



UPPSALA
UNIVERSITET

*Digital Comprehensive Summaries of Uppsala Dissertations
from the Faculty of Science and Technology 1424*

Extending Microsystems to Very High Temperatures and Chemically Harsh Environments

ZAHRA KHAJI



ACTA
UNIVERSITATIS
UPSALIENSIS
UPPSALA
2016

ISSN 1651-6214
ISBN 978-91-554-9686-9
urn:nbn:se:uu:diva-302658

Dissertation presented at Uppsala University to be publicly examined in Polhemsalen, Ångströmlaboratoriet, Lägerhyddsvägen 1, Uppsala, Monday, 31 October 2016 at 09:30 for the degree of Doctor of Philosophy. The examination will be conducted in English. Faculty examiner: Professor Frank Niklaus (Royal Institute of Technology (KTH), Stockholm, Sweden).

Abstract

Khaji, Z. 2016. Extending Microsystems to Very High Temperatures and Chemically Harsh Environments. *Digital Comprehensive Summaries of Uppsala Dissertations from the Faculty of Science and Technology* 1424. 45 pp. Uppsala: Acta Universitatis Upsaliensis. ISBN 978-91-554-9686-9.

Aiming at applications in space exploration as well as for monitoring natural hazards, this thesis focuses on understanding and overcoming the challenges of extending the applicability of microsystems to temperatures above 600°C as well as chemically harsh environments. Alumina and zirconia high-temperature co-fired ceramics (HTCC) with platinum as the conductor material, have in this thesis, been used to manufacture a wide range of high-temperature tolerant miniaturized sensors and actuators, including pressure and flow sensors, valves, a combustor, and liquid monopropellant microthrusters.

Interfacing for high temperatures is challenging. One solution is to transfer the signal wirelessly. Here, therefor, wireless pressure sensors have been developed and characterized up to 1000°C.

It is usually unwanted that material properties change with temperature, but by using smart designs, such changes can be exploited to sense physical properties as in the gas flow sensor presented, where the temperature-dependent electrical conductivity of zirconia has been utilized. In the same manner, various properties of platinum have been exploited to make temperature sensors, heaters and catalytic beds. By *in-situ* electroplating metals after sintering, even more capabilities were added, since many metals that do not tolerate HTCC processing can be added for additional functionality. An electroplated copper layer that was oxidized and used as an oxygen source in an alumina combustor intended for burning organic samples prior to sample analysis in a lab on a chip system, and a silver layer used as a catalyst in order to decompose hydrogen peroxide in a microthruuster for spacecraft attitude control, are both examples that have been explored here.

Ceramics are both high-temperature tolerant and chemically resistant, making them suitable for both thrusters and combustors. The corresponding applications benefit from miniaturization of them in terms of decreased mass, power consumption, integration potential, and reduced sample waste.

Integrating many functions using as few materials as possible, is important when it comes to microsystems for harsh environments. This thesis has shown the high potential of co-fired ceramics in manufacturing microsystems for aggressive environments. However, interfacing is yet a major challenge to overcome.

Keywords: HTCC, MEMS, MST, Microcombustor, Microthruster, Single-use valve, Wireless pressure sensor, flow sensor, in-situ electroplating, Monopropellant, Platinum

Zahra Khaji, Department of Engineering Sciences, Microsystems Technology, 516, Uppsala University, SE-751 20 Uppsala, Sweden.

© Zahra Khaji 2016

ISSN 1651-6214

ISBN 978-91-554-9686-9

urn:nbn:se:uu:diva-302658 (<http://urn.kb.se/resolve?urn=urn:nbn:se:uu:diva-302658>)

To my beloved mother and father

تقدیم به مادر و پدر نازنینم

*“Imagine yourself enjoying your better future
in such specific, believable detail that you soon
enjoy doing what takes you there!”*

-Spencer Jonson, Peaks and Valleys'

List of Papers

This thesis is based on the following papers, which are referred to in the text by their Roman numerals.

- I Sturesson, P., **Khaji, Z.**, Knaust, S., Klintberg, L., and Thornell, G. (2015) Thermomechanical Properties and Performance of Ceramic Resonators for Wireless Pressure Reading at High Temperatures *Journal of Micromechanics and Microengineering*, 25(9):095016–25
- II Sturesson, P., **Khaji, Z.**, Klintberg, L., and Thornell, G., Ceramic Pressure Sensor for High Temperatures – Investigation of the Effect of Metallization on Read Range *The IEEE Sensors Journal*, Submitted
- III Persson, A., **Khaji, Z.**, and Klintberg, L., Dynamic Behavior and Conditioning Time of a Zirconia Flow Sensor for High-Temperature Applications *Journal of Sensors and Actuators A: Physical*, Submitted, reviewed, and revised
- IV **Khaji, Z.**, Sturesson, P., Hjort, K., Klintberg, L., and Thornell, G. (2015) Manufacturing and Characterization of a Ceramic Microcombustor with Integrated Oxygen Storage and Release Element *Journal of Micromechanics and Microengineering*, 25(10):104006–12
- V **Khaji, Z.**, Klintberg, L., and Thornell, G. (2016) Manufacturing and Characterization of a Ceramic Single-Use Microvalve *Journal of Micromechanics and Microengineering*, 26(9):095002–11
- VI **Khaji, Z.**, Klintberg, L., Barbade, D., Palmer, K., and Thornell, G., Endurance and Failure of an Alumina-Based Monopropellant Microthruster with Integrated Heater, Catalytic Bed and Temperature Sensor *Journal of Micromechanics and Microengineering*, Submitted
- VII **Khaji, Z.**, Klintberg, L., Palmer, K., and Thornell, G., Catalytic Effect of Platinum and Silver in a Hydrogen Peroxide Monopropellant Ceramic Microthruster *Journal of Sensors and Actuators A: Physical*, Submitted

Reprints were made with permission from the respective publishers.

Author's contribution to the publications

- I Minor part of concept, major part of planning, experimentals and evaluation.
- II Major part of concept, planning, experimentals, and evaluation.
- III Major part of planning, experimentals, minor part of evaluation.
- IV Major part of concept and planning, most of experimentals and evaluation.
- V Major part of concept, all experimentals, and most of planning and evaluation.
- VI Minor part of concept, major part of planning and most of experimentals and evaluation.
- VII Minor part of concept, most of experimentals, and major part of planning and evaluation.

Contents

1. Introduction.....	9
2. Microsystems technology	13
3. Microsystems for high temperature and aggressive environments	15
3.1 Materials and manufacturing	16
3.1.1 Co-fired ceramics.....	16
3.1.2 <i>In-situ</i> electroplating.....	20
3.2 Principles.....	21
3.2.1 Sensing.....	21
3.2.2 Actuation	22
3.2.3 Sample preparation and analysis.....	22
3.2.4 Wireless communication	22
3.2.5 More than just an expensive conductor	24
3.2 Studied in this thesis.....	26
5. Conclusions and Outlook.....	34
6. Svensk sammanfattning	36
7. Acknowledgement	40
References.....	42

Abbreviations

CTE	Coefficient of Thermal Expansion
HTCC	High-Temperature Co-fired Ceramics
LTCC	Low-Temperature Co-fired Ceramics
MEMS	Microelectromechanical Systems
MST	Microsystems Technology
OGS	Optogalvanic Spectroscopy
PCB	Printed Circuit Board
RFID	Radio Frequency Identification
YSZ	Yttria Stabilized Zirconia

1. Introduction

Human beings have faced challenges all over history, challenges that were imposed to them, and challenges that they willingly chose to face mainly due to curiosity. Natural disasters are among the most well-known challenges that humans have had to face. Understanding the nature of these, and predicting and mitigating their consequences, are one of the long-term ambitions of humans. Thanks to developments in science, nowadays humans do not relate natural disasters to “Mother Nature’s fury” or any other metaphysical phenomenon. Even though there is still a lot to learn about the nature of natural disasters, humans have already gained a deep understanding of these phenomena. However, predicting, monitoring and tackling the disasters are still difficult. It is generally accepted among scientists and decision makers that there is no such thing as a natural disaster. All that exist are natural hazards which can end in disasters as a result of mismanagement and lack of control. Humans’ progress in monitoring and tackling the hazards will save lives and properties, and, consequently, prevent disasters. The total number of people killed by natural disasters has decreased significantly over the past century due to such progress. However, there are still thousands of people losing their lives each year, figure 1.

Making high-precision, sensitive monitoring and early warning systems will be a great contribution to natural hazard management. It is also crucial to make such systems inexpensive to promote coverage in vast and remote areas, and to make them affordable also for countries with poor economies.

Apart from inevitable challenges, humans also seek challenges because they are eager to know and learn. If humans had not been curious, if they had not embraced challenges, our ancestors would probably have been satisfied with staying in Africa. They voluntarily chose to face the challenges of travelling around the continent and across great waters to discover unknown lands. But not even exploring the entire Earth satisfied humans’ curiosity. Sky has also amazed us through our history. We have wondered what was up there, and if we could go, and even live, there. We have questioned ourselves if there is any extraterrestrial life somewhere in the universe.

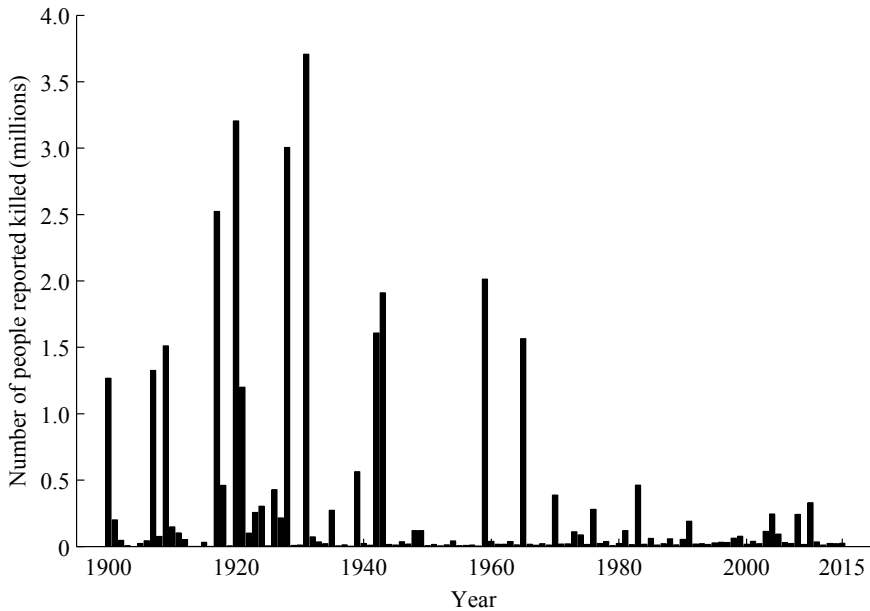


Figure 1. Total number of people reported dead in natural disasters over the past century [1].

Humanity was born on Earth. Are we going to stay here? I suspect - I hope - the answer is no. — Ann Leckie

Again, as a result of our endeavors, we have learnt enormously about the universe; we have even managed to step on moon. But the main question still remains: Is there or has there been life somewhere in space? Even if we are fortunate to find traces of extraterrestrial life one day and even if we are bright enough to understand what we found, universe is so large that what we have learnt will be negligible in comparison with what is still unknown. Manufacturing instruments and spacecraft, as well as launching them to space, has occupied mankind over decades. For space applications, in addition to high precision and sensitivity, decreasing the size and mass of hardware is important as this will result in reduced mission and launch cost.

It is obvious that both space and areas affected by natural hazards are hostile to humans. But these environments are also harsh to the equipment needed for exploration. This includes different types of sensors and actuators, sample preparation and analysis systems, and propulsion systems. Extremely high and low temperatures, high pressures, corrosion, and radiation are examples of challenging conditions that await these devices. Environments with these conditions are usually designated as harsh environments [2].

Harsh environments are not only limited to space and areas affected by natural hazards. There are also various industrial applications which impose harsh conditions to devices. Jet engines or downhole gas and oil industry, for instance, subject monitoring devices to high temperatures and pressures. Furthermore, harsh conditions are not only external. A device can also face internal harsh conditions [2]. A combustor and a propulsion system are examples of devices with extreme internal conditions, such as high temperature.

It is not just the device that should tolerate the harsh conditions. All the interfaces, valves, power drivers, energy harvesters, etc. should also be resistant to the condition.

Many harsh conditions are not clearly defined, but depend on the specific application. For instance, high temperature for commercial electronics is often limited to 80 °C, whereas in military-grade electronics, up to 125 °C is covered [3]. However, these temperatures are quite low compared with the temperatures above 1000 °C that can easily be reached in jet engines used in fighter aircrafts [4], and the lava temperature in volcanoes [5], and wildfires [6], and even the milder case of 450 °C, which is the maximum daytime surface temperature of Venus [7].

This thesis focuses on design, manufacturing and characterization of miniaturized devices and systems with high chemical resistances that are capable of working at very high temperatures (above 600 °C), based on the primary efforts of understanding the challenges facing miniaturized devices in such conditions. Two main applications are foreseen for such systems:

- Monitoring natural hazards with robust sensor systems.
- Space exploration for different purposes including searching for life outside Earth.

However, these systems have high potential for other areas of applications as well *e.g.*, in industry for monitoring processes that take place at high temperatures and in other aggressive environments.

The thesis was performed at the Division of Microsystems Technology (MST) at Uppsala University in collaboration with Swedish Centre for Natural Disaster Sciences (CNDS) and Ångström Space Technology Centre (ÅSTC).

CNDS is a national centre for research on natural disasters. It has created an interdisciplinary platform, where researchers from different scientific disciplines work together in order to provide society with knowledge and a better understanding of natural disasters. The research conducted at CNDS aims to contribute to improving the ability to monitor, prevent, and deal with risks in society by raising awareness of the dynamics and consequences of natural hazards.

ASTC is a research group within the MST division which performs research on microsystems for space applications, *e.g.*, aiming at increasing performance and decreasing the cost of space exploration and exploitation.

2. Microsystems technology

Extreme miniaturization with modern technologies, can be referred to as Microsystems Technology (MST). It can mean downscaling systems and devices already available at macroscale, which rarely is the case, or designing small-scale systems from scratch.

MST devices, as they are commonly called in Europe, also known as Microelectromechanical Systems (MEMS), as is the common name in the USA, are miniaturized and often application-specific systems comprising sensing, processing and/or actuation. These systems often combine two or more of electrical, mechanical, chemical, optical, and magnetic features into a single device [8]. MEMS features have at least two dimensions in the micrometer range, and the overall system size is often limited to a couple of centimeters.

Like any other concepts, miniaturization has its advantages and disadvantages. Decreased power consumption achieved by reducing the sizes of devices is always beneficial, but becomes of crucial importance in remotely deployed systems, where power is limited. In addition to that, small-size devices often have shorter response time and reach equilibrium faster than macroscale ones. Another advantage of miniaturization is the possibility to increase the sensitivity of the devices. Additionally, high level of integration between the different components of a device or system is possible using MST, *i.e.*, many parts of a system such as channels, valves, and communication units can be integrated into a single chip. This decreases the dead volumes and mitigates the challenges of interfacing between the internal parts of the system. Furthermore, batch production, which means performing the manufacturing processes for many devices at the same time, is often possible in MST. This decreases the manufacturing costs. Finally, redundancy is usually facilitated by MST.

MEMS devices are, however, more vulnerable to noise, as, for a small device, the working signals can be of the same order of magnitude as the noise from the environment. Another feature of MEMS devices is increased heat losses as downscaling increases the surface-to-volume ratio. This is a drawback for any MEMS device which includes a heating process, for instance a thruster or combustor. One challenge with MEMS devices, is to achieve high reliability since micro devices are usually less mature than macro devices, and therefore deserves more research.

Both space applications and natural hazard monitoring will benefit from reduction of power consumption, which is often intrinsic to miniaturized systems. Monitoring natural hazards often requires field deployment, which means that many of the devices are battery powered. The same applies to solar cell powered space devices. The high sensitivity and performance achievable by MEMS are beneficial for both the applications. Reducing mass is of more importance for space application as it significantly decreases the launch cost, which is usually a substantial part of the mission cost. On the other hand, monitoring natural hazards perhaps benefits even more from cost reductions as it is directly translated into these systems being affordable by more people. Additionally, the redundancy facilitated by MST is of substantial importance for both applications.

Developments in MST, have contributed to a concept called Lab on a Chip (LOC), which several disciplines, including space sciences and analytical systems that can be used in the field, significantly benefit from. Lab on a chips are not distinctly defined, and the term includes a wide range of devices. These are aiming at downscaling human-scale and macroscale sample analysis laboratories in to a single chip with the dimensions of a few millimeters to a few centimeters [9]. These can include many channels, chambers, valves, sensors and actuators. As they are aiming at high level of integration, the dead volumes are decreased, resulting in less sample waste [10]. Also responses are mainly faster because of shorter transportation paths.

3. Microsystems for high temperature and aggressive environments

Making devices for temperatures above 600 °C faces two main challenges, the first being finding the suitable materials and the second being making robust interfaces. When it comes to the material choice, high-temperature tolerance, *e.g.*, a high melting point, is important, but it is not the only requirement. Also, the material performance should not degrade with increase of temperature.

The challenge of interfacing stems from three sources: 1) transfer of power and signal to and from the device at high-temperature environment, 2) choice of proper materials for the interfaces such as tubings and contacts with the same criteria as the choice of material for the device, and 3) thermomechanical effects. Thermomechanical effects, *e.g.* mismatch in thermal expansions of different materials, and the resulting residual stresses, figure 2, can influence the operation of the device.

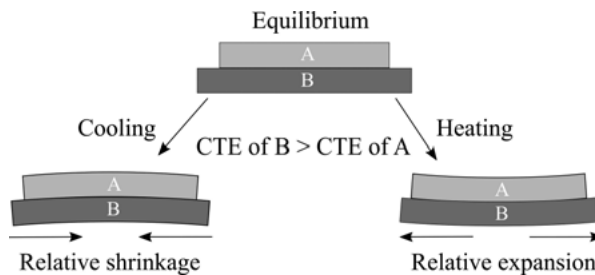


Figure 2. Influence of mismatch between the coefficients of thermal expansion (CTE) at the interface of two materials, here causing deformation.

Choosing chemically inert materials is important for devices to be subjected to chemically aggressive environments.

It should be noted that especially in case of high-temperature devices, performing the experiments and characterizations may require customized set-ups, and in some cases building the set-up is a study of its own since same device challenges also apply to the measurement set-up.

The limitation in material choice, as well as the need for developing experiment set-up, becomes more highlighted for each additional harsh condi-

tion being combined with high temperatures, *e.g.*, set-ups for high temperature and high pressure measurements, or high temperature measurement set-ups that have to cope also with corrosive chemicals.

In the following sections, an overview of the manufacturing processes as well as the principles used in this thesis, and how they contributed to overcoming the already mentioned challenges of making devices for harsh applications will be given.

3.1 Materials and manufacturing

3.1.1 Co-fired ceramics

Silicon, being a semiconductor material, is the revolutionary material of our century. Without silicon, the significant developments in electronics, featured in for instance, mobile phones and computers, would not have been possible. Silicon is also the most established material in MST. Although it is a very suitable choice for lower-temperature applications and relatively friendly environments, it is not a proper alternative for high temperature as it may deform slowly at temperatures above 600 °C if loaded mechanically [11]. Furthermore, it will lose its semiconducting properties at temperatures above 250 °C [12]. Another drawback of silicon for high-temperature applications which require heating, *e.g.*, thrusters and combustors, is its high heat conductance [13] that can result in increased heat losses.

Silicon has a quite high chemical resistance. However, it can still be corroded by some chemicals such as hydrazine [14] which is a conventional propellant for spacecraft.

Therefore silicon should be substituted with a more suitable material. Having high temperature and chemical resistances, ceramics are a promising alternative.

Ceramics and their processing in form of pottery have been known to humans for a long time. The earliest known human-made ceramic objects, one example being shown in figure 3, are at least 25,000 years old [15]. Humans had learnt that by mixing the abundant material clay with water, they could shape it into the desired form, which became rigid after drying and firing. They knew by experience that mechanical processing of the fired ceramics was difficult.

Being similar in fundamentals to pottery, but a more modern way of processing, which allows integration of metal patterns as conductors, is the co-fired ceramic technology. In this technology, ceramic powders are mixed with polymer binders and solvents and are cast into thin tapes. These tapes are soft and flexible, and can therefore be easily shaped and patterned.

In order to create 3-D structures, tapes processed individually are joined together. Structures like cavities and channels are created by techniques such as milling, punching and hot embossing. The metal patterns are often created by screen printing, a simple technique, which comprises of transferring a metal ink or paste through openings made in a screen. Making arbitrary Pt patterns in HTCC is rather straightforward and just requires designing a screen containing the desired pattern.



Figure 3. Venus of Dolní Věstonice figurine dating from 29,000 to 25,000 BC. Courtesy of Petr Novák, Wikipedia.

These individually processed tapes are then aligned, stacked and laminated to each other at high pressures before being sintered, figure 4. As can be seen in this figure, the alignment and stacking can be done by using plates with alignment pins integrated in them. Corresponding holes are made in all sheets of the ceramic tape to align them.

The lamination is often done in a hot press, in which the stack is subjected to both elevated temperature (70 °C) and isostatic pressures (200 bar). Lamination can be done in one or several steps. When working with alignment plates, lamination is done in two steps, with the first step being performed at much lower pressures than the final one, just for tapes to stick to each other. For the second lamination step, the alignment plates are substituted with

metal sheets without pins, and full nominal pressure is applied. This is done to avoid the stresses that pins can cause on the tapes at high pressures.

By sintering, the solvents and binders burn away and the ceramic grains are grown together, to form a dense material [15].

However, during lamination and or sintering, 3-D structures, especially those with structures suspended on top of cavities can deform and sag. In order to avoid this problem, a graphite sacrificial material can be inserted into the cavities to provide mechanical support [16]. The name sacrificial comes from the fact that this material will be burned away during the sintering. The sacrificial graphite can come into two forms: tapes and pastes. The tapes can be shaped into desired shapes by processes such as milling. Larger thicknesses can be achieved by stacking and laminating them. Graphite pastes however are screen-printable. They can be used to form narrower channels in ceramic structures. In addition to that, sacrificial paste can be mixed with conductive pastes with different proportions. On burning the graphite away, pores will be created in the metal patterns.

The High-Temperature Co-fired Ceramics can be distinguished from Low-Temperature Co-Fired Ceramics by its lack of glass content [15]. This increases both the sintering and working temperatures of HTCC to temperatures above 1000 °C [17].

Aluminum oxide (alumina) and zirconium oxide (zirconia) are the most common HTCC ceramics whereas the metal choices are limited to platinum group metals and tungsten, as their melting point needs to be above the HTCC sintering temperature of 1600 °C, and their thermal properties should match with the ones of ceramics.

The use of HTCC in the electronics industry goes back to the 60's when it was mainly used as a packaging technology [18]. In figure 5, one can see an electronics package made in co-fired ceramics. HTCC has recently gained interest also in MST due to its suitability for high temperatures and chemically harsh environments. Being a new MST manufacturing technique, HTCC subjects the researchers using it to challenges that may be trivial for conventional MEMS manufacturing processes and industry. There are cases when one has to adapt to using available resources and therefore a researcher may have to perform extra underlying research and development to make the resources compatible with this new technique. For instances, making gas tight thin membranes which is a well-developed process in silicon processing, deserves its own study if it should be done in HTCC, especially with metal patterns on it.

Overview of HTCC processing

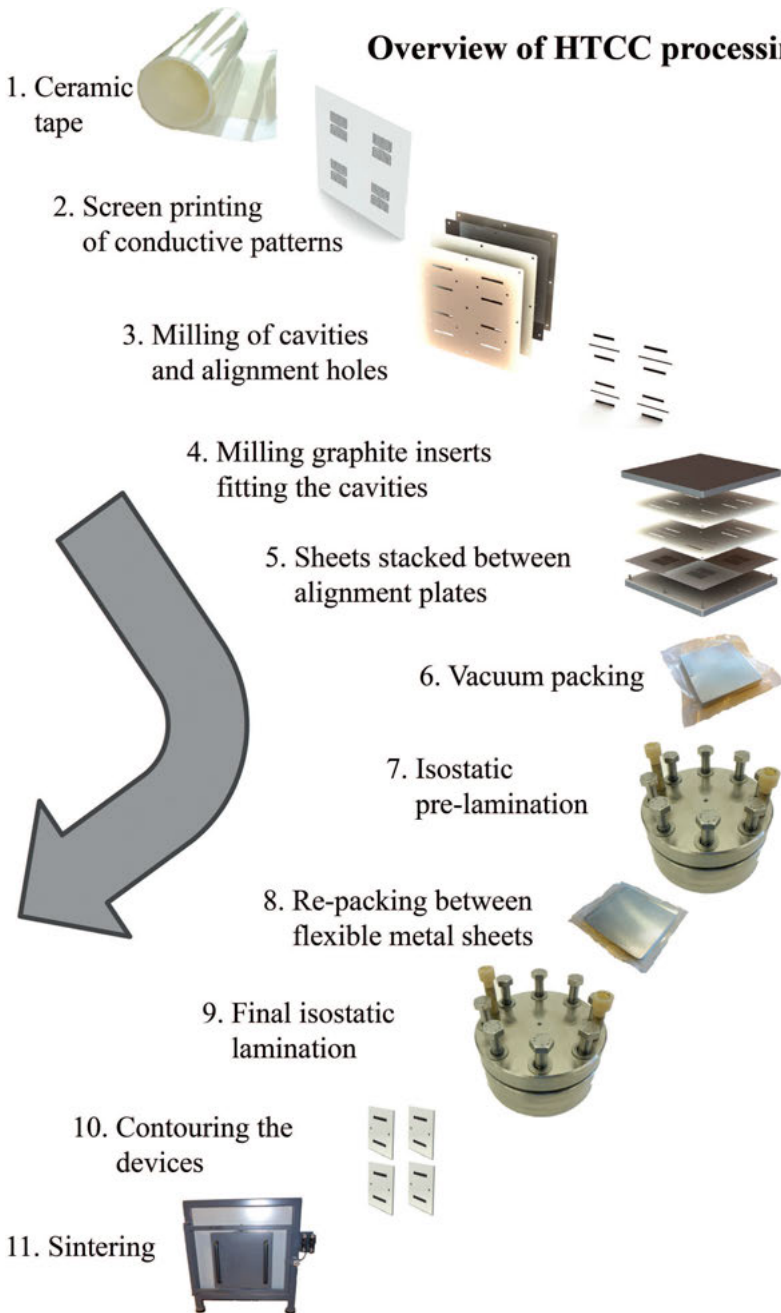


Figure 4. Schematic of co-fired technology manufacturing process, showing how flexible green ceramics that come in form of tape rolls (1) are milled and screen printed individually and then stacked and stepwise laminated together. (7) and (9) show the pressure chamber in which the stack is isostatically pressed by pumping water into the chamber.

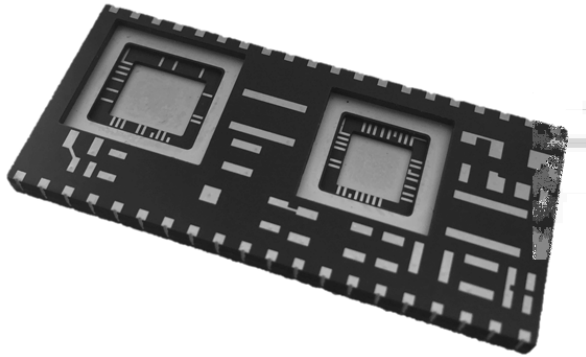


Figure 5. A package for electronic devices made of co-fired ceramics.

A disadvantage of ceramic material in general, including HTCC, especially alumina HTCC, is their weakness in handling stresses caused by thermal gradients and transients, *i.e.*, in particular tensile stresses are undesirable [19]. This makes the ceramics prone to cracks and demands a proper design of the devices to minimize these effects.

3.1.2 *In-situ* electroplating

As already mentioned, the high sintering temperature of HTCC limits the metal choices. To be able to combine more metals with HTCC, a post-sintering *in-situ* electroplating technique after sintering was devised and exploited in this thesis. In electroplating, a conductive coating is deposited on a surface by passing electric current through a conductive ion-bearing solution [20]. Electroplating is widely used in Printed Circuit Board (PCB) technology [20]. However, using it in MST for electroplating inside cavities and enclosed structures is not common. Using this technique, many metals such as nickel, copper, silver, and gold can be added to HTCC devices after sintering. An example of an *in-situ* electroplated surface is shown in figure 6.

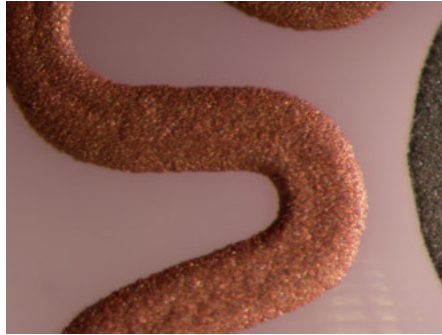


Figure 6. Copper electroplated on a platinum meander pattern, Paper IV. To the right a glimpse of a non-electroplated platinum pattern can be seen.

The roughness of electroplated surfaces can be affected by parameters such as agitation and temperature. Although less rough surfaces are often desired, there are applications where rougher surfaces are beneficial, e.g., in order to increase the surface area, as shown in Papers VI and VII.

3.2 Principles

3.2.1 Sensing

Should one want to monitor the performance of any device, system or even a natural phenomenon, using some sort of sensor is almost inevitable. There are numerous physical and chemical parameters that can be measured. Working with high-temperature applications, the most obvious parameter to be measured is perhaps temperature. Measuring pressure and flow rate are also highly demanded in fluidic systems, including the ones working at high temperatures. Sometimes sensors are used for external measurements, as presented in **papers I - III** in this thesis, and sometimes they are integrated in a system to monitor the performance of the internal sub-systems, presented in **papers IV, VI, and VII**.

As mentioned in the beginning of section 3, one of the challenges of making microsystems for high temperatures, is integration and interfacing of components made of different materials with different thermal behaviors. Therefore it is wise to decrease the number of different materials to be integrated. One way of doing this is by using the bare minimum of materials and using them to their maximum extent. For instance, the temperature dependence of a conductor path's resistivity can be used as a thermal sensor, **Papers IV, VI, and VII**, or the ion conductivity of a structured ceramic can be used to make flow sensor, **Paper III**.

3.2.2 Actuation

Sensing and actuation are complementary concepts with sensors being responsible for sensing or measuring, and actuators being responsible for performing actions, or movement. In sensor-based control systems, some actuations may be done based on the sensed parameters. For instance, one may want to activate a pump or open or close a valve based on a change in a pressure or temperature of some parts of the system. An example of such a valve is shown in **paper V**. Actuators can also be standalone devices, *e.g.*, propulsion systems that produce thrust for spacecraft, such as the one studied in **papers VI and VII**.

3.2.3 Sample preparation and analysis

Sometimes the concept of sensing becomes so complicated that advanced sample analysis systems are needed instead of single sensors. For instance, analyzing the content, origin, or age of a sample may require advanced isotopic studies. A wide range of applications in biology, archeology, forensic sciences, geology, and space exploration use sample analysis systems [21]–[23]. In, for instance, volcanology, isotopic measurements have great potential in studies related to seismic or magmatic activity [24].

Many sample analysis systems demand the sample to be in a specific phase, *e.g.*, gas form, or be pre-processed prior to the measurement, *e.g.*, being combusted. In carbon isotope ratio analysis of $^{13}\text{C} / ^{12}\text{C}$, for example, the sample often has to be combusted and converted to carbon dioxide [25]. Therefore, sample preparation systems, such as the one presented in **paper IV**, are as important as the sample analysis systems.

Decreasing sample consumption is highly desirable. This can be achieved by integrating the sample preparation system with the analysis system, which will decrease the dead volumes, and consequently the sample lost. Furthermore, integrating sensors, actuators, and valves in an analysis system may be desired. Hence, developing analysis systems in form of labs on a chip with all the benefits described in the end of section 2, is quite beneficial.

3.2.4 Wireless communication

Sensors made by high-temperature tolerant materials must still be powered and read. Working with plenty of wires in order to transfer power and signal can be troublesome, even in non-harsh environments. When it comes to harsh environments, wiring can become impossible. The signal transmission in wires may be substituted by wireless communication. However, in addition to be high-temperature tolerant, the wireless system should also be very simple by including few components and contacts to avoid problems of interfacing.

Powering is probably an even bigger issue. Although batteries operating at 550 °C for 48 hours have been reported [26], batteries have not yet been developed to sustain temperatures above 600 °C.

Harvesting energy from energetic phenomena in the surrounding of a sensor, such as vibrations or thermal gradients, can be an alternative for powering [27]. This is, however, challenged by low efficiency of the energy harvesters. Furthermore, if the conditions for harvesting are variable, they will inhibit the ability to power a device at all times with a stable and continuous power. An alternative can be wireless power transfer, which, generally speaking, can be done both by near and far magnetic fields. In the near-field domain, inductive coupling of magnetic fields between a sensor and a powering circuit will activate and read a sensor; *i.e.*, the sensor receives its required power from the transmitted radio frequency signal. This is commonly used in Radio Frequency Identification (RFID) applications [28]. Inductive coupling is studied as a powering and reading solution for electrical devices such as electrically driven automobiles [29], [30], and body-implanted devices [31]. Although being generally limited in read range, inductive coupling has the advantage of being easily implemented by a resonator consisting of practically only two components: a capacitor (C) and an inductor (L). In fact, a capacitor is just a dielectric material integrated between two metal plates, and an inductor is simply a coil. As illustrated in figure 7, both can be highly integrated and easily manufactured with HTCC. Using inductive coupling, the LC resonator can be used to transfer both signal and power from and to the harsh environment by having an LC resonator in the harsh environment as the transponder, and another one outside as the reader and source of power, figure 8. This was implemented in **papers I and II**.

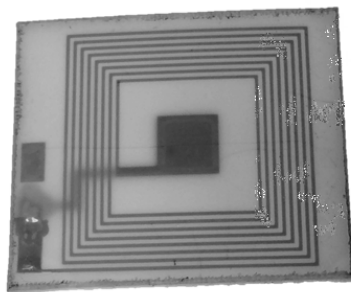


Figure 7. A device containing a series LC resonator implemented in HTCC, papers I and II. The loop is an inductor. The square at the center is one of the capacitor plates.

Not being deployed in the harsh environment, the reader resonator does not have the design limitations of the transponder and can even contain conventional electronic components.

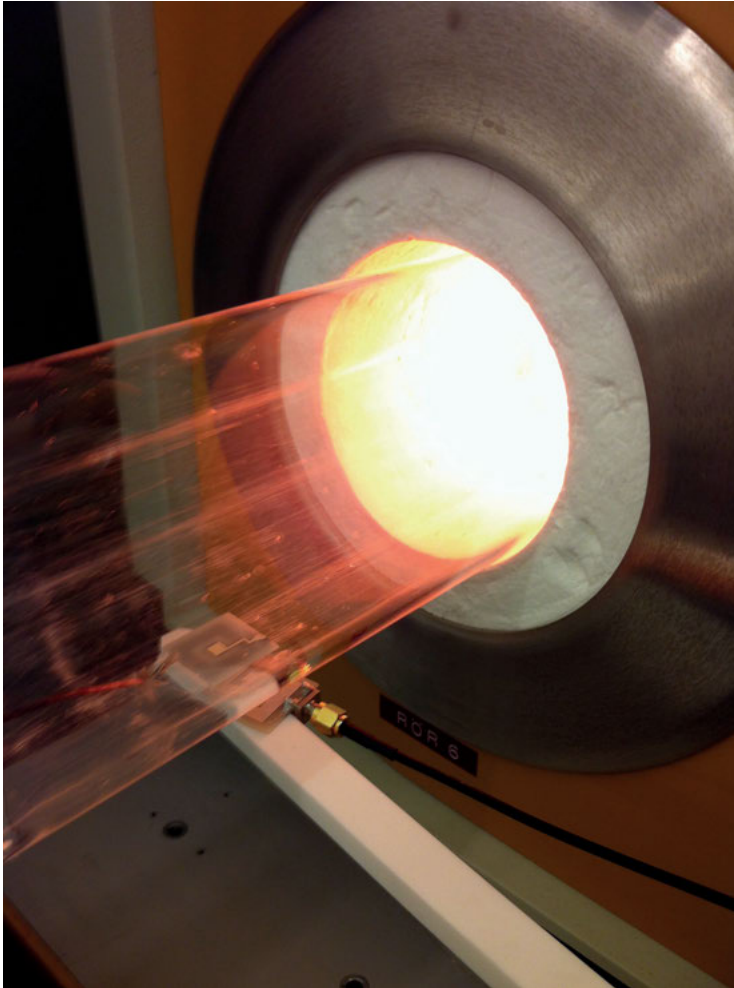


Figure 8. A device, integrated with an LC resonator, inside a high-temperature oven being read and powered with another LC resonator at room temperature. The reader resonator is located under the glass tube just below the device inside.

3.2.5 More than just an expensive conductor

One solution for mitigating the challenges of integrating components at high temperatures, is decreasing the number of components with different materials to be interfaced with each other by, *e.g.*, exploiting their material properties as much as possible. Due to its high melting point as well as having compatible thermal properties, platinum is one of the few metal alternatives compatible with HTCC. Despite having lower electrical resistance, in comparison with conventional conductors, such as copper or even silver, platinum has several properties that can be exploited.

In fact platinum is a very good temperature sensor as its resistance is linearly and highly dependent on temperature [32]. The small loop in the middle of figure 9 is an example of a temperature sensor made in platinum, a concept which as used in **papers III, VI, VI, and VII**.

Also, the fact that its resistance is relatively high, can also work in favor by being capable of generating heat when high electrical currents are passed through, **papers III - VII**. The meander in figure 9 is a platinum heater. In order to produce more heat, the resistance is further increased by the meander shape, which increased the heater's length. These two components are not only made of the same material, but in fact in the very same process.

Platinum is one of the most famous catalysts for many reactions. In addition to that, it is a chemically inert material which widens its application as a catalyst to cases where aggressive chemicals are used.

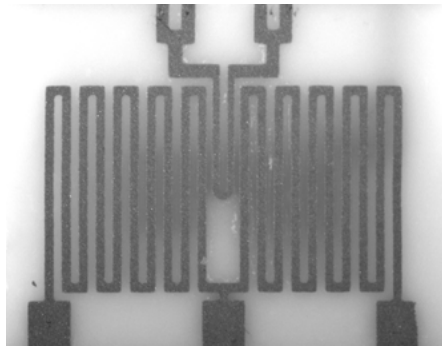


Figure 9. A Pt temperature sensor (the loop in the middle) and a Pt heater (the meander) made of alumina HTCC.

Being a conductive material, platinum can be used to make electromagnetic components. For example, it can be used to make capacitor plates and coils or inductors and therefore can be used to make LC resonators as shown in figure 7. However its high resistance can result in a low quality factor of the resonators. However, taking advantage of electroplating technique described in section 3.1.2, one can decrease the resistance by electroplating metals with higher conductance, such as silver, on top of the platinum, **paper II**.

In the same manner, many other metals with properties different from those of platinum can be added to HTCC components after sintering. An example of this is adding metals such as copper and nickel to HTCC components, such as the copper electroplated platinum surface shown in figure 6. The oxides of these metals reduce at very high temperatures [33] and therefore can be used as oxygen storage elements in microsystems, **paper IV**. It should be noted that bi-metallization may also subject the interface between the metals to already mentioned thermomechanical effects. Therefore, the

performance of these bi-metals at high temperatures requires a study of its own, **papers II and IV**.

3.2 Studied in this thesis

Papers I - III focus on high-temperature ceramic sensors. In **paper I**, a wireless pressure sensor made of alumina HTCC which uses an LC resonator for wireless transfer of signal and power, is evaluated. The actual sensing part is a thin membrane on top of a cavity. When pressure is applied, the membrane deflects, resulting in decreasing the height of the cavity. The change of the height will change the capacitance of the capacitor, and consequently, the frequency of the LC resonator. This can be detected by the reader resonator. A cross-section of the LC transponder, with the sensing element integrated with it, is shown in figure 10.

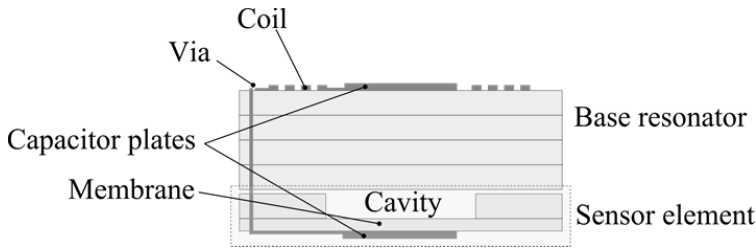


Figure 10. Cross-section of a wireless pressure sensor node including an LC resonator and a sensing element.

The LC resonator presented in **paper I** can be used for wireless transfer of power and signal for other types of sensors as well. Hence improving its performance, including its read range as well as simplifying its manufacturing process is of great value. Therefore these were studied in **Paper II**, and a new manufacturing scheme for making the cavity without mechanical machining was demonstrated. Furthermore, silver electroplated and double screen printed LC resonators were compared with single platinum layer resonator, with respect to their DC resistances, quality factors and read ranges. In addition, a thorough study of interdiffusion between Pt and Ag with change of temperature and time was performed.

The wireless pressure sensors presented in these papers were successfully operated at pressures up to 2.5 bar and at temperatures up to 1000 °C. Although both the metallization methods used in **paper II**, improved the read range in comparison with **paper I**, interdiffusions of silver and platinum with increase of elapse time and temperature, were observed which have impact on the resistance and read range of the resonators. Furthermore, at high-temperatures, the mismatch between the CTEs of the alloy of silver and

platinum, and ceramics can result in loss of adhesion between the metals and ceramic, figure 11.

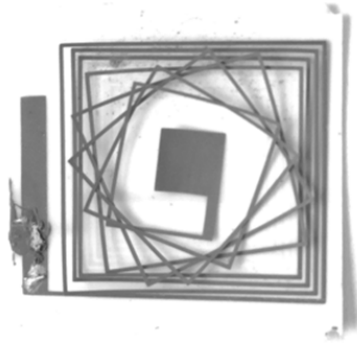


Figure 11. An LC resonator with silver-platinum metallization after 72 hours at 900 °C. The top capacitor plates and a large part of the coil have detached from the surface substrate.

Also focusing on sensors for high-temperature applications, a calorimetric flow sensor was previously developed in yttria stabilized (YSZ) zirconia HTCC within the group [34]. Calorimetric flow sensors are a common type of thermal flow sensors that involve temperature sensors located up- and downstream of a heating element placed in a flow channel. These sensors measure the asymmetry of the temperature profile caused by the flow in a channel [35]. The calorimetric sensor in [34], used the temperature dependent ion conductivity of YSZ for measuring the temperature profile.

The performance of that sensor was limited by thermal cross-talk with increase of temperature. **Paper III** investigates the feasibility of integration of thermal isolation in the form of sealed cavities with different geometries to mitigate the effect of this. A fabricated flow sensor with the fixture used to interface with it is illustrated in figure 12 (left). A schematic cross-section of the sensor after integration of the thermal isolation cavities is shown in figure 12 (right).

The results showed that by using insulation cavities, the conditioning time of the sensors, *e.g.*, the time needed to reach equilibrium, was improved by up to 5 times and the power consumption was slightly decreased. However, the performance seemed to be highly dependent on positioning and alignment of the cavities.

Both the above-mentioned pressure and flow sensors can be used in a wide range of applications as stand-alone sensors or as sensors integrated in a big system, including lab on a chip

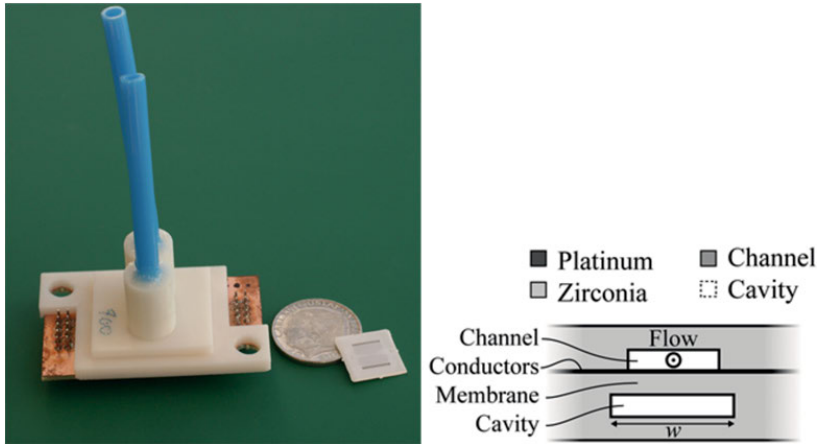


Figure 12. A calorimetric flow sensor with a Swedish 1 krona coin as a reference together with its mounting fixture (left), and a schematic of its cross section, perpendicular to the flow direction, after integrating isolation cavities (right).

In paper IV, the design and characterization of an alumina microcombustor are presented. Having an integrated heater, temperature sensor and oxygen storage source, this combustor contains all the functionality necessary. Copper *in-situ* electroplated on the heater pattern and thermally oxidized later, was used as the storage source of oxygen. An X-ray picture of the combustor is shown in figure 13 (left).

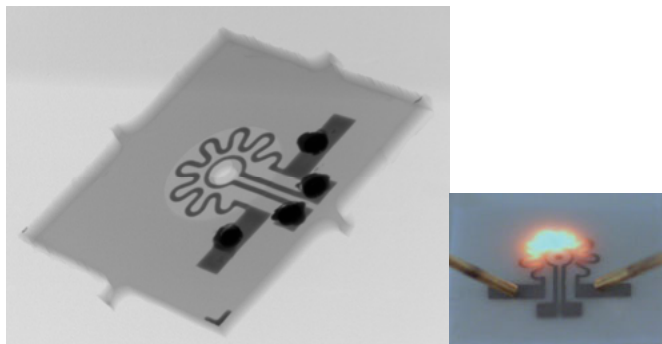


Figure 13. X-ray image of a microcombustor (left). The meander pattern in the middle is the heater with the loop at the center being the temperature sensor, both made of platinum. The brighter color circle in the middle of the image is the combustion chamber in which the combustion takes place. (Right) is a combustor glowing when being heated to 1000 °C.

Having the copper oxide layer on top of the heater, the heater can be used both for heating the sample to be combusted and the copper oxide to decompose and release oxygen. Very small amounts of solid samples can be handled in this combustor, which is of great value for all the applications where very small amount of samples are available such as in archeology and foren-

sic sciences. Furthermore, the small size of the combustor makes it proper for integration into greater systems.

This combustor was successfully operated at temperatures up to 1000 °C, figure 13 (right). To date, this is the smallest known combustor ($8 \times 8 \times 0.3 \text{ mm}^3$) of its kind, *i.e.*, with all these integrated features and such a high temperature tolerance. The feasibility of using copper oxide as the source of oxygen was demonstrated. However, the stresses from oxidation and reduction caused delaminations of the copper film, which may make the combustor more suitable for single-use.

In **paper V**, different manufacturing schemes for, and characterization of, a ceramic single-use isolation valve are shown. In conformity with the already mentioned ambition of exploiting the material properties for sensing and actuation, as much as possible, this valve consists of a thin ceramic membrane on top of a cavity with a platinum resistive heater printed on it. The actuation mechanism is based on the weakness of alumina in handling thermal shocks. Therefore the valve is opened by fast resistive heating of the membrane which causes it to crack. Picture frames of the opening process of a valve, taken by a high-speed camera, can be seen in figure 14.

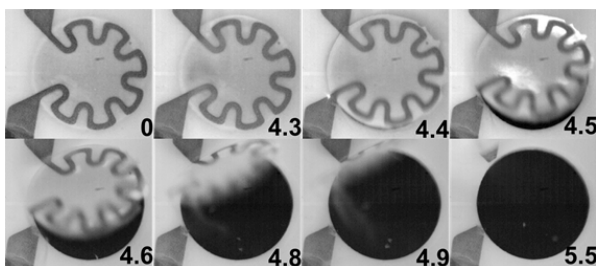


Figure 14. Frames recorded by a high-speed camera during opening of a ceramic valve with the diameter of 3 mm. Elapsed time in ms is displayed in each frame.

Opening sizes varying from microcracks to the whole valve being opened was observed depending on the valve design. The low energies (few hundred mJ) consumed for opening the valves, make them suitable for application where limited energy is accessible. But before being integrated in bigger systems, the influence of pressure differences on the performance of the valves and the behavior of the fragments after breaking should be well studied. In addition the possibility of integrating filters for collecting particles and fragments should be investigated in the future.

For the devices presented in **papers IV and V** in addition to general applications, a specific application was also foreseen.

As a part of an ongoing project at ÅSTC, a combined sample preparation and analysis system in form of a lab on a chip is being developed [36]. The targeted application for this device is finding traces of extraterrestrial life on Mars. Conventional rovers, such as NASA's Curiosity, have mobility limita-

tions by being capable of only coping with flat and smooth terrain, not slopes, mountains and canyons, which, as a matter of fact, would be very interesting to study [37]. Furthermore, conventional large rovers are very slow. Therefore, highly mobile and faster rovers are needed. This means that the sample analysis and preparation systems should also be miniaturized.

ÅSTC's lab on a chip uses Optogalvanic Spectroscopy (OGS), which is a very sensitive technique for distinguishing between different isotopes. OGS measures the change of impedance in a plasma when it is irradiated with a certain wavelength of laser light. Particular advantages with OGS, is that the output is electrical from the beginning, which makes it easy to handle the signal. It also makes the instrument compact as there is no need for extra components to convert the signal. Furthermore, the detection principle doesn't consume the sample's signal output which makes it very sensitive [23]. Employing this technique, one can use the ratio between different isotopes of carbon as an indication of the organic origin of a sample, since samples originating from photosynthesis contain much less ^{13}C than ^{12}C . Small amounts ($< \text{ng}$) of solid samples can be analyzed using this device [38]. The plasma here is created by ionizing carbon dioxide from the sample, which means that solid samples must be converted to carbon dioxide before being analyzed, and therefore a combustor is a vital subsystem of this device. The combustor presented in **paper IV** can be used for this purpose.

Valves are important building blocks of most of the lab on a chip devices, and ÅSTC's lab on a chip is not an exception to this general rule. The isolation valve characterized in **paper V**, can be used as a breakable sealing between different parts of the device, and opened when needed, *e.g.*, to introduce the gases produced after the combustion to the plasma source. The first developed lab on a chip using the devices presented in **papers IV and V** is shown in figure 15 with the isolation valve being on the left (the meander shape) and the plasma source on the right side (the circle) of the image.

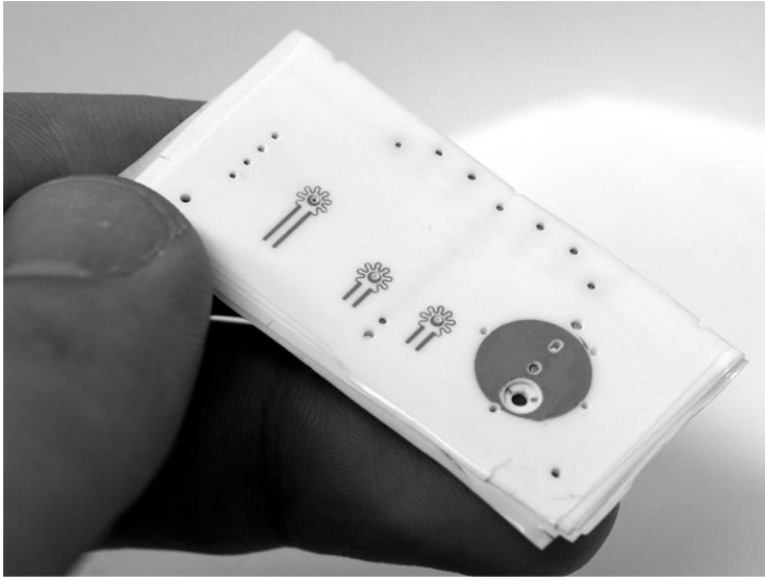


Figure 15. A sample preparation and analysis lab on a chip developed at ÅSTC for isotopic measurements of carbon. Note that this device contains embedded parts, such as the combustor (paper IV), which cannot be seen in this picture. The isolation valve (paper V) and the ground plane of the plasma, are visible in the left and right most part of the device, respectively. The two meander shapes in the middle of the image, which look quite similar to the isolation valve, are in fact single-use sealing valves that are not presented in this thesis.

It is worth mentioning that isotopic studies are not limited to space applications. They are widely used in other fields of natural sciences, including geology and volcanology [39]. Furthermore, the application of the micro-combustor is not just limited to producing carbon dioxide. It can also be used to burn a fuel and produce energy. This can be exploited by, *e.g.*, remotely deployed sensors, as a source of energy.

Papers VI and VII are strictly space oriented, and contain the results of studies on ceramic monopropellant microthrusters, which can be used for precise attitude control and positioning of small spacecraft. In order to reduce the mass and size of spacecraft, and consequently reducing the launch and mission costs, making spacecraft with a mass of less than 100 kg, has recently attracted a lot of attention [40], and has been a focus of research, with some of them already having been tested in space [41]–[44].

In addition to space exploration, precise attitude control of small satellites is beneficial for metrological and geological remote sensing [45]. This can contribute to monitoring and controlling of natural hazards as well climate changes. Remote sensing may also contribute to increasing our knowledge of natural sciences.

Monopropellant thrusters, in which a propellant (often a liquid) is decomposed in presence of a catalyst, are an attractive and seemingly simple type of thrusters.

The microthrusters investigated in this thesis, include a heater for heating the propellant and two temperature sensors for monitoring this, figure 16.

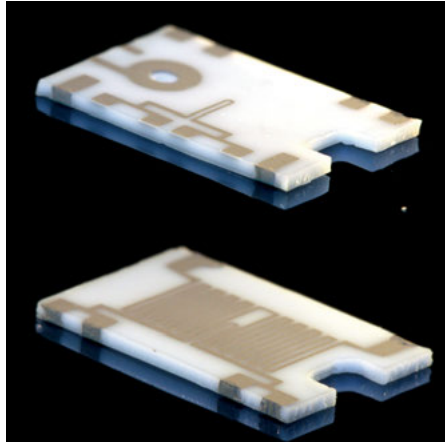


Figure 16. A microthruster made in alumina HTCC with its top side containing a temperature sensor (the loop in the middle), a propellant inlet, and connection pads (top), and its bottom side containing a heater with a temperature sensor close to its center (bottom).

In **paper VI**, the influence of variations in design on thermal behavior of the microthruster was investigated, whereas, in **paper VII**, the catalytic effect of platinum and silver on hydrogen peroxide as the monopropellant was studied.

High-concentration hydrogen peroxide is a highly reactive chemical that has an old history in space industry as a monopropellant. Hydrogen peroxide has recently regained attraction since it is considered a green propellant, the products of its decomposition being just water and oxygen. Toxic propellants, such as the conventional hydrazine, may be banned in the near future as they are considered as dangerous for the environment [46]. Furthermore handling them is very costly.

Here the catalytic effect of platinum was exploited, and as another catalyst, in **paper VII**, silver was *in-situ* electroplated on platinum. In order to increase the surface area, platinum was mixed with graphite sacrificial paste as described in section 3.1.1.

Paper VI, proved smaller sizes of decomposition chamber to result in less temperature gradients over devices and giving higher temperature tolerance. This signifies the importance of using smart designs where thermal gradients and transients in ceramic devices are minimized, consequently increasing their temperature tolerance. Furthermore, more elaborations on

both electrical and fluidic interfaces were found necessary when working with heated and high concentrated hydrogen peroxide.

To mitigate the interface challenges, in **paper VII**, a custom designed fixture was made and used for mounting the thrusters and implementing the electrical and fluidic interfaces during measurements. This, resulted in more viable interfaces in compare with **paper VI**, however, the fixture was still limited in working temperature.

Making porous platinum surfaces by using graphite paste and electroplating silver on the porous platinum surface was successful, but the resulted silver film was less rough than the platinum surface. However, this can be steered by optimizing the electroplating parameters. It was found that platinum was qualitatively shown to be a more effective catalyst, but quantitatively very little difference could be resolved between silver and platinum. Platinum is therefore recommended to use as a catalyst in the microthruster since it implies less processing steps.

5. Conclusions and Outlook

This thesis shows the high potential of high-temperature co-fired ceramics technology in manufacturing a variety of microsystems, including: sensors, actuators, sample preparation systems, and wireless devices with a wide range of applications in industry, space exploration, and different areas of science.

It was demonstrated that the intrinsic properties of the constituent materials, both the metal and the ceramics, can be exploited to a high degree as sensing or actuating mechanisms in order to reduce the need for adding extra components or processing step. However, and if needed, introducing other metals, especially the ones incompatible with HTCC, to the devices after standard HTCC manufacturing, was proven to be possible by electroplating, most notably in the *in-situ* version. This adds further capabilities to HTCC technology.

The here manufactured HTCC devices were successfully operated at temperatures up to 1000 °C, which is well above what conventional microsystems can tolerate. In **papers VI and VII**, HTCC's suitability also for chemically harsh environments was confirmed. Hence, HTCC was proven harsh environment tolerant.

Also, the first attempt in integrating the devices presented in **papers IV and V** in a HTCC miniaturized sample analysis system was promising which shows the potential capabilities of HTCC in making rather advanced lab on a chip devices without access to expensive clean room facilities.

However, developing microsystems for high-temperature application is a long journey. Several achievements have been reported in the literature, especially when it comes to materials and processing. But there are still challenges to overcome, with the major one, both application and research wise, being interfacing and integration. Adding a new type of interface, electrical, mechanical, etc., will bring its own challenges. The problem is of course intensified when there is more than one harsh condition imposed to a component, such as for the microthruster presented here, where the device was subjected to both an aggressive chemical and high temperatures.

The wireless interface solution presented in **paper I**, which is based on magnetic coupling, has a limited read range, and is, to a high degree, susceptible to noise. Being related to the same fundamental physics, on which the device relies, these issues are difficult to solve by engineering.

Therefore, despite the read range improvements presented in **paper II**, the read range is still severely limited. Hence, even though using this type of wireless transfer of power and signal is an alternative, other principles should also be investigated.

Another example of even larger interface challenges is found in the microthruster study, where there are electrical, mechanical and fluidic interfaces to the device. The custom-designed fixture, devised in **paper VII**, was a significant improvement compared with the setup used for **paper VI**, and seemingly other studies in the literature. However, aiming at very high working temperatures, the temperature tolerance of the fixture, especially its fluidic connection, should be further improved. And beyond this, completely new interfaces are needed when the thruster should be integrated with other subsystems to form a full propulsion system.

When integrating materials, investigating their thermal compatibility individually, is not sufficient. Employing two-step metallizations, such as copper, **paper IV**, and silver, **papers II and VII**, electroplated on platinum, will lead to interdiffusion of the metals, especially at the high temperatures aimed for here. This will affect the performance and reliability of the component in many ways. Signs of stresses and reduced adhesions were seen in **papers II and IV**, and the resistivity of the second-generation pressure sensor, **paper II** changed with time and heating. This phenomenon needs deep understanding and more research.

Even though weakness in handling thermal shocks is intrinsic to ceramics, especially alumina, design improvements to minimize thermal gradients and transients are worth a study of its own.

Continuing research in the field of high-temperature microsystems, in fact independently of the manufacturing process and material choice, demands large efforts and investments in developing viable measurement set-ups.

Similar to any proof of concept, especially in microsystems technology, even after solving these problems, there will be years of optimizing the manufacturing processes, achieving high reliabilities, and finally devising economically beneficial production processes for high-temperature microsystems.

Considering the foreseen applications in space exploration and monitoring natural disasters, the product realization time is even longer due to the less fault tolerant nature of these applications.

It will definitely be decades before the first ceramic lab on a chip is tested in a Mars rover or in a volcano crater. But great achievements are worth long journeys.

6. Svensk sammanfattning

Genom historien har mänskligheten ständigt stått inför utmaningar. Ibland har utmaningarna varit i form av oundvikliga naturkatastrofer, t ex jordbävningar, översvämningar och vulkanutbrott. Naturkatastrofer har alltid både förvånat och skrämt oss och vi har genom historien arbetat med att försöka förstå varför de uppstår och hittat sätt att övervaka, förutse och förhindra dem. Tack vare forskningen kopplar vi inte längre naturkatastrofer till "Naturens raseri", utan vi har numera en hel del kunskap om deras orsaker. Det är dock fortfarande lika viktigt för oss att kunna bevaka och hantera naturkatastrofer.

Det finns också en andra grupp av utmaningar som naturen ställer oss inför som är resultat av våra egna handlingar. Klimatförändringarna är det mest uppenbara exemplet. Jorden har blivit varmare som en följd av en ökad och okontrollerad produktion av växthusgaser. Glaciärerna smälter och havsnivåerna höjs. Dessa förändringar kan också resultera i naturkatastrofer som översvämningar och jordskred. Vi kan inte fortsätta så här utan vi måste vidta åtgärder. Det är nu allmänt accepterat bland forskare och beslutsfattare att "naturkatastrofer" egentligen inte existerar. Det som finns är istället faror i naturen som kan sluta i katastrofer till följd av misskötsel och bristande kontroll. Nu är frågan: Hur kan naturen övervakas på det mest effektiva sättet och hur kan naturkatastrofer hanteras?

Ofta är det vi människor som själva söker efter utmaningar, mest för att vi är nyfikna och vill veta så mycket som möjligt om världen. Redan tidigt i historien ville vi kunna allt om jorden och vi började därför resa för att hitta andra kontinenter att utforska. Men det slutade inte där. Någon gång började vi att fundera: Är vi ensamma i hela universum? Finns det liv på andra planeter? Kan vi åka till rymden? Förmodligen finns det intressanta saker att göra för människor också utanför vår egen planet. Kanske kan vi inte leva på jorden i framtiden utan måste hitta nya platser. Vi har lärt oss en hel del om rymden och vi har till och med varit på månen. Men vad vi har lärt oss är försumbart i jämförelse med vad som fortfarande är outforskat i universum. Dessutom återstår fortfarande huvudfrågan: Finns det liv där ute eller har där åtminstone funnits liv?

För att svara på de svåraste frågorna och lösa de svåraste problemen måste vi utforska de svåraste miljöerna. Det innebär att vi bland annat måste bemästra miljöer där temperaturen är väldigt hög. Dessa miljöer är inte bara svåra att uthärda för oss människor utan även väldigt krävande för vår utrus-

ning. Bland annat kan man nämna att den maximala dagstemperaturen på vår grannplanet Venus är 450°C, och att temperaturen kan vara över 1000°C i lavan från vulkaner eller i skogsbränder. Ofta är dessa miljöer även kemiskt aggressiva.

Både när man ska utforska rymden och övervaka farligheter i naturen behövs utrustning som både är känslig och svarar snabbt. Eftersom det är lättare att hantera små saker så är det en fördel om utrustningen är lätt och liten. I många fall finns det endast tillgång till begränsad mängd energi och då är det bra om utrustningen också är effektsnål.

Genom att använda mikrosystemteknik kan många av de funktioner man efterfrågar uppfyllas. Med mikrosystemteknik menar man, ofta tillämpnings-specifika, komponenter som har åtminstone två dimensioner som bäst yttrycks i mikrometer (1/1000000 meter). Måttet på hela systemet är ofta begränsat till ett par centimeter. Mikrokomponenter finns i form av sensorer, som känner av ett fysisk eller kemisk fenomen, till exempel, temperatur, flöde eller tryck, och som aktuatorer, som är komponenter som kan utföra ett mekaniskt arbete, t ex röra på sig. Ventiler som öppnas och stängs vid behov, motorer och förbränningskammare som producerar dragkraft och effekt, är exempel på några olika typer av aktuatorer.

Det finns avancerade system som kan användas för analys av prover och för att kunna undersöka deras innehåll, ursprung eller ålder. Sådana system gynnas mycket av miniatyrisering, framförallt genom att provåtgången minskar när mängden anslutningar mellan olika komponenter minskas. Eftersom transportvägarna kortas blir systemen också snabbare. Potentiellt behöver många olika sensorer och aktuatorer integreras i ett provanalyssystem.

Genom att sikta på tillämpningar för utforskning av rymden samt för övervakning av naturkatastrofer, fokuserar denna avhandling på att skapa förståelse för och övervinna de utmaningar det innebär att vidga tillämpningen av mikrosystem till temperaturer över 600° C samt att utsätta dem för kemiskt tuffa miljöer. Detta är utmaningar som konventionella mikrosystemmaterial, t ex kisel, inte klarar. I detta arbete används istället keramer som har bättre materialegenskaper för högttemperaturlämpningar.

Eftersom sintrade keramer är hårda och svåra att forma används istället flexibla keramiska tejper som består av keramiskt pulver blandat med ett polymert bindemedel. Tejperna bearbetas individuellt och förses med metalliska ledarmönstrer innan de staplas och lamineras till varandra. I ett sista steg bränns bindemedlet bort och keramkornen sintras samman. Resultatet blir keramiska komponenter som tål över 1000°C. Man brukar benämna tekniken HTCC vilket står för "high-temperature co-fired ceramics". En mängd olika mikrosystem kan tillverkas genom att använda HTCC-tekniken, men dessa måste också anslutas elektriskt, och ibland behöver man koppla in anslutningar för att leda in gas eller vätska. För att hela systemet ska tåla de höga temperaturerna, måste också materialen och teknikerna som används

för anslutningarna väljas med stor omsorg. Trådlös överföring av effekt och signal till och från sensorer är ett alternativ.

Det är också klokt att använda sig av en smart och finurlig design som utnyttjar de egenskaper som finns hos materialen som används. Detta kan utnyttjas både för att skapa sensorik och aktivering. För högtemperaturtillämpningar är det en stor fördel att använda så få material som möjligt och att begränsa antalet övergångar mellan olika materialen. Smart design kan också betyda att man behöver göra anpassade fixturer för att ansluta till komponenterna.

I denna avhandling har flera olika mikrosystem i HTCC utvecklats, tillverkats och utvärderats. De tänkta tillämpningarna sträcker sig från att göra mätningar på jorden till system som är tänkta för utforskning av rymden. Resultaten visar att HTCC har stor potential att användas i dessa krävande miljöer.

Till exempel har en trådlös trycksensor tillverkats och utvärderats vid temperaturer upp till 1000°C. I avhandlingen ingår också ett arbete som förbättrar designen av en högtemperaturflödesgivare genom att införa strukturer som begränsar värmeledningen. Med en tänkt tillämpning i ett miniaturiserat keramiskt analysystem för att analysera isotopsammansättningar, har en keramisk engångsventil och en mikroförbränningskammare utvecklats och karaktäriserats. Båda komponenterna är av central betydelse i analysystemet och mikroförbränningskammaren kan användas för att förbränna fasta prover och omvandla dem till koldioxid, något som är nödvändigt för att kunna analysera vilka kolisotoper som ingår. Information om isotopsammansättningen kan användas för att utvärdera om ett prov har organiskt ursprung. Slutligen har mikroraketer som kan användas för att exakt kontrollera och styra positionen hos små satelliter tillverkats i HTCC.

I detta arbete har flera olika egenskaper som finns hos platina utnyttjats. Till exempel har den höga resistiviteten och det faktum att resistansen förändras sig linjärt med temperaturen utnyttjats för att tillverka integrerade värmare och temperatursensorer. Det är välkänt att platina är ett utmärkt katalysatormaterial vilket har utnyttjats i arbetena med mikroraketer. De höga temperaturer som används i sintringssteget under HTCC-processningen gör att antalet metaller som kan integreras är ytterst begränsat. Men genom att använda *in situ* elektroplätning, det vill säga göra beläggningar i formrum hos redan färdigsintrade strukturer, kan ytterligare funktionalitet uppnås. I detta arbete har denna teknik använts för att infoga koppar som sedan oxiderats för att kunna användas som en syrekälla i mikroförbränningskammaren. På liknande sätt har elektropläterade beläggningar av silver använts både för att skapa elektriska element med lägre resistans och för att skapa alternativa katalysatorer. Genom att undersöka flera av komponenter vid temperaturer upp till 1000°C och även i kontakt med aggressiva kemikalier, har arbetet visat att HTCC är ett lämpligt konstruktionsmaterial för mikrosystem som ska tåla extremt tuffa miljöer.

Men det återstår flera utmaningar som behöver övervinnas, den kanske viktigaste är hur dessa system ska kunna kopplas ihop med omvärlden. Framförallt är det de höga termiska spänningarna mellan material som utvidgar sig olika mycket med temperaturen, samt kemiska reaktioner, t ex mellan olika metalliseringar, som behöver övervinnas.

7. Acknowledgement

For me it was a unique experience to do my PhD at MST. During this long journey, I indeed enjoyed working and/or socializing with MST, TMV, MSL, and CNDS people. Hereby, I would like to thank you all.

Klas, thanks for trusting and giving me this opportunity. Your valuable advices are highly appreciated.

Lena, you are a “not very often seen” combination of a good engineer, researcher and teacher. But all those are overshadowed by what a nice person you are! I was lucky to have you as my supervisor, both in research and our common hobby, gardening.

Greger, you are a man of perfectionism and details which was one, but not the only reason I was honored to work with you. You see people and you care about them. I always enjoyed our fruitful discussions about almost everything, with commas, and well, your dear carnivorous plants being the most frequent topics. Maybe hard to believe, but I was always very sorry for that table.

Soon to be Major Dr. Peter Sturesson, our collaboration was like HTCC, unique and challenging but with good results. Thanks for all the good times and special thanks to you and Jenny for the Christmas dinners and Kebab adventures, and last but not least, for all the friendship.

Sven, I really miss our Swedish courses, maybe not that early in the morning though, the debates on mechanics versus electronics, and my Bilema bicycle. You are such a nice friend. I always wish you and your family all the best.

Dear Sara and Gemma, I would like to specially acknowledge your kindness and friendship.

Maryam your nice company always made me forget my homesickness.

Johan, Stefan, and Ville, I appreciate all your help during my first days at MST and the thereafter collaborations.

Dear fellow PhD students, Ana-Maria, Frida, Javier, Martin A, Mingzhi, Peter and Seung Hee (in fact Dr. Seung Hee now) thanks for all the co-teachings, courses, conferences and socializations. Wish you all, best of luck with your research.

Martin A, how nice that we always had interesting chats about chemistry, and what a shame that we never managed to do our dream project.

Martin B, your ideas are inspiring and your help unsparing. By the way, I will never forget the photo you took for the report to “Rymdstyrelsen”.

Anders, I would have definitely regretted, if I had left MST without doing a project with such a kind and competent person like you.

Alireza, Fredric, Hugo, Maria, Mathias, Mats, and Stefan, it was very nice to get to know you and have you as colleagues.

Dhananjay and Kristoffer it was a wonderful experience to work on the rocket project with you and share the excitement of a successful firing!

Ingrid, Maria, Per-Richard, and Sara, would I manage with a single administrative task without your help? Definitely not. Thanks for all you do.

Jonatan, thanks that you always show interest in our research and support it by keeping our IT system up running and of course for your nice company.

Aida, Azadeh, Ehsan, Guilnaz, and Hamed, your friendship and support during the adventure of moving to a new country were of great value for me. All the sweet memories I share with you are unforgettable.

Azadeh, no surprise that we are friends for more than 20 years. You are a real friend, a friend that I can always trust and consult with.

Dear Ashkan, Azadeh, and Sana thanks for proofreading, especially with such a short notice, and thank you Amir for your help with the front page design.

My dearest family, relatives, and friends (especially my lovely Elmira) in Iran, nothing that I was granted in life was as valuable as having you, and nothing that I have achieved in life made me as delighted as the times I spent with you. Thanks for all your support.

Mother and father, no word is enough to describe what you have done for me and how much you mean to me. No sentence can show my deep appreciation. You are the best.

Iman, Amir, and Sana: how fortunate I am to have siblings like you. Always love you, always proud of you. Ashkan, now my happiness is complemented, having also you as my dear brother.

Grandfather, the only regret of my dissertation is you not being alive to see it coming true. I will never forget you.

خانواده و دوستان عزیزم،

هیچ نعمتی در زندگی من به ارزشمندی داشتن شما نیست و هیچ دست آوردی برایم لذتی به شیرینی لحظات با شما بودن به همراه نداشت.
از صمیم قلب از همه حمایت‌هایتان سپاسگزارم و عاشقانه دوستتان دارم.

از دست و زبان که برآید، کز عهده شکرش به در آید...

References

- [1] “EM_DAT, The International Disaster Database,” *Université catholique de Louvain*. [Online]. Available: <http://www.emdat.be>. [Accessed: 14-Sep-2016].
- [2] S. Knaust, “Microsystems for Harsh Environments,” *Acta Universitatis Upsaliensis*, 2015.
- [3] “Environmental engineering considerations and laboratory test, MIL-STD-810G,” 2008.
- [4] W. C. Wilson and G. M. Atkinson, “Passive wireless sensor applications for NASA’s extreme aeronautical environments,” *J. IEEE Sensors*, vol. 14, no. 11, pp. 3745–3753, Nov. 2014.
- [5] H. Pinkerton and G. Norton, “Rheological properties of basaltic lavas at sub-liquidus temperatures: laboratory and field measurements on lavas from Mount Etna,” *J Volcanol. Geotherm. Res.*, vol. 68, no. 4, pp. 307–323, Nov. 1995.
- [6] L. Giglio and J. D. Kendall, “Application of the Dozier retrieval to wildfire characterization: a sensitivity analysis,” *J. Remote Sens. Environ.*, vol. 77, no. 1, pp. 34–49, 2001.
- [7] L. Del Castillo, W. C. West, T. Vo, and E. A. Kolawa, “Extreme temperature sensing system for Venus surface missions,” in *Proc. of IEEE Aerospace*, 2008.
- [8] M. J. Madou, *Fundamentals of microfabrication : the science of miniaturization*. CRC Press, 2002.
- [9] L. R. Volpatti and A. K. Yetisen, “Commercialization of microfluidic devices,” *J. Trends Biotechnol.*, vol. 32, no. 7, pp. 347–350, Jul. 2014.
- [10] P. Abgrall and A.-M. Gué, “Lab-on-chip technologies: making a microfluidic network and coupling it into a complete microsystem—a review,” *J. Micromechanics Microengineering*, vol. 17, no. 5, pp. R15–R49, May 2007.
- [11] V. Domnich, Y. Aratyn, W. M. Kriven, and Y. Gogotsi, “Temperature dependence of silicon hardness: experimental evidence of phase transformations,” *Rev. Adv. Mater. Sci.*, vol. 17, pp. 33–41, 2008.
- [12] W. Bludau, A. Onton, and W. Heinke, “Temperature dependence of the band gap of silicon,” *J. Appl. Phys.*, vol. 45, no. 4, p. 1846, 1974.
- [13] C. J. Glassbrenner and G. A. Slack, “Thermal conductivity of silicon and germanium from 3°K to the melting point,” *J. Phys. Rev.*, vol. 134, no. 4A, pp. A1058–A1069, May 1964.
- [14] P. Kundu, A. K. Sinha, T. K. Bhattacharyya, and S. Das, “MnO₂

- nanowire embedded hydrogen peroxide monopropellant MEMS thruster,” *J. Microelectromechanical Syst.*, vol. 22, no. 2, pp. 406–17, 2013.
- [15] C. B. Carter and M. G. Norton, *Ceramic materials: science and engineering*, 2nd ed. Springer, 2013.
- [16] L. E. Khoong, Y. M. Tan, and Y. C. Lam, “Overview on fabrication of three-dimensional structures in multi-layer ceramic substrate,” *J. Eur. Ceram. Soc.*, vol. 30, no. 10, pp. 1973–1987, 2010.
- [17] F. Bechtold, “A comprehensive overview on today’s ceramic substrate technologies,” in *Proc. of 17th European Microelectronics and Packaging Conference*, 2009.
- [18] W. J. Greig, *HDI Substrate Manufacturing Technologies: Cofired Ceramic*. Boston, MA: Springer US, 2007.
- [19] W. D. Callister and D. G. Rethwisch, *Materials science and engineering: an introduction*, 9th ed. Wiley, 2015.
- [20] C. F. Coombs, *Printed circuits handbook*, 5th ed. McGraw-Hill, 2001.
- [21] D. Rouwet, F. Tassi, R. Mora-Amador, L. Sandri, and V. Chiarini, “Past, present and future of volcanic lake monitoring,” *J. Volcanol. Geotherm. Res.*, vol. 272, pp. 78–97, 2014.
- [22] M. Salehpour, K. Håkansson, and G. Possnert, “Accelerator mass spectrometry of ultra-small samples with applications in the biosciences,” *J. Nucl. Instruments Methods Phys. Res. Sect. B Beam Interact. with Mater. Atoms*, vol. 294, pp. 97–103, 2013.
- [23] M. Berglund, “Miniature Plasma Sources for High-Precision Molecular Spectroscopy in Planetary Exploration,” *Acta Universitatis Upsaliensis*, 2015.
- [24] D. R. Hilton, E. Furi, M. D. T. Tryon, A. L. Labonte, and K. M. Brown, “Towards developing the capability for near-continuous sampling and storage of helium in geothermal fluids from regions of crustal unrest,” in *Proc. of 4th Mini Conference on Noble Gases in the Hydrosphere and in Natural Gas Reservoirs held at GFZ Potsdam*, 2007.
- [25] H. J. Tobias and J. T. Brenna, “Microfabrication of high temperature micro-reactors for continuous flow isotope ratio mass spectrometry,” *J. Microfluid. Nanofluidics*, vol. 9, no. 2–3, pp. 461–470, Aug. 2010.
- [26] X. Ning, S. Phadke, B. Chung, H. Yin, P. Burke, and D. R. Sadoway, “Self-healing Li–Bi liquid metal battery for grid-scale energy storage,” *J. Power Sources*, vol. 275, pp. 370–376, 2015.
- [27] J. E. Köhler, L. G. H. Staaf, A. E. C. Palmqvist, and P. Enoksson, “Analytic modeling of a high temperature thermoelectric module for wireless sensors,” in *Proc. of 14th International Conference on Micro and Nanotechnology for Power*, 2014, vol. 557, p. 012085.
- [28] K. Finkenzerler, *RFID handbook : fundamentals and applications in contactless smart cards, radio frequency identification and near-field communication*, 2nd ed. Wiley, 2003.
- [29] S. Li and C. C. Mi, “Wireless power transfer for electric vehicle

- applications,” *IEEE J. Emerg. Sel. Top. Power Electron.*, vol. 3, no. 1, pp. 4–17, 2015.
- [30] J. M. Miller, O. C. Onar, and M. Chinthavali, “Primary-Side Power Flow Control of Wireless Power Transfer for Electric Vehicle Charging,” *IEEE J. Emerg. Sel. Top. Power Electron.*, vol. 3, no. 1, pp. 147–162, Mar. 2015.
- [31] J. S. Ho, A. J. Yeh, E. Neofytou, S. Kim, Y. Tanabe, B. Patlolla, R. E. Beygui, and A. S. Y. Poon, “Wireless power transfer to deep-tissue microimplants,” *Proc. Natl. Acad. Sci. United States Am.*, vol. 111, no. 22, pp. 7974–9, Jun. 2014.
- [32] L. B. Hunt, “The Origin of the Platinum Resistance Thermometer,” *J. Platin. Met. Rev.*, vol. 24, no. 3, pp. 104–112, 1980.
- [33] D. A. Merritt, K. H. Freeman, M. P. Ricci, S. A. Studley, and J. M. Hayes, “Performance and optimization of a combustion interface for isotope ratio monitoring gas chromatography/mass spectrometry,” *J. Anal. Chem.*, vol. 67, no. 14, pp. 2461–2473, Jul. 1995.
- [34] V. Lekholm, A. Persson, L. Klintberg, and G. Thornell, “Investigation of a zirconia co-fired ceramic calorimetric microsensor for high-temperature flow measurements,” *J. Micromechanics Microengineering*, vol. 25, no. 6, p. 065014, Jun. 2015.
- [35] J. T. W. Kuo, L. Yu, and E. Meng, “Micromachined Thermal Flow Sensors—A Review,” *J. Micromachines*, vol. 3, no. 4, pp. 550–573, Jul. 2012.
- [36] M. Berglund, Z. Khaji, L. Klintberg, A. Persson, P. Sturesson, J. Söderberg Breivik, and G. Thornell, “Extreme-temperature lab on a chip for optogalvanic spectroscopy of ultra small samples – key components and a first integration attempt,” in *Proc. 27th Micromechanics and Microsystems Europe Workshop (MME 2016)*, 2016.
- [37] D. Mège, J. Gurgurewicz, J. Grygorczuk, Ł. Wiśniewski, and G. Thornell, “The Highland Terrain Hopper (HOPTER): Concept and use cases of a new locomotion system for the exploration of low gravity Solar System bodies,” *J. Acta Astronaut.*, vol. 121, pp. 200–220, 2016.
- [38] A. Persson, M. Berglund, Z. Khaji, P. Sturesson, J. Söderberg, and G. Thornell, “Optogalvanic spectroscopy with microplasma sources—current status and development towards a lab on a chip,” *J. Micromechanics Microengineering*, vol. 26, no. 10, p. 104003, Oct. 2016.
- [39] H. Jochen, *Stable Isotope Geochemistry*. Berlin, Heidelberg: Springer Berlin Heidelberg, 2009.
- [40] J. Mueller, C. Marrese, J. Polk, E.-H. Yang, A. Green, V. White, D. Bame, I. Chadraborty, and S. Vargo, “An overview of MEMS-based micropropulsion developments at JPL,” *J. Acta Astronaut.*, vol. 52, no. 9–12, pp. 881–95, May 2003.
- [41] J. Guo, J. Bouwmeester, and E. Gill, “In-orbit results of Delfi-n3Xt: lessons learned and move forward,” *J. Acta Astronaut.*, vol. 121, pp.

- 39–50, 2016.
- [42] T. M. Davis, T. L. Baker, T. A. Belchak, and W. R. Larsen, “XSS-10 micro-satellite flight demonstration program result,” in *Proc. 17th Annual AIAA/USU Conference on Small Satellites*, 2003, pp. SSC03–1–IV–1.
 - [43] W. P. Wright and P. Ferrer, “Electric micropropulsion systems,” *J. Prog. Aerosp. Sci.*, vol. 74, pp. 48–61, 2015.
 - [44] K. Palmer, A. Z. Salaverri, J. Sundqvist, Z. Li, B. Kang, B. Yanping, C. Zhang, S. Wu, and T.-A. Grönland, “On the development and demonstration of the NanoSpace Cubesat propulsion module with close-up thrust control,” in *Proc. Spcae propulsion*, 2016, no. 2, p. 3124889.
 - [45] I. Colomina and P. Molina, “Unmanned aerial systems for photogrammetry and remote sensing: A review,” *ISPRS J. Photogramm. Remote Sens.*, vol. 92, pp. 79–97, 2014.
 - [46] ECHA, *Support document member state committee, support document for identification of hydrazine as a substance of very high concern because of its CMR properties*. 2011.

Acta Universitatis Upsaliensis

*Digital Comprehensive Summaries of Uppsala Dissertations
from the Faculty of Science and Technology 1424*

Editor: The Dean of the Faculty of Science and Technology

A doctoral dissertation from the Faculty of Science and Technology, Uppsala University, is usually a summary of a number of papers. A few copies of the complete dissertation are kept at major Swedish research libraries, while the summary alone is distributed internationally through the series Digital Comprehensive Summaries of Uppsala Dissertations from the Faculty of Science and Technology. (Prior to January, 2005, the series was published under the title “Comprehensive Summaries of Uppsala Dissertations from the Faculty of Science and Technology”.)

Distribution: publications.uu.se
urn:nbn:se:uu:diva-302658



ACTA
UNIVERSITATIS
UPSALIENSIS
UPPSALA
2016