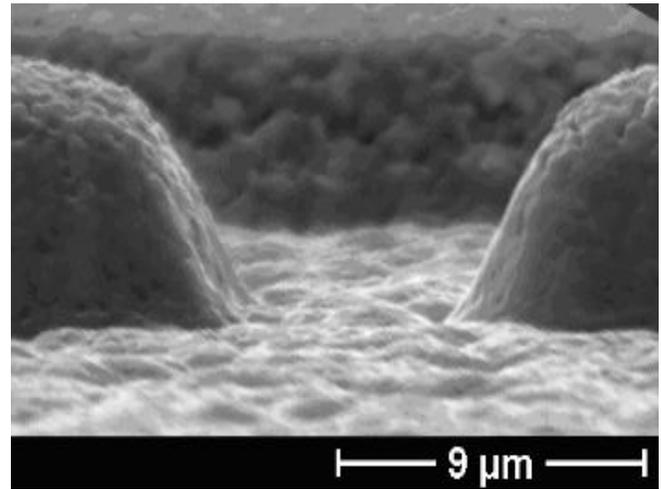


(a)

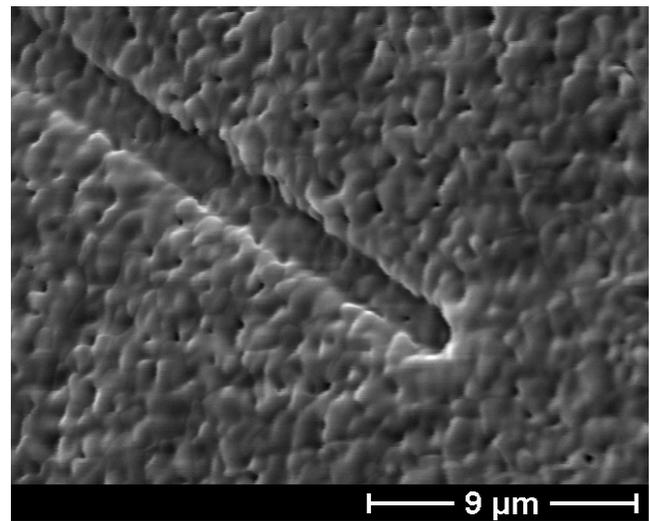


(b)

*Figure 1: SEM images of embossed structures in fired YSZ8. (a) Deep embossing of a micronozzle structure. (b) Close-up of its throat side wall. Top left is upper surface, bottom right is lower surface. Samples are tilted 45°*



(a)

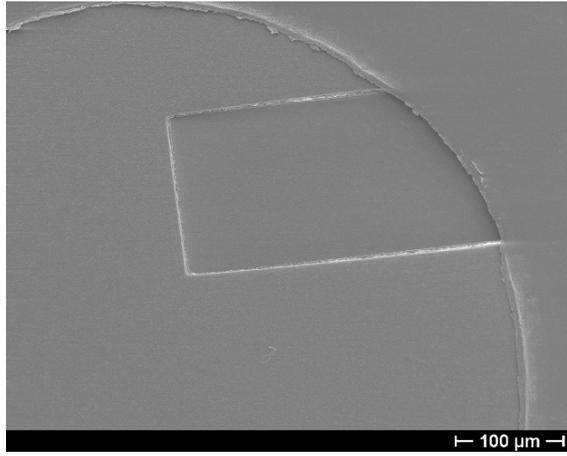


(b)

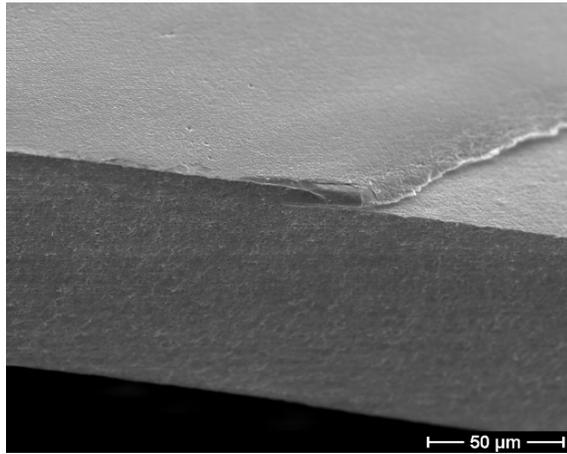
*Figure 2: Shallow embossing. The sample in panel (a) is tilted almost 90°. (b) Top view of narrow point.*



*Figure 3: Optical microscope image of an 800 μm long, 400 μm wide and 12 μm thick cantilever beam. Dimensions after firing. A pin is placed at the tip of the beam.*



(a)



(b)

Figure 4: SEM images of laminated YSZ8 tapes. (a) shows a 1 mm diameter hole punched in 15  $\mu\text{m}$  tape, laminated on top of a channel embossed in 125  $\mu\text{m}$  tape. (b) shows a cross section of the same sample.

Measurements were made on the punched beams, Figure 3, using a micromanipulator [2], where, using a force below 1 mN, the tip of the beam could be deflected approximately 100  $\mu\text{m}$  before fracturing.

Figure 4 (a) shows a 15  $\mu\text{m}$  tape with a punched hole, laminated onto a 125  $\mu\text{m}$  tape with an embossed channel. Figure 4 (b) show a cross section of the same sample. Channels fabricated in this manner have been verified to remain open after sintering.

The results of screen printing of platinum paste after firing is seen in Figure 5.

## DISCUSSION

This initial study on yttria-stabilized zirconia shows that it is a promising candidate for very high temperature MEMS components, although there are challenges. The tapes used shrunk approximately 18-26%, which, even though it can be easily compensated for in the design, may make integration with other materials challenging. Handling of the tapes, especially the thinner one, was very difficult, both in

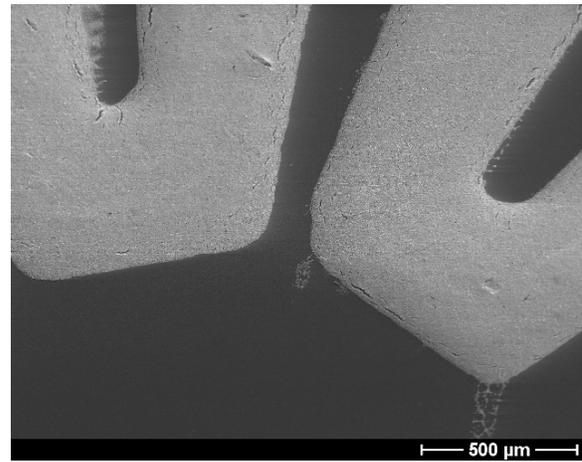


Figure 5: SEM image of screen printed platinum conductors cofired on 125  $\mu\text{m}$  YSZ8 tape.

green state and sintered. Extreme care needs to be taken in every step.

Embossing structures with a resolution of a few micrometers is possible with relatively straightforward methods and common clean-room equipment. Hence, silicon-based MEMS fabrication can be used to create tools, and only a few steps need to be added for transfer of the shapes to the tape. The differences between shallow and deep embossing can be seen in Figure 1 (b) and Figure 2 (a). Shallow embossing appears to deform the surface, whereas deep embossing seems to dislocate material from the edges – almost as if torn. This gives somewhat rougher side walls with deep lamination. However, the side walls remain close to vertical, regardless of depth.

The resolution of the load cell in the micromanipulator was insufficient to reliably measure the applied loads on the tested beams before fracture. However, the order of magnitude was in line with what listed values of 215-266 GPa [3]. The elasticity, and ultimately the fracture strain, of the material at this scale is important for the fabrication of for example pressure sensors, which rely on deformation of the material.

Lamination of several layers of tape was easily accomplished using the equipment used also for embossing and punching, but this time at increased pressure and temperature. Notice in Figure 4 (b), the combined thickness of one layer 125  $\mu\text{m}$  tape and one layer 15  $\mu\text{m}$  tape after firing is about 80  $\mu\text{m}$ , which is approximately 25% less than expected, due to the pressure in the lamination stage. The embossed channel was roughly 40  $\mu\text{m}$  deep before lamination and firing, but less than half this depth in the final component.

Much remains to be tested with regard to metallization of YSZ, but preliminary tests with screen printing of platinum paste shows great promise. The metal exhibits microcracks, but whether this is from drying of the paste, or from firing, is unknown. At this

time, the limiting factor with this process is the resolution of the stencils used. In this case all structures were 300  $\mu\text{m}$  or wider.

Further studies are needed to fully detail the processing steps, particularly regarding metallization. Currently, this is the only remaining part of a truly monolithic ceramic microcomponent capable of surviving 1400°C.

## CONCLUSION

Yttria-stabilized zirconia proved to be a very useful and promising material with respect to processing, and a number of processing steps have been developed and successfully demonstrated.

- Embossing structures as small as a few microns is achievable.
- Embossing has been demonstrated to work well, even at depths of up to 50 microns.
- Mechanical properties have been shown to be favorable for micromechanical components.
- Channels for microfluidics created by embossing and lamination remain unobstructed and sealed.
- Metallization is possible.

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