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MULTILAYER CERAMIC TECHNOLOGY APPLICATIONS TO MASS FLOW CONTROL

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Abstract: *Micro-fluidic devices suitable to MFC were fabricated using LTCC technology from the emerging field of Meso-Scale systems. A multiplicity of hybrid devices can be developed using this flexible technology because this is a hermetic and compatible material ideal for fluidic systems.*
Meso-fluidic devices as micro-valve, pressure, temperature sensor and critical orifices associated with fluidic interconnections can be integrated using LTCC technology in order to implement mass flow control systems for low flow and pressure ranges. We have measured several device parameters; micrographs of the fabricated devices and drawing of the proposed MFC are presented.

Keywords: *MST (Micro-System Technology), LTCC (Low Temperature Cofire Ceramics), Meso-systems, Sensors, Actuators, MFC (Mass Flow Control), MEMS (Micro-electromechanical systems).*

1. INTRODUCTION

The arrival of MST and MEMS techniques provide the means for Micro-system devices fabrication with decreased size and cost as well as increased performance and reliability. Chemical, semiconductor & biotechnology industries have increased needs for control and distribution of very low gas flows. Mass Flow Control systems suitable for this task must respond to the following requirements:

- Small sizes;
- Less dead volumes;
- Short response time;
- Minimum wettable surfaces;
- Corrosion resistant materials;
- Low power consumption;
- Non particulate generation;
- Non-moving parts that can block or clog the devices;
- Integration of several fluidic devices;
- High temperature operation
- Low cost.

Green ceramic tape technology (LTCC technology) displays excellent properties for doing packaging, interconnection and passive component integration. It has been used in the last twenty years for high reliability applications in military, avionics and automotive areas, as well as in MCM's (Multi Chip Modules) for portable wireless [Ref. 1] and computer applications. We have expanded its extent of application to the sensor and actuator area, rendering a technology suitable for MST.

In this work we would like to report sensors and actuators adequate for MFC systems exploring ceramic multilayer tape possibilities in the following ways:

- Fluid media realization of vias, holes, cavities, channels and manifolds;
- Critical orifices for flow control;
- Pressure sensors;
- Temperature sensors
- Actuators for hybrid Micro-valves & Micro-pumps.

2. LTCC TECHNOLOGY

For larger structures, in the intermediate size (meso-structures with minimum feature size in the range from 50 µm to several hundred µm, it would be desirable to have a material compatible with hybrid Micro-electronics, with the right thermal, mechanical and electrical properties, easy to fabricate and inexpensive to process.

Green ceramic tapes are such a material system [Ref. 2, Ref. 5, Ref. 6, Ref. 28].

Main reasons for using LTCC green ceramic tapes techniques as a MST technology are:

- Simplicity of tape machining with feature size of 50µm to several mm;
- Mass production methods can be immediately applied;
- Thermo-physical properties can be promptly modified, e.g. thermal conductivity;
- Tapes of different compositions can be formulated to obtain desired layer properties, e.g. magnetic permeability;
- Multilayer interconnections (electric or fluidic) can be easily outfitted;
- Embedded passive components is a normal feature;
- Integration of electronic circuits, due to it's hybrid nature, can be readily done;
- Layer count can be high;
- Possibility of auto-packed devices fabrication;
- Fabrication techniques are simple, inexpensive and environmentally benign.

Tapes are easily fabricated while still in the green, they are soft, pliable, and easily dissolved or abraded. Once the material is fired and fully sintered, it becomes tough and highly rigid. Small structures can also be carved and machined once fired using diamond tools.

Green Ceramic Tapes are glass-ceramic composite materials. The composition includes a ceramic filler, usually alumina, Al_2O_3, a glass frit binder to lower processing temperature and an organic vehicle for binding and viscosity control. This renders a material compatible with thick film technology, They are commercially produced in flat tapes of various thickness but usually in the range of 100 to 400 µm. called green ceramic tapes because they are manipulated in the green stage, that is before firing and sintering.

One of the important features of Green ceramic tape technology is the possibility of fabricating three-dimensional structures using multiple layers of Green ceramic tapes. Each layer is fabricated in the green (before firing) with whatever feature in the form of vias, cavities, channels, internal electrical elements (such as capacitors, resistors, and interconnections) are needed for the overall function of the 3D structure.

Then the individual layers are arranged in the proper order (stacked) as to yield the desired structure, placed in registry and laminated. The location holes for registry and the vias are usually punched, although they can be chemically dissolved, etched, abraded or cutted.

The next step moves this stack to the press where heat and pressure are applied to complete the lamination process. At this point the laminates are ready for sintering in air furnace. Complete processing sequence for green ceramic tapes is depicted in Figure 1.

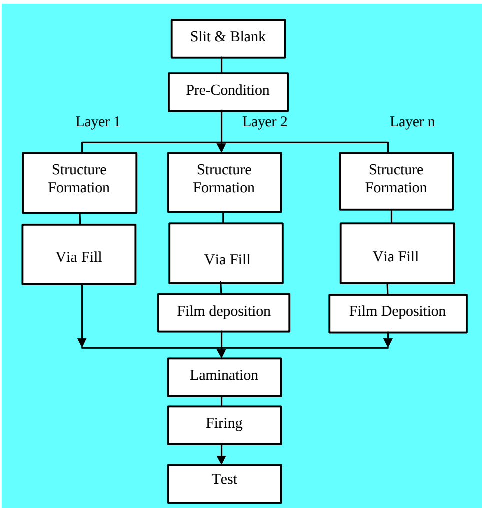


Figure 1. Green ceramic tape processing

The LTCC ceramics shrinks upon sintering or heat-treating. A nominal alumina LTCC formulation shrinks 12 % in the x, y plane and 15 % in the z-axis. Of course the shrinkage is uniform and predictable and one can incorporate the shrinking in the design scheme. The fracture strength of an alumina formulation is 320 MPa, which render the fired structure extremely tough. The porosity of the LTCC system is very low due to the use of vitreous material as alumina binder. The grain size and therefore the mean surface roughness of the fired ceramic are about 0.5 µm.

The thermal conductivity of a nominal fired alumina formulation is 3 W / mK, and the thermal expansion coefficient is 5.8 PPM/C.

The ease of forming both electronic and MST type hybrid structures is one of the great advantages of LTCC technology. A typical alumina formulation such as the DuPont's LTCC 951 can be glued after firing to most transparent glasses when viewing ports are desired in a structure. The hermetic binding of other hybrid structures is accomplished with die-bonding glass formulations, epoxies or eutectic bonding.

There are multiple metallization schemes that are shrinkage matched to the green ceramic tape and can be applied in the green. Metals such as Au or Ag (air fired), Cu (reducing or neutral atmospheres) are used for interconnections, electrodes and via filling. An ample gamma of resistors and dielectric formulations are available with complete shrinkage compatibility to the LTCC materials.

Due to the high fusion temperature of materials involved in green ceramics fabrication, a sintering process is needed to convert the green ceramic tape into a solid dense material. In Figure 2 a temperature profile for green ceramic tape sintering is displayed. This profile presents two plateaus. The first one is at 350^{o}C; in order to burn all organic components, the second is related to the viscous sintering process. Since the material is a composite glass-ceramic material the sintering is of the viscous flow type, see sintering model in Figure 2.

After sintering, green ceramics become a very stiff materials and partially resistant to glass leaching. After firing the system allows rework, it is possible to deposit thick films (cermets or polymeric) and perform further sintering at a lower temperature. Finally these materials can be machined using Laser or Diamond tools, in order to position internal structures or define its final shape.

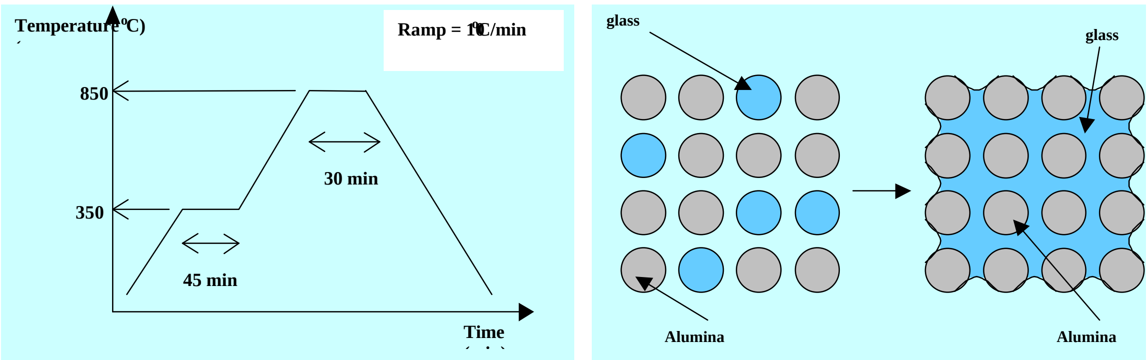


Figure 2. Temperature profile and sintering model for LTCC materials

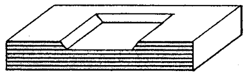
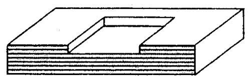
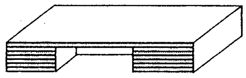
3. CAVITY REALIZATION FOR MST TECHNOLOGY

In MST or MEMS there is a need for three-dimensional devices. As in LTCC technology there are some steps to follow in order to fabricate cavities in multilayer applications:

- Machine cavity in tape or laminate;
- Use of some sagging control scheme [Ref. 7];
- Join patterned tapes and laminate parts;
- Co-fire laminate.

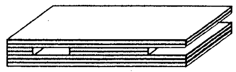
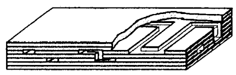
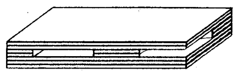
Top layer cavities found applications from sensor carrier to pressure sensor as shown in Table 1. Principal requirements are usually edge quality that can be assured using uniaxial or isostatic lamination.

Table 1. Top layer cavities in LTCC technology from [Ref. 3]

Cavity requirements	Application examples	View of set-up
no special requirements	sensor carrier actuator carrier gas sensor	
high edge quality	sensor carrier pressure sensor dies for innerlayer cavities	
thin membran under /over cavity	pressure sensor	

Inner layer cavities are used to implement sensor, cooling and Micro-fluidic applications as shown in Table 2. Some times it is possible to use insert techniques to solve lamination problems but for more complicated structures fugitive phase techniques are indicated.

Table 2. Inner layer cavities in LTCC technology from [Ref. 3]

Cavity requirements	Application examples	View of set-up
simple through holes (x- or y-direction)	flow sensor cooling functions	
small capillary tubes capillary systems	chemical sensor cooling systems microfluidic systems	
expanded innerlayer cavities	pressure sensor chemical sensor	

4. SOME MICRO-FLUIDIC DEVICES

LTCC technology displays simplicity to implement channels with internal reduced dimension as well as cavities without geometrical limitations when compared with other MST technologies .

In this section some basic Micro-fluidics applications using multilayer ceramic tapes will be presented, specifically micro-channels and critical orifices.

Micro-channels

First studies on micro-channels were performed by Poiseuille [Ref. 24] in 1846. He managed to do some experiments using glass capillaries of hundreds of microns. From this study the classical expression that relates pressure drop with volumetric flow rate was obtained. Micro-channels were studied by Pfahler [Ref. 23] and Harley [Ref. 16] for liquid and gas behavior on silicon substrates.

In Figure 3 it is possible to verify a simple way to implement micro-channels using green ceramic tape technology, in this case three layers are enough to fabricate the channel. Top layer makes media interconnection, middle layer makes the channel itself (that could be straight, in L, Y, U, spiral or any desired complex shape), bottom layer makes the device base, a Y micro-channel fabricated using green ceramic tapes and glass, it is also presented.

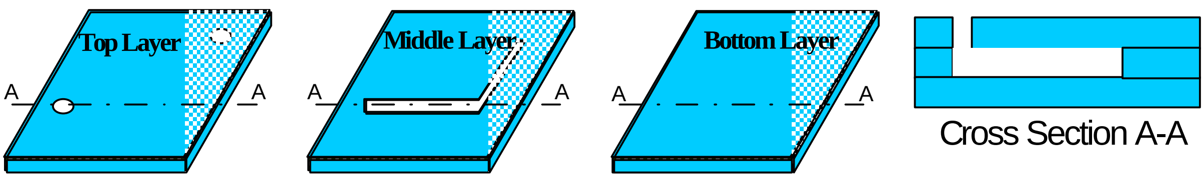
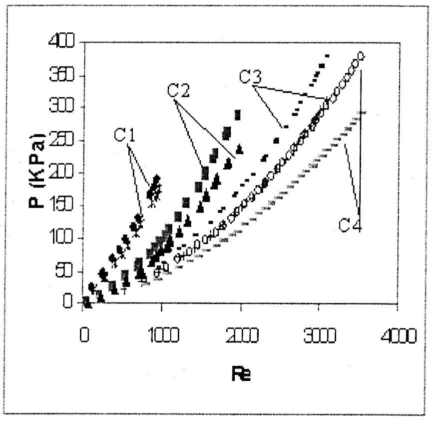


Figure 3. L micro-channel implementation using LTCC technology.

Measurements performed by Moon [Ref. 19] in straight conduits with dimensions shown in Figure 4 display linear pressure drop for the low Reynolds number region.



The Dimensions of the straight conduits used in the experiments and the Poiseuille number

Conduit's Designation	C1	C2	C3	C4
Conduit's Height (mm)	200	200	200	200
Conduit's Width (mm)	220	210	410	400
Conduit's Length, L (mm)	44	10.5	21.5	10.6
$L_1^+ = \left(D_H \mathrm{Re}_1\right)^{-1} L$	1.43	.78	.15	.05
$L_2^+ = \left(D_H \mathrm{Re}_2\right)^{-1} L$	.22	.026	.027	.011
Experimentally determined Po # at L_1^+	56.4	62.1	71.8	120.4
Theoretically determined Po # at L_1^+	58.1	58.8	70.9	87.8
Relative difference between theory and experiment (%)	3	5.6	1.3	37
Theoretical, fully developed, Po Number	57.06	56.94	62.45	62.19

Figure 4. Pressure drop Vs. Reynolds number for straight micro-channels

4.2 Critical Orifices

Critical orifices as nozzles are passive devices for gas flow control, using Choked flow phenomena. Choked flow happens when gas reaches sound velocity in the orifice passing cavity. At certain critical input pressure this phenomena arises and volumetric flow remains constant despite output pressure variations [Ref. 18, Ref. 22]. Critical orifices do not have moving parts and can control volumetric flow passively.

There are several micro-fluidics applications for these devices, Figure 5 shows a critical orifice fabricated using LTCC materials and machined using CNC techniques. In addition critical orifice behavior for input pressure vs. volumetric flow and different orifice diameters (90, 180, 210 μm) is displayed.

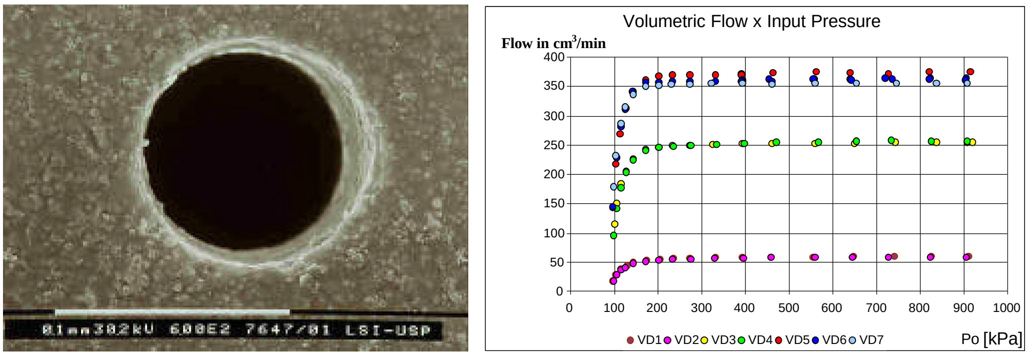


Figure 5. Critical orifices and its input pressure vs. flow behavior.

5. MESO-SCALE PRESSURE SENSOR

Pressure sensors with promising characteristics at high pressure and temperatures have been developed using conventional (LTCC) tape technology [Ref. 10, Ref. 20]. All parts for the transducer were machined from DuPont 951 series LTCC tapes utilizing either a numerically controlled milling machine or exfoliation.

The pressure is measured as a function of the membrane deformation where two piezo-resistors are screen-printed. Two piezo-resistors were used to achieve temperature compensation. Using shrinkage matched paste; nominal thick film technology was used in the screen printing of the piezo-resistors.

The rest of the sensor was fabricated using several layers of LTCC tapes, which were laminated and fired. Devices of different sizes have been fabricated and compared. Computer simulations of membrane deflection as a function of the vacuum load where obtained and used for the design and scaling of the piezo-resistors. Fabricated sensor as shown in Figure 6. has a 3.8 mil thick membrane and two screen printed piezo-resistances in order to sense radial and tangential strain they display very good dynamic response as compared to a silicon pressure sensor. For high-pressure sensors when space is not a drawback and good temperature stability is needed this technology is a good choice.

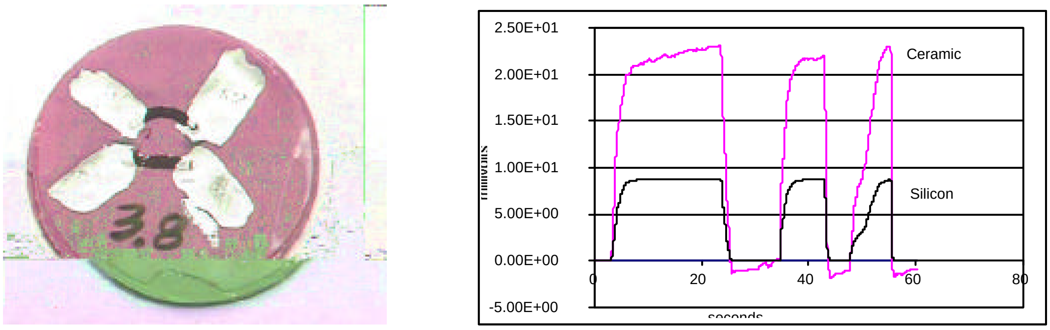


Figure 6. Fabricated Pressure Sensor and its dynamic response compared with a silicon one

6. TEMPERATURE SENSORS

A meso-scale gas flow sensor was fabricated as shown in [Ref. 9, Ref. 14]. The basic sensor structure consists of a thick film resistive heater and two thermistors printed on a thermally isolated bridge in a cavity.

The flow sensor embodies a gas temperature sensor shown in figure 8. A ceramic bridge in a cavity has two screen printed thermistors that measures the mean gas temperature, for testing purposes the heater has been excited with a electrical current. Figure 8 also display the measured bridge temperature for variable heater current excitation.

The device was fabricated following conventional LTCC technology process and sagging prevention method. NTC Thermistors pastes compatible with the green ceramic tape system used [Ref. 25] display good temperature sensitivity.

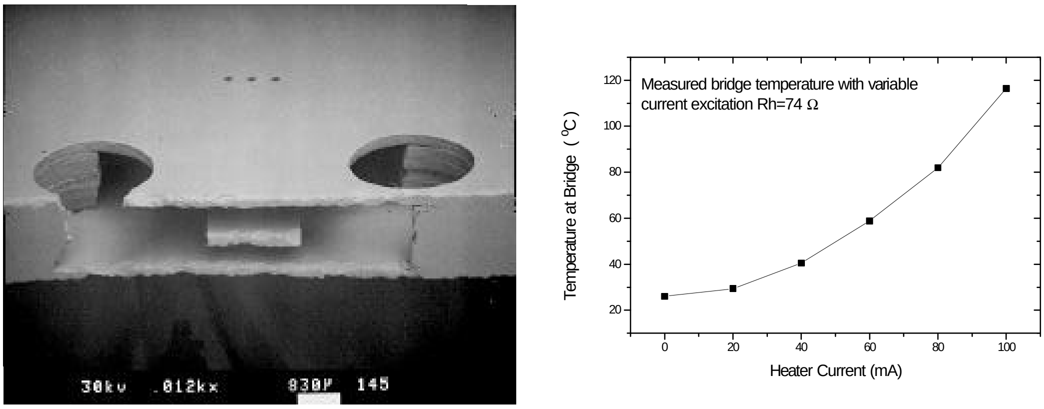


Figure 7 Cross section of a fabricated structure for the measurement of gas temperature in a cavity and bridge temperature response.

7. ACTUATOR APPLICATIONS

Actuators with adequate characteristics for aggressive environments and high temperatures have been developed using low temperature co-fired ceramic tape technology. We would like to report an electro-magnetically actuated normally closed valve [Ref. 13].

7.1 Hybrid Micro-valve

Micro-valves are necessary to execute fluid control functions in Micro-fluidic applications, some advantages of miniaturization of this devices are:

- Small sizes;
- Short response time;
- Low power consumption;
- Low inactive volume;
- Good dynamic characteristics.

Main problems with silicon Micro-machined valves are:

- Manipulation of biological fluids having cells or bacteria hundreds of microns;
- Moving parts used for Micro-valves can block or clog the devices.

In this work a non-moving parts hybrid electromagnetic Micro-valve, fabricated using LTCC, thick film and silicon technologies, is presented. Forces of magnetic origin can be generated by the interaction of a magnetic field intensity H with an electrical current I [Ref. 4, Ref. 26]. As a result of this force the spring generate a displacement proportional to the force divided by the equivalent spring constant *k.*

$$\Delta z = F_z \cdot k^{-1} \quad (1)$$

Electromagnetic techniques are suitable to hybrid meso-systems, [Ref. 11] because:

- Can generate large forces;
- Can produce large displacements;
- Good performance with temperature
- Adequate velocity response
- Is a robust and non expensive technique

Using some of LTCC possibilities a hybrid Micro-valve was implemented. This device has a multilayer coil, a fluidic system and a flexible diaphragm with a magnet bonded in its topside, associated to a media interface, as shown in Figure 9.

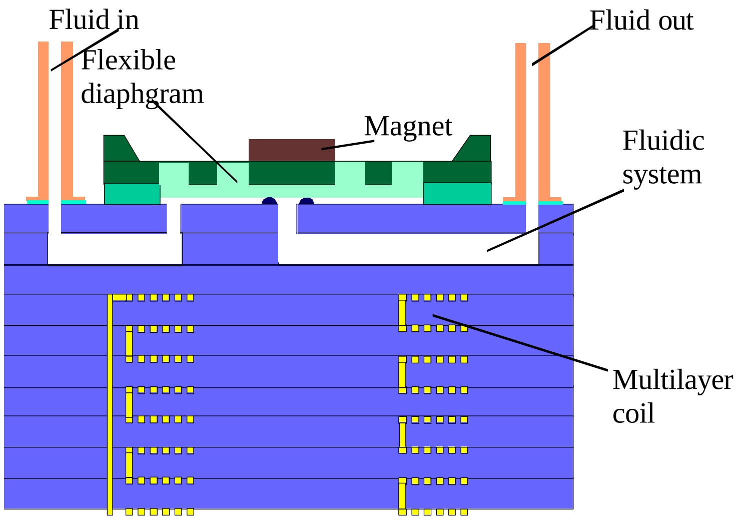


Figure 8. Conception of a hybrid Micro-valve.

Multilayer coil fabrication is described elsewhere [Ref. 12, Ref. 14], fluid system can be implemented, as pointed out in section 4.1, with three LTCC tapes as presented in Figure 9.

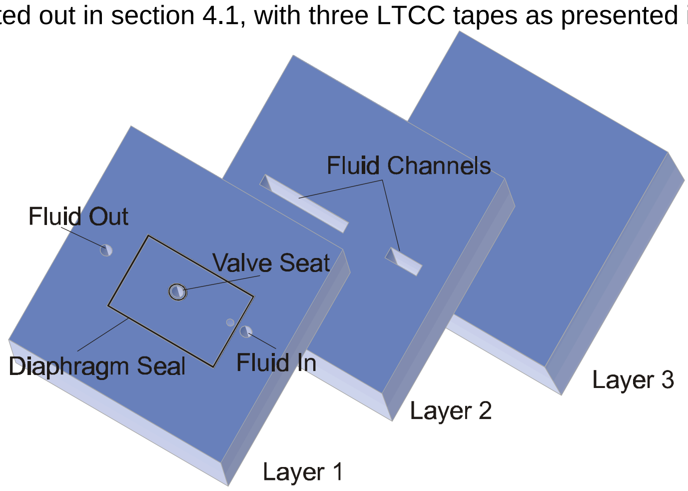


Figure 9. Micro-valve fluidic system

Flexible diaphragm with a rare earth magnet attached allows electromagnetic actuation, the diaphragm was implemented using silicon technology for a spiral spring that is covered with an RTV film. Fabricated flexible diaphragms using process described elsewhere [Ref. 11] are shown in the digital photographs of Figure 10, where one can see the complete devices after rare earth magnet bonding and displacement response Vs. coil current is also shown.

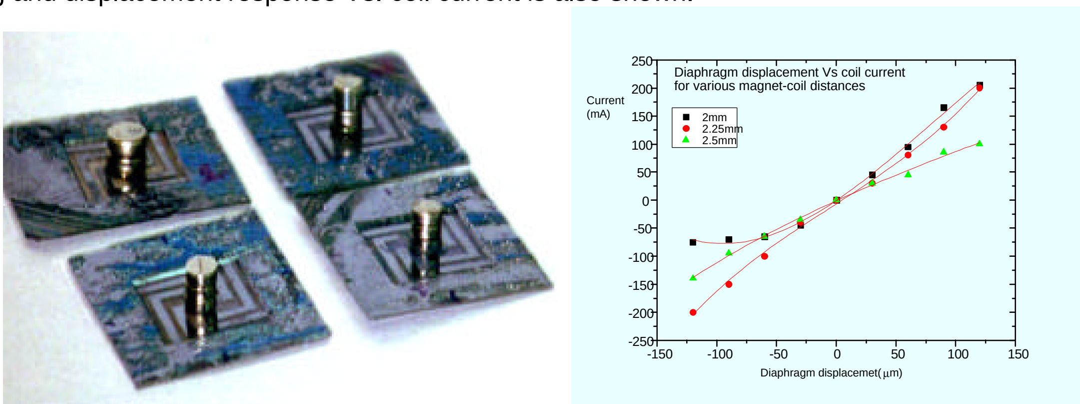


Figure 10. Flexible diaphragms fabricated and it's displacement vs. coil current

Complete fabricated Micro-valve is shown in Figure 11, This is a hybrid device which utilizes a purely LTCC tape electro-magnet and fluid flow manifold, combined with an anisotropically etched silicon rectangular planar spring, and a high-energy product SmCo mini-permanent magnet.

Device dimensions are in the meso (intermediate) range with the smallest features (fluid conduit in the manifold) of 400 micrometers and the largest (the electromagnet, coil) of 12 mm.

All parts of the electromagnet and the fluid flow channels were machined from DuPont 951 series, alumina based LTCC tapes utilizing either a numerically controlled milling machine, a puncher or an isotropic etching technique involving the glassy binder of a partially sintered LTCC tape. The hybrid device consists of 5 layers of planar spiral coils. The total coil resistance is high (120 Ohms) and thermal considerations limits the current to 150 mA.

Using a 900 Gauss SmCo magnet (1mm diameter) we obtained 200 micrometers deflection of the silicon 30 microns thick rectangular planar spring with a polysiloxane sealing element.

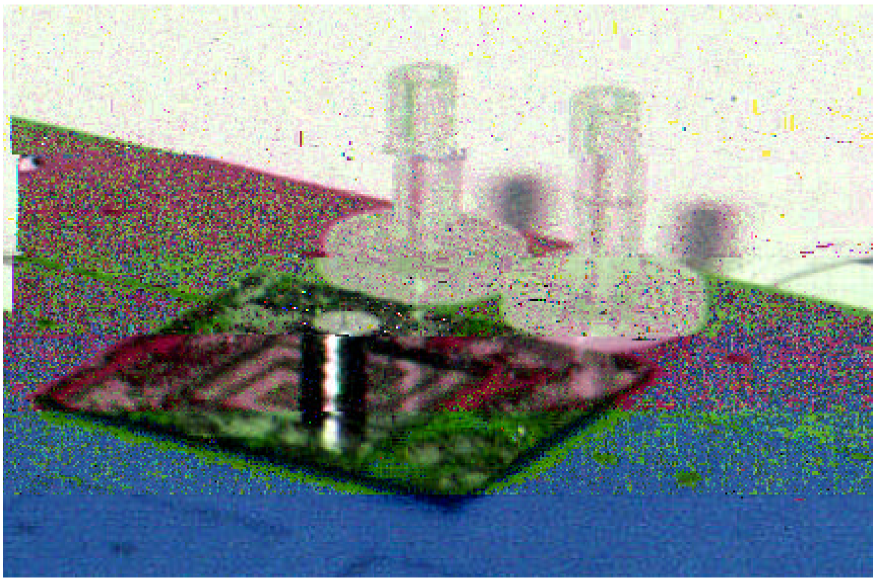


Figure 11. Hybrid Micro-valve fabricated

This is an inexpensive, easy to fabricate meso-scale valve fabricated in the same material as many IC packaging systems. We are currently developing a meso-scale pump using the same approach reported in this work. This may lead to fluidic systems where the fluidic devices can simultaneously serve as part of the IC package.

8. APPLICATIONS TO MASS FLOW CONTROL

Mass flow control devices are used to control gas flows within very tight specifications in the range of (<1 sccm to 30 slm). This device consists of a mass flow monitor and a servo controlled valve in a control loop as well as an electronic computation and controller circuits.

A widely used established method for mass flow control is thermal dispersion type [Ref. 20], the advent of MST and MEMS techniques [Ref. 17] has opened the possibility of implementation of several other methods.

In Figure 12 a block diagram of a complete inferential type [Ref. 26] mass flow controller is shown. Differential pressure sensing is used to measure pressure drop in orifice. Absolute pressure and temperature measurements give density measurement. This data is applied to computation circuits in order to get mass flow information necessary to activate the PID digital controller and compare with set point input. An output error signal is feedback to a servo controller valve in order to change internal pressure and match mass flow rate with the set point, within the appropriate resolution and accuracy specifications.

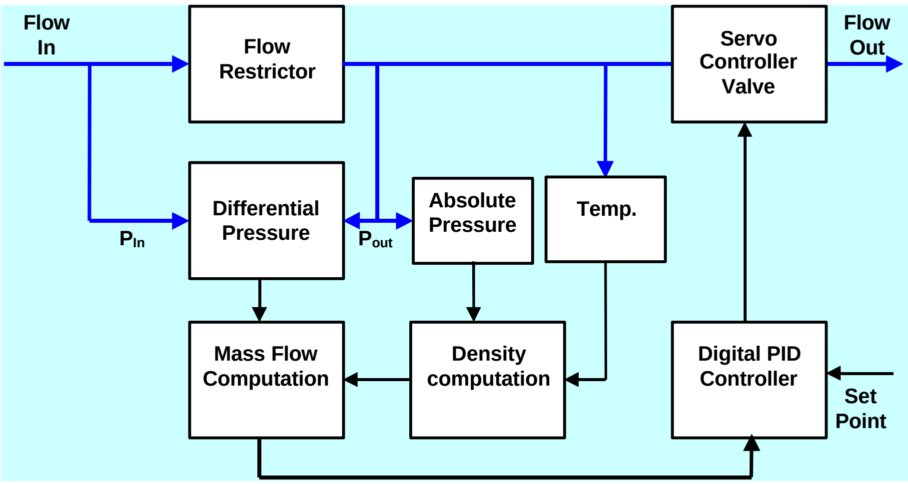


Figure 12 Block diagram of a typical inferential mass flow controller

As demonstrated in the latter sections, we can readily integrate several building blocks (pressure, temperature, valves) and integrate them, with microelectronic digital computation and control circuits using hybrid techniques, to obtain a meso-system suitable for gas flows control and distribution.

Several implementations of mass flow control devices have been studied [Ref. 17] in the subsonic and sonic flow regimes, using critical orifices which defines the flow range and resolution. The utilization of look-up tables during computation in order to compensate temperature dependent information leads to a simpler hardware configuration.

Figure 13 reproduce a drawing with the conception of an integrated LTCC multilayer ceramic meso-system, under development, for applications to mass flow control.

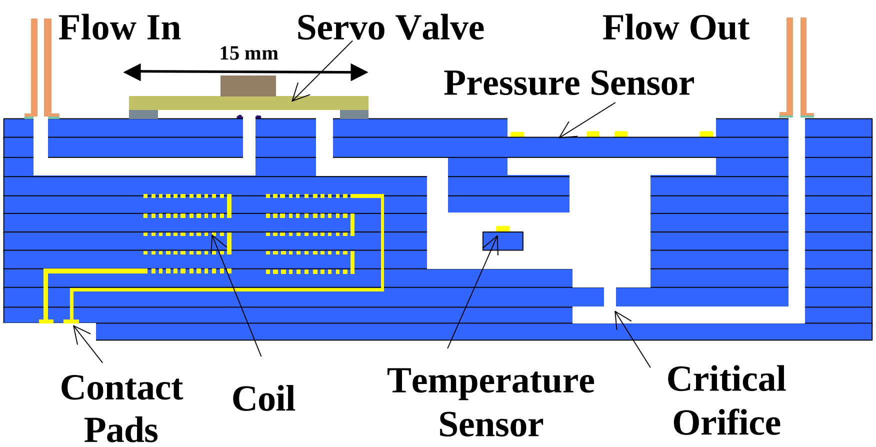


Figure 13 Conception of a LTCC meso-system for control and distribution of very low gas flows.

9. CONCLUSIONS

Meso-fluidic devices suitable to mass flow gas control system implementation have been developed using the LTCC multilayer ceramic technology.

Fabricated meso-fluidic devices as micro-valve, pressure, temperature sensor and critical orifices associated with a fluidic interconnection can be integrated using LTCC technology in order to accomplish mass flow control systems for low flow and pressure ranges

We have been considered and simulated some mass flow control configurations and early results indicate the feasibility of this approach

In this work we reported sensors, actuators and other fluidic components that can be suited for several fluidic control functions exploring ceramic multilayer tape possibilities; rendering integrated building blocks for more complex applications in the emerging field of MST.

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