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Miniature Plasma Sources for High-Precision Molecular Spectroscopy in Planetary Exploration

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Abstract
Berglund, M. 2015. Miniature Plasma Sources for High-Precision Molecular Spectroscopy in Planetary Exploration. *Digital Comprehensive Summaries of Uppsala Dissertations from the Faculty of Science and Technology* 1253. 53 pp. Uppsala: Acta Universitatis Upsaliensis. ISBN 978-91-554-9245-8.

The prospect of finding life outside Earth has fascinated mankind for ages, and new technology continuously pushes the boundary of how remote and how obscure evidence we can find. Employing smaller, or completely new, types of landers and robots, and equipping them with miniature instruments would indeed revolutionize exploration of other planets and moons.

In this thesis, microsystems technology is used to create a miniature high-precision isotope-resolving molecular spectrometer utilizing the optogalvanic effect. The heart of the instrument, as well as this thesis, is a microplasma source.

The plasma source is a split-ring resonator, chosen for its simplicity, pressure range and easily accessible plasma, and modified to fit the challenging application, e.g., by the adding of an additional ground plane for improved electromagnetic shielding, and the integration of microscopic plasma probes to extract the pristine optogalvanic signal.

Plasma sources of this kind have been manufactured in both printed circuit board and alumina, the latter for its chemical inertness and for compatibility with other devices in a total analysis system. From previous studies, classical optogalvanic spectroscopy (OGS), although being very sensitive, is known to suffer from stability and reproducibility issues. In this thesis several studies were conducted to investigate and improve these shortcomings, and to improve the signal-to-noise ratio. Moreover, extensive work was put into understanding the underlying physics of the technique.

The plasma sources developed here, are the first ever miniature devices to be used in OGS, and exhibits several benefits compared to traditional solutions. Furthermore, it has been confirmed that OGS scales well with miniaturization. For example, the signal strength does not decrease as the volume is reduced like in regular absorption spectroscopy. Moreover, the stability and reproducibility are greatly increased, in some cases as much as by two orders of magnitude, compared with recent studies made on a classical OGS setup. The signal-to-noise ratio has also been greatly improved, e.g., by enclosing the sample cell and by biasing the plasma. Another benefit of a miniature sample cell is the miniscule amount of sample it requires, which can be important in many applications where only small amounts of sample are available.

To conclude: With this work, an important step toward a miniature, yet highly performing, instrument for detection of extraterrestrial life, has been taken.

Keywords: MEMS, MST, Optogalvanic Spectroscopy, Molecular Spectroscopy, Split-Ring Resonator, Microplasma

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Till Karin, Thea och Erik

List of papers

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

I *Operation Characteristics and Optical Emission Distribution of a Miniaturized Silicon Through-Substrate Split-Ring Resonator Microplasma Source*
M. Berglund, A. Persson, and G. Thornell
Journal of Microelectromechanical Systems, 23(6), 2014, 1340-1345

II *Evaluation of a microplasma source based on a stripline split-ring resonator*
M. Berglund, M. Grudén, G. Thornell, and A. Persson
Plasma Sources Science and Technology, 22(5), 2013, 055017

III *Microplasma source for optogalvanic spectroscopy of nanogram samples*
M. Berglund, G. Thornell, and A. Persson
Journal of Applied Physics, 114(3), 2013, 033302

IV *Stripline split-ring resonator with integrated optogalvanic sample cell*
A. Persson, M. Berglund, G. Thornell, G. Possnert, and M. Salehpour
Laser Physics Letters, 11(4), 2014, 045701

V *Improved optogalvanic detection with voltage biased Langmuir probes*
A. Persson, M. Berglund, and M. Salehpour
Journal of Applied Physics, 116(24), 2014, 243301

VI *Evaluation of dielectric properties of HTCC alumina for realization of plasma sources*
M. Berglund, A. Persson, and G. Thornell
Journal of Electronic Materials, Submitted

VII *Manufacturing Miniature Langmuir probes by Fusing Platinum Bond Wires*
M. Berglund, P. Sturesson, G. Thornell, and A. Persson
Journal of Micromechanics and Microengineering, Submitted

Author's contribution to the publications

I. Most of planning and analysis, all experimental work.
II. Most of planning, experimental work, and analysis.
III. Major part of planning, experimental work, and analysis.
IV. Major part of planning and analysis.
V. Major part of planning and analysis, part of experimental work.
VI. Most of planning and analysis, all experimental work.
VII. Most of planning, experimental work, and analysis.

Contents

Abbreviations

EMC	Electromagnetic Compatibility
FFT	Fast Fourier Transform
HTCC	High-Temperature Co-fired Ceramics
ICOGS	Intra Cavity Optogalvanic Spectroscopy
IR	Infra Red
MEMS	Microelectromechanical Systems
MSRR	Microstrip SRR
MST	Microsystems Technology
OG	Optogalvanic
OGE	Optogalvanic Effect
OGS	Optogalvanic Spectroscopy
PCB	Printed Circuit Board
QCL	Quantum Cascade Lasers
RF	Radio frequency
SNR	Signal-to-Noise Ratio
SRR	Split-Ring Resonator
SSRR	Stripline SRR

1. Introduction

Does life exist outside of Earth? This is a question that has puzzled mankind for thousands of years. We find traces of these thoughts in legends from as early as the Sumerian civilisation 5000 years ago, through written sources from ancient Greek history to early modern thinkers like Giordano Bruno, the 16th century philosopher [1].

> Innumerable suns exist; innumerable earths revolve about these suns in a manner similar to the way planets revolve around our sun. Living beings inhabit these worlds. – Giordano Bruno [2]

In the search for life outside of Earth, Mars is one of the best candidates because of its similarities and proximity to earth. Next to the Moon, it is also the easiest celestial body to land on, making it convenient for exploration. In the late 19th century, optical illusions on the Martian surface sparked theories of canals dug by extinct civilizations. This theory was the inspiration behind H. G. Wells *The War of the Worlds* published 1898, further popularizing the idea of aliens on Mars.

When speaking of extraterrestrial life, we really don't know what we should expect to find. The only known biochemistry of life is found on Earth, using

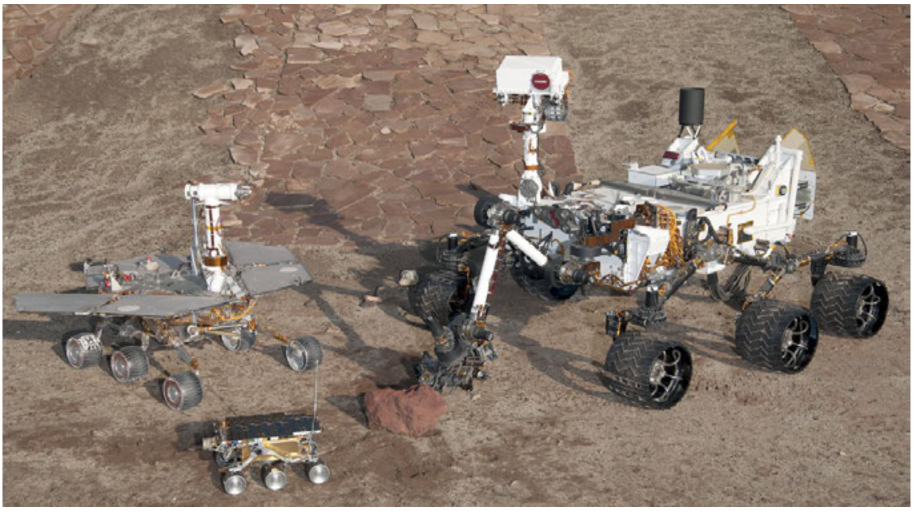

Figure 1.1. Three generations of Mars rovers. From left to right, Spirit/Opportunity, Sojourner, and Curiosity. Courtesy of NASA/JPL-Caltech.

carbon for structural and metabolic functions, with water as solvent. However, life could theoretically be based on other atoms such as silicon or germanium [3]. To prove any form of life, past or present, there are indicators, so called biomarkers which are anything from molecules and isotopes to phenomena, deemed needed to support life, or be caused by life. Two of these biomarkers, water and oxygen, were searched for in the earliest spectroscopy measurements [4, 5] of the Martian atmosphere in the end of the 1800's. In one of these, W. Campbell tried to disprove the existence of water canals on the planet [5].

Regardless, we have not given up on the idea of finding traces of life on Mars. A manned mission would be preferable, enabling extensive searches for biomarkers on Mars. Since most humans would like to return to Earth after visiting Mars, the mission is a major undertaking and no serious mission is yet planned. Although, there is a lot of fuss around the *Mars One* project to colonize Mars from crowd funding and by producing a reality TV-show, the project is not very likely to lift off [6].

> It's a unique opportunity to spend the rest of your life with three people who hate humanity so much they want to live on Mars! – The Guardian on Mars One

Whether or not Mars One succeeds, it contributes by raising the interest for pushing the boundaries of humanity and further popularizing space exploration.

The most promising project to facilitate human exploration of Mars is the Orion spacecraft [7], developed by NASA. The Orion spacecraft is not to be confused with the nuclear propulsion project [8] from 1950's, also named Orion, although the latter also has the potential to facilitate human exploration of other planets. However, the Orion spacecraft is still in testing, with the first crewed tests planned for 2021, and the first Mars mission not possible until the 2030's.

For now, we can still send rovers or other robots to search for life and explore the surface of the red planet with more and better instruments and capabilities. In exploring our solar system, as with most things, cost dictates much of what we can do. A significant part of the cost for anything sent into space, is the launch cost, which depends on the weight of the payload. To that end, remotely controlled robots and rovers have a lot to offer compared with human exploration.

As we have started to look more closely on other celestial bodies, Mars in particular, the weight of the rocket increases with the complexity of the mission. The cost of missions to Mars increases exponentially as the mass of the payload increase. Decreasing the mass of a Mars rover would significantly reduce the size of of the rocket needed to put it on the surface of the red planet and thereby the cost. It could also enable one rocket to deliver multiple small rovers in order to cover more ground and to attain redundancy.

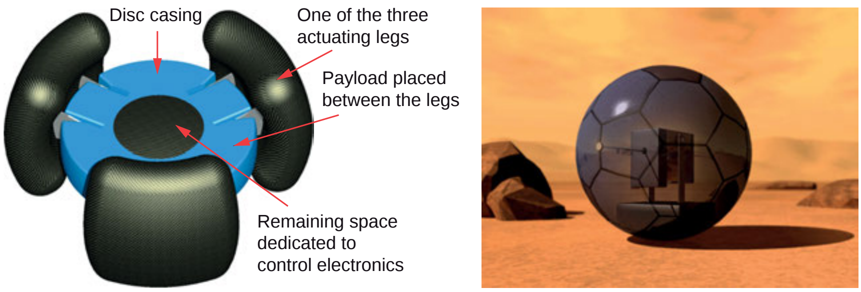

Figure 1.2. HOPTER vehicle (left) capable of exploring rough terrains thought to be inaccessible or too risky to venture by wheel-driven platforms. The platform is being developed by Astronika Sp. z o.o. and SRC PAN. SMIPS (right) is a low energy consumption, dust protected, agile, and compact robot with its characteristic inflatable shell. Courtesy of Per Samuelsson.

The first rover mission to Mars launched in 1996, Sojourner of the Mars Pathfinder mission, cost \$0.28 billion and weighed 11 kg, while the most recent, as of 2015, Curiosity, cost \$2.5 billion and weighed 900 kg. Curiosity, the rover of the Mars Science Laboratory mission, is large compared with all the earlier rovers sent to Mars. The second and third heaviest, Spirit and Opportunity, weigh 180 kg, figure 1.1. The scientific payload of Curiosity is 75 kg [9]. Out of the scientific instruments, the heaviest is the Sample Analysis at Mars (SAM) suit. SAM weighs 40 kg [10] and can analyze the composition of solid or atmospheric samples. The suit contains three instruments: a gas chromatograph, a quadropole mass spectrometer, and a tunable laser spectrometer. SAM also features a sample handling system that can vaporize solid samples, or sample the surrounding atmosphere and channel the gas to each instrument. The SAM suit primarily looks at carbon-containing samples such as carbon dioxide and methane and their isotope variants.

Miniaturizing this SAM system's three devices and sample handling system, would be a good first step in significantly reducing the mass, and thereby the total cost, of a future miniaturized rover, lander or other platform. A miniaturized high-precision sample analysis system could provide entirely new capabilities, like fractionated exploration, covering a larger area with many small, but capable explorers, like the *Highland Terrain Hopper* [11] (HOPTER). The HOPTER is a new locomotion system that instead of wheels uses three legs to jump its way forward, figure 1.2, utilizing the reduced gravity to its advantage and thereby enabling exploration of previously impassable terrain. The explorer only has about 5 kg scientific payload, making miniaturization of the equipment paramount for its capabilities.

Another locomotion platform is the so called *Spherical Mobile Investigator for Planetary Surface* (SMIPS) [12], where an inflatable shell serves as the wheel, protection, solar panel, and housing of scientific instrumentation, figure 1.2. Here as well, the miniaturization of the scientific payload is central to getting as good performance as possible out of the platform.

This thesis describes the first steps in developing a system that can replace part of the instruments and the sample handling system used in the SAM. The purpose of the work presented here was to miniaturize the largest instrument of the largest rover, and thereby build the foundation for a microrover or another small-scale exploration platform.

It should be noted, as with any space exploration driven technical development, that this instrument should provide technological advances in other areas as well. Examples of these include archaeology, medicine (e.g. breath analyzers) and security.

2. Background

As with any new space technology, miniaturized systems have a hard time establishing themselves in the space industry, due to the Catch 22 of the business: Only already flown technology is approved for flying. This is contrary to popular beliefs that, for instance, the technology on satellites sent into space is on the cutting edge of what is possible. Instruments have to not only fit the mission, but need extensive testing before being rated space qualified [13]. However, before the qualification process can begin though, the instrument needs to be designed, manufactured, and proved working.

One tool for manufacturing microscale hardware is Microsystems Technology (MST) [14]. This is used to manufacture microscale sensors, actuators, fluidics, optics, radio frequency (RF) components, energy harvesters and more.

In order to miniaturize a whole scientific instrument, a measurement technique that scales down well with size needs to be chosen, and then, using MST, e.g., be made as small as necessary. Scaling well means that the physics that governs the function of an instrument is either not affected or behaves better at a smaller scale. Typically, miniaturizing the core of a system is not enough, but the surrounding apparatus also needs to be made smaller and lighter. This may be accomplished by providing as much integration as possible into a so called *lab on a chip* with an uncomplicated control system, and as few and simple interfaces, as possible.

In this work, the ^{12}C to ^{13}C isotopic ratio biomarker in CO_2 has been the main goal of detection. The reason the ^{12}C to ^{13}C ratio is a biomarker is that living organisms on earth prefer ^{12}C in its biosynthesis, and if earth-like life ever existed on Mars, this ratio should, like on Earth, vary in fossil deposits compared with the average ratio [15].

Detection of other molecules, such as methane and oxygen [16] and their isotopic variants, is also important, but ^{12}C and ^{13}C isotopes in CO_2 are a good starting point.

To find these biomarkers, both a good baseline and time dependent measurement of the atmosphere, in addition to samples of regolith and bedrock, needs to be analyzed. This puts some specific demands on the instruments, which needs to be versatile and able to analyse both the atmosphere and solid samples.

Before being analyzed, solid samples need to be gasified, for example using combustion, that is, high temperatures and oxygen, before being analyzed in a gas spectrometer. Therefore a microcombustor is needed, which is being developed in parallel [17] to the work presented in this thesis, to be integrated with the detector. This combustor presents new challenges in MST, as high-temperature materials are needed to accommodate the process. Together with the combustor and the spectrometer, components between the two, such as valves, filters, and external interfaces, are developed as well. All of this is going into a complete integrated analytical system, a so called Micro Total Analysis System [18].

2.1 Microsystems Technology

The work described in this thesis was performed at the Ångström Space Technology Centre (ÅSTC), a research group in the Division of Microsystems Technology at Uppsala university. ÅSTC is devoted to the implementation of microsystems in space applications to reduce cost, increase performance and enable new technologies in space and other harsh environments for exploration and exploitation. As described above, space technology has a lot to benefit from miniaturization, and at ÅSTC this is the core of most projects, where a wide variety of MST techniques and devices are developed for harsh environments.

MST, or Microelectromechanical systems (MEMS), is the technology for manufacturing very small devices. MST originates from the integrated circuits industry, where it was first used to translate an external effect to or from electrical signals, i.e. sensors or actuators. The field has, however, expanded and now includes many different miniaturized systems and components in, e.g., fluidics and optics. Nowadays MST also includes a large variety of materials other than those inherited from the semiconductor industry [19], including plastics and ceramics.

The scale at which MST fabrication is applied varies over several orders of magnitude, from the system level in the scale of centimeters down to thin layers of atoms in the nanometer range [20]. Some scaling effects can be utilized, e.g., in [21], where a laser steering mirror is manufactured, and seemingly impossibly thin arms hold a relatively large mirror with no problem. The laser mirror also accomplishes a large sweep, using the small scale to fit multiple segments, each contributing a small angular deflection, into a total large deflection, in a small envelope.

A drawback of MST devices is sensitivity to its thermal environment. Noise from a variety of sources has to be countered at the same time as sensitivity is maintained.

There are a number of benefits from miniaturizing sensors, actuators, structures and electronics beyond being small. Response times are generally shorter as equilibrium is more readily achieved. Hence, with lower thermal mass, the thermal time constant is shorter, for instance. Also, the power consumption is usually low. For consumer MST, manufacturing is easily done in batches, and

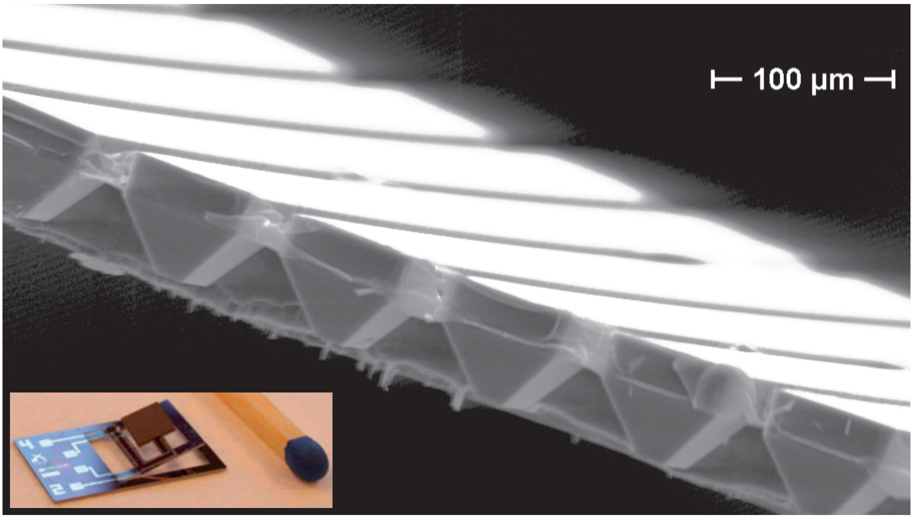

Figure 2.1. Joint of a thermally actuated laser mirror [21]. The difference in thermal expansion between the silicon and the plastic makes the joint flex as it is heated. Each groove contributes with a small angle to the large total deflection.

large amounts of devices can be manufactured simultaneously, lowering the financial and environmental costs of each device drastically. High integration density allows complex systems to be closely integrated.

On the microscale, the phenomena utilized in the, sometimes thousands of times larger, macroscopic counterparts, cannot be assumed to behave the same way. Thus, a good understanding of the physical processes involved and how they scale is central. Modeling and simulations can be a great help to this [22].

In order to reduce the complexity of the electronics and external support equipment, as many parts as possible of an entire system should be integrated, leaving only the absolutely necessary parts macroscopic in size.

A lab on a chip is a device that includes many of the steps needed for an analysis or synthesis on microscale on a single chip. However, the complexity of a lab on a chip can vary drastically. The benefits of including as many or all steps of a process on a single chip are many. First and foremost is the small sample volume of analytes required, because of none or small dead volumes, furthermore the interfaces are often fewer and simpler. The size of the entire system is minimized.

One challenge with a lab on a chip is to integrate widely different components into a monolithic device when they operate under different conditions or put contradictory requirements on the shared facilities. Moreover, manufacturing a chip with many various components often increases the complexity and the number of steps, and therefore reduces the yield.

MST manufacturing is often done in layers. By either adding a layer or removing parts of a layer in a series of steps, complex structures can be realized.

In this work, one of the manufacturing methods used involves stacks of cut and metal patterned ceramic tapes that are laminated and baked into a device. Extensive use of prototyping in plastics and composites are also made to evaluate scaling effects and because of the short idea-to-prototype lead times. In this work a number of MST tools were used, not only the manufacturing techniques usually associated with MST/MEMS production like lithography, deep silicon etching, and sputtering. Characterization, physical evaluation, modeling, and statistical analysis techniques were also performed using a variety of instruments, both commercial and in-house developed.

2.2 Spectroscopy

In this thesis, MST is used to create a miniaturized spectrometer for use in planetary exploration. A spectrometer can be used to detect trace molecules and isotopic variants by analyzing the quantum physical properties of a gas, either its mass, or its emission or absorption of light. To choose one of these spectroscopic techniques for this application, one had to look at the advantages and disadvantages with respect to, e.g., scalability and interface simplicity.

2.2.1 Mass spectroscopy

In mass spectroscopy the atoms' or molecules' charge-to-mass ratio is measured. By ionizing and accelerating the particles through a magnetic field, and then measuring the effect on the ionized atoms' or molecules' velocity, the mass can be determined. One example is the quadrupole mass analyzer, where the ions are filtered by the trajectory they assume in the presence of a specific RF field between the poles [23].

One problem is that mass analysers require some distance for the particles to travel in order for them to separate themselves according to mass. This, depending on the method used, puts hard restrictions on the minimum size. For example, time-of-flight mass spectrometers need at least 50 mm in one direction [24].

Some of the properties of mass spectrometers benefit from miniaturization; reducing the dimensions enables higher pressure systems, requiring smaller vacuum-pumps. However, the sensitivity does not scale well as the pressure increase makes it harder to distinguish between small differences in mass.

2.2.2 Emission spectroscopy

Emission spectroscopy exploits the fact that the excited electrons in an ionized gas, or plasma, emit light with specific wavelengths as they decay to lower energy bands. The light emitted from one such decay has a specific wavelength making each atom emit a characteristic set of wavelengths. Collecting the light emitted from a plasma and diffracting it into its different wavelengths using a grating, and then projecting it onto a sensor gives a spectrum, identifying the gas.

Such emission spectrometers are quite mature and there are some great examples of capable miniaturized systems [25]. Emission spectrometers are capable of detecting many different gases in a mixture. The sensitivity, however, is not the best, with optical losses in the mirrors and gratings putting a strain on the detector used in order to get an acceptable performance.

Emission spectrometers benefit from long path lengths for the wavelengths to separate, and miniaturization is therefore limited, much like for mass spectrometers.

When scaling down the optics, lenses in particular, it is hard to avoid diffraction effects [26]. It also puts high demands on the performance of the optical elements' surface smoothness. Also, by reducing the optical aperture, the amount of light entering the system, and consequently the signal, is reduced. Without moving parts, good line discrimination is hard to achieve.

2.2.3 Absorption spectroscopy

Infrared (IR) absorption spectroscopy is used to identify molecules, using the fact that molecules absorb light of specific wavelengths depending on the molecules characteristic different rotation and vibration frequencies. By illuminating the sample with narrow-linewidth light from a laser and measuring the intensity of the light leaving it, the number of absorbing molecules are determined.

In order to enhance the signal-to-noise ratio (SNR) some spectrometers, so called cavity-enhanced IR spectrometers, bounce the light back and forth through the sample, using mirrors [27]. Cavity-enhanced cells increase the effective path length through the sample at the cost of the intensity of the light leaving the cell for the detector, pushing the detector toward more expensive and complex designs.

Using cavity-enhanced optical cells is an effective way of reducing the length of a cell. However, the distance between the mirrors cannot be made infinitely short because of optical losses in the mirrors. Therefore, some distance is needed for the cell to be effective.

Recently, IR spectroscopy has gotten a boost by the advent of high-performing quantum cascade lasers (QCL). Modern QCLs [28] can deliver single mode, narrow-linewidth, and tunable mid-IR laser light, well suited for absorption spectroscopy. Furthermore, they are more affordable and more compact than ordinary lasers providing the same performance, potentially over a wide range of IR wavelengths.

2.2.4 Optogalvanic spectroscopy

An effect, previously used to monitor gas lasers, called the optogalvanic effect (OGE) [29], is the effect light has on the impedance of a plasma.

In optogalvanic spectroscopy (OGS), the plasma impedance instead of the transmitted laser intensity is monitored. The impedance change can be detected by monitoring the matching network on the plasma power supply, as this compensates for the impedance change in the plasma. However, a drawback is noise pickup in the power supply.

Another way of measuring the impedance is by electrodes, so called Langmuir probes, inserted into the plasma, which gives a more exact reading. One of the main advantages of optogalvanic IR absorption detectors is that the detector effectively is transparent and enjoys the benefits of cavity-enhanced methods without the loss of signal strength, Paper V.

One of the more recent studies of the usage of the OGE in molecule detection spectroscopy [30] claimed sensitivity enough to detect radiocarbon, ^{14}C, using intracavity optogalvanic spectroscopy (ICOGS). ICOGS is a cavity-enhanced OGE detector utilizing the laser cavity instead of a separate cell. In [30], the ^{12}C to ^{14}C ratio was determined using lasers specific to each isotope, using a rather simple RF plasma [31] to detect the signal. Attempts to reproduce the results [32] have shown that the ICOGS setup suffers from problems with instability and reproducibility, making the technique unsuitable for radiocarbon detection. Many of these problems can be attributed to the crude plasma source used.

OGS is a form of absorption spectroscopy and therefore shares many of its properties. However, since the impedance of the plasma is measured only at a single point of the plasma, the total volume of gas illuminated by the laser is not as important as the number of bounces through the measured plasma, and OGS does not have a minimum length between the mirrors to effectively exploit cavity-enhanced cells.

Hence, a new, more stable and reliable plasma source could bring cavity-enhanced OGS up to speed.

Given the low vacuum requirement, the high sensitivity and the simplicity, the OGS was chosen for this thesis work.

2.3 Plasma

At the heart of three of the four spectrometers mentioned above, is often an ion source, light source, or an excited gas, that can be supplied by a plasma.

2.3.1 Definitions

The fourth state of matter, after solid, liquid, and gaseous, is the plasma, where a gas is ionized. On Earth, some of the natural sources of plasma are lightning as well as fire. Man-made sources of plasma include neon signs, low-energy lamps, plasma TVs and plasma processing.

A plasma is defined as a collection of neutral molecules, free electrons, and ions, in close enough proximity to each other to interact. Inside a plasma, the essential mechanisms are excitation and relaxation, ionization and recombination of the gas into ions and electrons. The recombination process must be countered by an external energy source continuously ionizing the gas in order to maintain the plasma [33].

The bulk of a plasma is neutral, that is, contains an equal amount of electrons and ions. At the boundaries, the balance of the number of electrons and ions is shifted.

A plasma is described by a number of parameters; similarly to a gas, it is described by its molecule content, pressure, volume and temperature. The temperature and density are stated individually for each particle category: neutral gas, ions, and electrons.

Plasma also has a number of unique attributes, among others, Debye length, plasma density, and plasma potential, which is the potential in the neutral bulk of the plasma.

2.3.2 Plasma sheath and probes

In addition to the plasma potential, there is the floating potential that is generated on an electrically isolated conductor inserted in the plasma, as it gets struck by electrons and ions. The initial current density of electrons is much higher than that of ions since the mobility of electrons is much higher. Because of this difference, the conductor starts to build a negative charge with respect to the plasma, and the conductor begins to repel electrons and attract ions. This continues until the ion and electron currents cancel each other and the system reaches equilibrium, figure 2.2. The potential at equilibrium is the floating potential [34].

The bulk of a plasma is virtually free from electric fields since the mobility of ions and electrons readily compensates for the smallest perturbation. This is not the case for objects inserted in or around the plasma. Because of the high mobility of electrons, the floating potential is always lower than that of the plasma. In fact, this mechanism makes the plasma potential the highest potential in a system.

Around an object inserted into a plasma, grounded or biased to a potential, a region around the object with a lower density of electrons is formed as described above. This is called a plasma sheath. Across this sheath, a potential difference is present in order to repel the electrons, called sheath potential. Because of the lower density of electrons, this region does not glow as much from electron relaxations, and is therefore also called dark space.

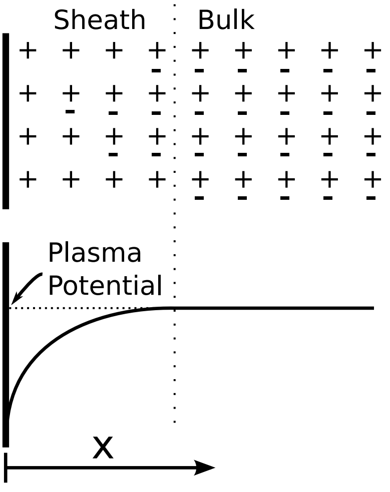

Figure 2.2. Sheath formed around a conductor inserted into a plasma, with its charge distribution (top) and potential distribution (bottom).

If a probe, maintained at a potential, is inserted into a plasma, a current will start to flow, the direction being dependent on its relation to the floating potential. By varying the potential, usually with respect to the grounded chamber walls, and measuring the current, an I-V, or Langmuir, curve can be determined. From this, a number of plasma parameters can be determined, such as, electron and ion density and temperature, floating potential, and plasma potential. Such plasma probes are often called Langmuir probes.

2.4 Microplasma

Often when we think of plasma, it is on large scale, like stars or aurora borealis/australis, or in, e.g. plasma processing and lights on the scale of a few hundred mm. Microplasmas, which much less often comes to mind, are plasmas the size of a few millimetres down to micrometres. These exhibits a large surface-to-volume ratio that very much affects the balances in the plasma. One example is the electron and ion densities. Since much of the volume of a plasma chamber in these scales is dominated by dark space, the densities can no longer be said to be equal. With a higher ion than electron density, the plasma potential in microplasmas is generally higher.

Microplasma sources are of interest for microsystems that require reactive environments, ionization or light sources. Some of the uses microplasma sources have been put to so far, include plasma screens, gas analysis, photodetectors, micro lasers, radiation sources, biomedicine, synthesis, sterilization, micro-chemical analysis, microwave components, and air flow control. [35]

During the last two decades' development of MST technologies, the microplasma field has thrived.

Because a plasma cannot be much smaller than a few Debye lengths the pressure in microplasmas are generally very high compared with macroscopic plasmas. Microplasmas are often very cold, that is, the electron temperature is much higher than the ion temperature. Even though a microplasma source requires little power, the power density in the plasma is generally high [36].

Some of the problems plaguing microplasmas are short lifespans and contamination [36]. The plasma is sometimes hard to characterize as it is difficult not to disturb the measuring of the plasma with the measuring probes.

Some of the non-invasive ways to characterize microplasmas used are emission spectroscopy, absorption spectroscopy, and laser-assisted Thomson scattering. Langmuir probes can still be used for relative measurements and for special applications.

As with regular plasma sources, there are several ways to drive microplasma sources. In fact, plasma sources are often categorized by their power source. Direct current and high-frequency plasma sources generally require very simple power sources but the plasma is hotter, energy is lost to ions moving through the sheaths, and the electrodes gets sputtered. Pulsed plasmas have a lower ion temperature and a higher electron temperature, and more effectively use the power supplied. Microwave, or RF, plasma sources require more complex power supplies. However, low-effect components are small, readily available, and reasonably priced. The plasma created by a microwave plasma source is dense, stable, cold, and can be operated at higher pressures, and the sheath potential scales down with the frequency. Microwave microplasma sources are a good choice for analytical applications, because of the plasma parameters and favourable scaling. [37]

There is quite a range of RF plasma sources. One of the more recent designs is the split-ring resonator (SRR) [38], which is a dipole antenna folded into a circle where the endpoints meet and form a discharge gap. This design has an appealing simplicity, the plasma is generated in a small concentrated volume, and the structure requires no additional matching network. In addition, performance is very good.

The plasma source used in OGS needs to be stable, shall operate at around 10 mbar, and have an easily accessible plasma. Hence, the SRR microplasma design was chosen as the basis for the plasma source in the miniature optogalvanic (OG) spectroscope of this thesis work.

3. Miniature spectroscopy

3.1 Split-ring resonator plasma sources

After choosing SRR as the most promising design, it needs to be optimized and prepared for integration.

3.1.1 Designing an SRR

The design process of a microstrip SRR (MSRR) is straightforward; knowing the basic properties of the substrate and choosing a frequency, a microstrip can be designed. The quality factor, characteristic impedance, width, and length of a half period microstrip, is then used to calculate the entire geometry. The SRR is fed RF power at the resonance frequency of the resonator at a small offset angle from the center of the strip, in order to match the impedance of the resonator with that of the feeding waveguide. This offset angle, θ, is determined by the impedance and the quality factor:

$$\theta = \arccos\left[1 - \frac{Z_{in}\pi}{Z_0 Q}\right] \tag{3.1}$$

where Q is the quality factor, Z_{in} is the input impedance, usually 50 Ω, and Z_0 is the characteristic impedance of the microstrip that makes up the MSRR. In figure 3.1 a 900 MHz SRR is ignited in atmosphere. The ring has a plasma gap of 300 µm and a diameter of 30 mm.

3.1.2 Early SRR

The first experiments made in this thesis were to reproduce previous work by Iza et al. [38] with MSRR microplasma sources, where plasma sources were designed and manufactured using printed circuit board (PCB), figure 3.1.

The limiting factor of how small an SRR microplasma can become is that the circumference of the ring has to be half a wavelength. The main method to reduce the size is thus to increase the frequency and/or to switch the dielectric material to a higher relative permittivity material. Silicon is a very attractive material to work with when building miniaturized components, especially when planning to integrate several components into a single chip solution. The fact that many advanced processes are developed specifically for silicon also helps. Silicon also has a high relative permittivity, ε_r, of around 11, which

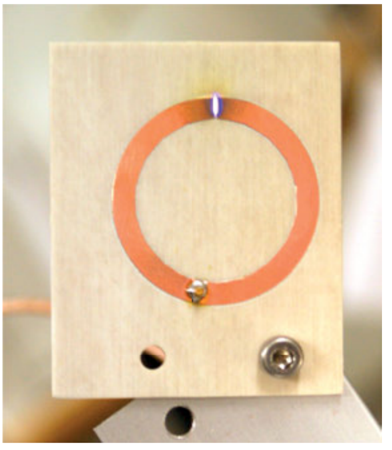

Figure 3.1. The first prototype based on earlier work done by Iza et al. [38]. The plasma source is operated at 900 MHz in ambient air and measures 30 mm in diameter.

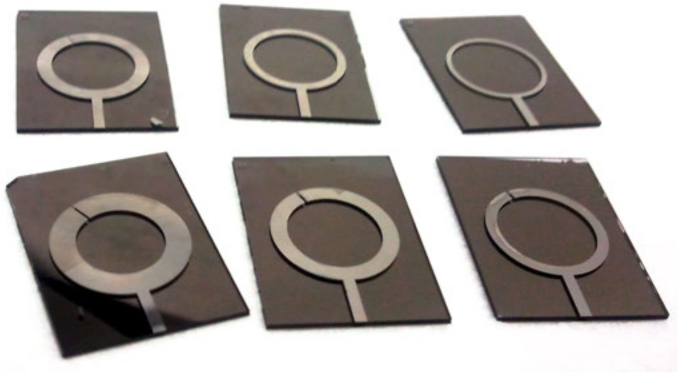

Figure 3.2. MSRR plasma source manufactured using plated nickel on Pyrex.

in combination with a frequency of 2.6 GHz gives a ring diameter of about 6 mm.

By manufacturing MSRR microplasma sources with silicon as the dielectric material and using plated nickel for the conductors, a fully working miniature version of the original design was realized.

This fabrication technique also worked well for other materials such as Pyrex, figure 3.2. Pyrex was used for its optical transmission properties, with the intention of accessing the light from the plasma, out-of-plane of the plasma source. The Pyrex plasma sources were photogenic, but poor adhesion and differences in thermal expansion coefficients made the resonator structure detach when only small amounts of power was fed into the resonator or even during fabrication. Using Pyrex or some other glass material is still feasible, but a metal system with better adhesion is preferred.

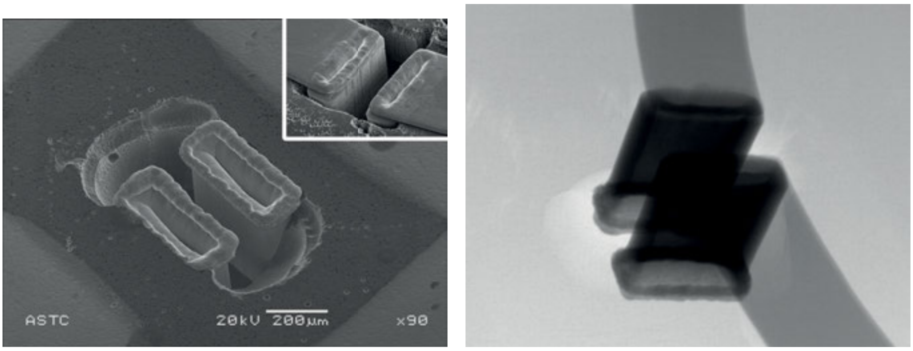

Figure 3.3. MSRR with nickel electrodes folded to line the walls of a opening through the silicon substrate. Scanning electron microscopy image of the electrodes entering (inset) and exiting the substrate are seen to the left, and an X-ray image of the electrodes is seen to the right.

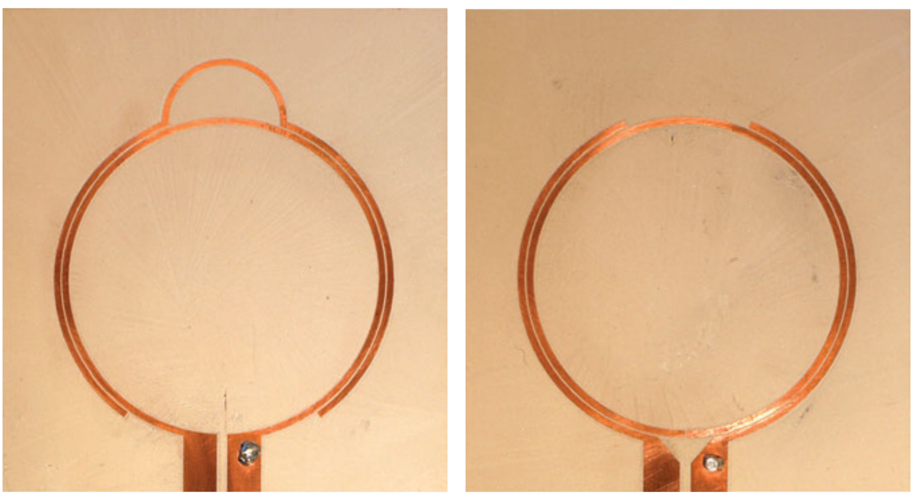

Figure 3.4. Coplanar SRR manufactured on PCB.

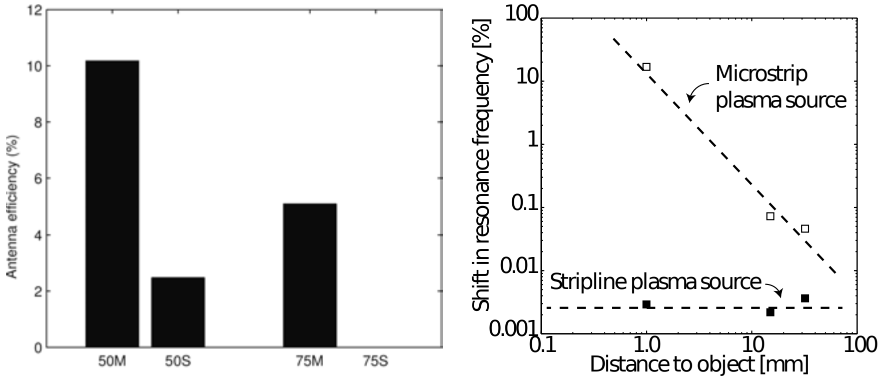

Figure 3.5. Antenna efficiency of four devices, two stripline, 50S and 75S, and two microstrip, 50M and 75M, (left). Resonance frequency shift as two metal plates are introduced in proximity to the devices, showing the detuning of the MSRR (right).

The next step was instead to make an opening through the substrate, using deep reactive ion etching to access the plasma from both sides of the substrate. In an attempt to also increase the volume of ionized gas, electrodes were plated alongside the walls of the opening, figure 3.3. In Paper I, the performance of such plasma sources are characterized. Two modes of plasma ignition were found at different pressures. At relatively low pressure plasma was ignited on the bottom side of the plasma source where the electrodes exited the substrate, but not inside the hole, hence with no new advantage over the original design. At higher pressure, on the other hand, the plasma was confined in the hole between the two electrodes, and narrow and high intensity emission lobes were observed out of the plane of the device.

Although the larger plasma volume could be useful, the manufacturing of these plasma sources in silicon was far from trivial and not suitable for rapid iteration of design changes. Therefore, this material system and the extended electrode concept was put aside until the rest of the system was more mature and needed the features it provided.

Instead of using the original microstrip RF waveguide, a co-planar waveguide device was designed and prototyped, figure 3.4. The co-planar waveguide had no ground plane but instead featured an extra waveguide in the same plane as the ring resonator. The idea was to further simplify the manufacturing to only require one side of the substrate to be metallized and patterned. Another change was to feed the structure with two ports using a balun. Although the design was interesting it was less than optimal for integration with some obvious disadvantages. With no ground plane, the structure became relatively large and very susceptible to its surrounding electromagnetic environment.

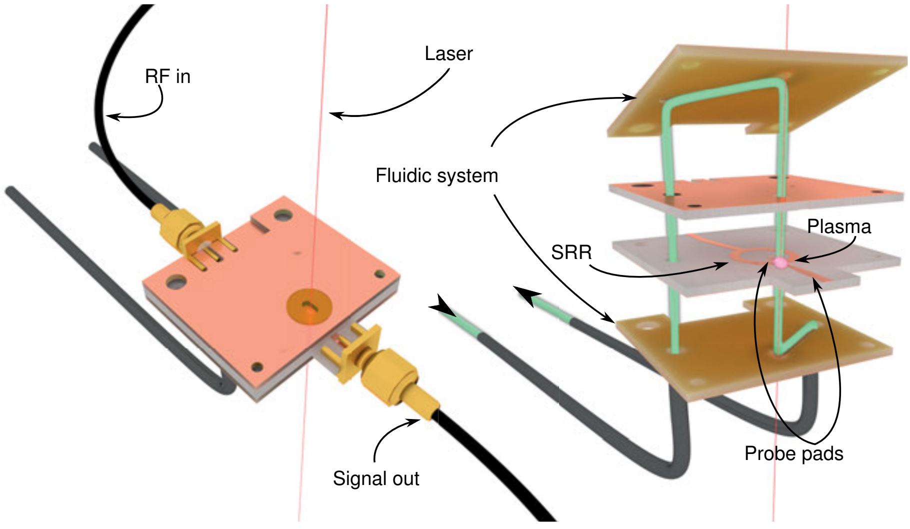

Figure 3.6. Schematic drawing of an SSRR prototype built in PCB with IR-transparent zinc selenide windows, plasma probes, and fluidic system. Two electrical connections, one for RF-power and the other OG signal, are added for completeness. Courtesy of Karin Berglund.

3.1.3 Shielded SRR

The susceptibility to electromagnetic interference inspired the testing of yet a new design, a stripline split-ring resonator (SSRR). It was prototyped and found to work well. A stripline waveguide is a microstrip with ground planes and dielectrics on both sides, effectively enclosing it. Some of the benefits of this design are that the lateral size decreased as compared with the MSRR as the effective permittivity increases. Also, the electromagnetic compatibility (EMC) provided by the shielding of the ground planes enabled close integration to other systems on both sides of the plasma source. In Paper II, a study to determine the performance of the SSRR design compared with the MSRR was carried out. In order to compare the EMC of the two plasma sources, a Wheeler cap antenna efficiency experiment was used in Paper II. A Wheeler cap measurement is performed by measuring the reflected power with and without a metallic enclosure around the plasma source. The results, figure 3.5, indicates that SSRR radiates less power than MSRR. Figure 3.5 also shows the robustness of the SSRR as metal plates were introduced at different distances from the devices.

The studies in Paper II show that with only a minor increase in power consumption, the EMC was greatly improved in the SSRR design. Moreover as an effect of the more confined plasma in the SSRR, the plasma potential increased as more electrons recombined at the walls.

At this point, the plasma was stable and reproducible, and accessible out of the plane of the device. Hence, the SSRR microplasma source was mature

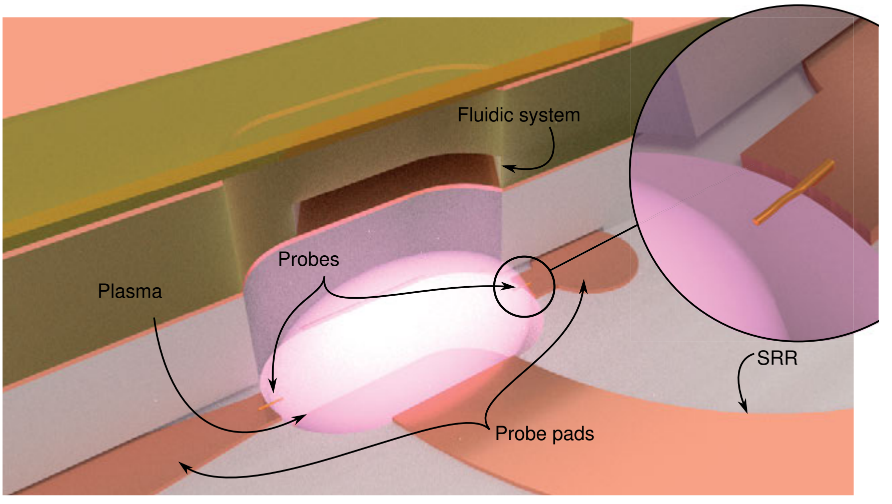

Figure 3.7. Schematic drawing of SSRR plasma source equipped with plasma probes for OGE signal detection. Half of the ground plane and fluidic system is cut away to reveal the internal structure of the SSRR and the fastening points of the probes. Courtesy of Karin Berglund.

enough for close integration, figure 3.6. The first two additions were plasma probes inserted into the plasma, figure 3.7, and a fluidic system that was easily fitted onto the plasma source, conducting the gas through the created plasma cell. Now, the OG signal could be measured.

3.2 Optogalvanic spectroscopy

From previous studies [32] we know that classical OGS suffers from stability and reproducibility issues. In this thesis several studies were performed to investigate and improve these shortcomings, and improving the SNR. In addition to the efforts to improve the signal, some work was put into better the understanding of the underlying physics of OGE as this field is not at all as developed as other spectroscopic methods.

3.2.1 The optogalvanic signal

In order to measure the OG signal, electrical probes were chosen as the method of detection, since the reflected power from the plasma source was hard to get free of noise, and separating the power supply from the signal detection also makes sense for precise measurements.

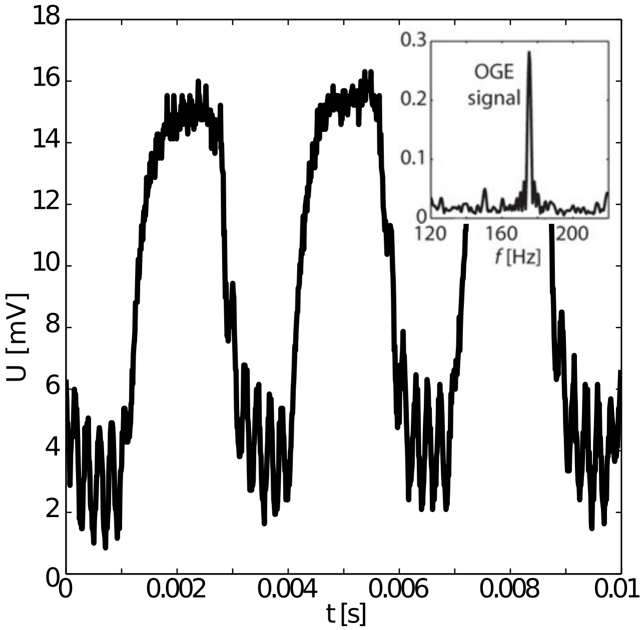

Figure 3.8. Optogalvanic signal in a plasma of 22 Torr CO_2 using a CO_2 laser. As the laser illuminates the plasma the potential drops. The inset shows a typical OGE signal in the frequency domain.

The plasma probes, most often gold bond wires, were inserted into the plasma from copper pads in the same layer as the resonator structure, figure 3.7.

When measuring the OG signal, the laser beam is modulated, usually using an optical chopper, and the perturbation in the plasma appears as a potential shift over the two probes in the form of a square wave, figure 3.8. This approach is convenient in order to remove the background by comparing the potential with and without the laser beam present. The square wave amplitude is in the simplest approximation of the OGE linearly dependent on the volume density, n_m, of the studied molecule. Using the fast Fourier transform (FFT), the amplitude of the OG signal was easily extracted. The OG signal, U_{OG}, can then be described by:

$$U_{OG} = n_m I \sigma_L \pi l r^2 K, \qquad (3.2)$$

where I is the laser intensity, σ_L the absorption cross-section of the photon-molecule interaction, $\pi l r^2$ the cylindrical volume in the plasma that is exposed to the laser beam, and K is a proportionality parameter, which will be discussed in more detail in section 3.2.3.

In Paper III the OG signal dependence on several external parameters were investigated. The chopper frequency, RF driving frequency and power were tested.

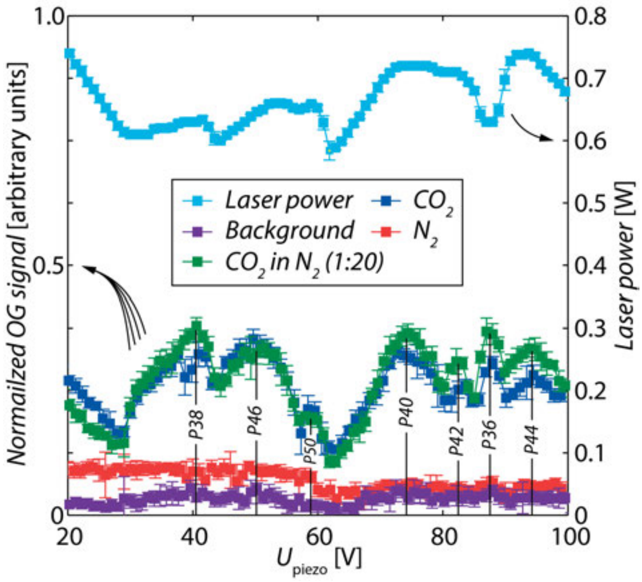

Figure 3.9. Spectra of different gases and mixtures acquired by scanning the wavelength of the CO_2 laser. The measured OG signals were normalized by the laser power, which is shown for comparison.

3.2.2 Stability

In Paper III a semi open cell operated inside a vacuum chamber was used, whereas, in Paper IV the enclosed nature of the SSRR was utilized when attaching a simple fluidic system, figure 3.7. This enclosure resulted in improved stability, from 20 to 40 s, measured using Allan variance. The Allan variance describes for how long a signal can be averaged before the standard deviation starts to increase from, e.g., drifts in the system. Additionally, the SNR of the system increased with 10 dB after enclosing the SSRR.

In addition to increasing stability the miniaturized plasma chamber provided another useful property; the total amount of sample gas becomes minuscule. Paper III demonstrates that analysis of samples down to the nanogram range will become feasible.

In Paper IV the reproducibility of the measurements with the SSRR was investigated. In this study, a much better reproducibility was found compared with the original RF OG cell used for ICOGS, where the average level was improved by a factor of 10.

3.2.3 Probe biasing

In equation (3.2) the parameter K is said to be a constant. However, in most circumstances this is not accurate, as K depends on many parameters, particularly to those of the plasma.

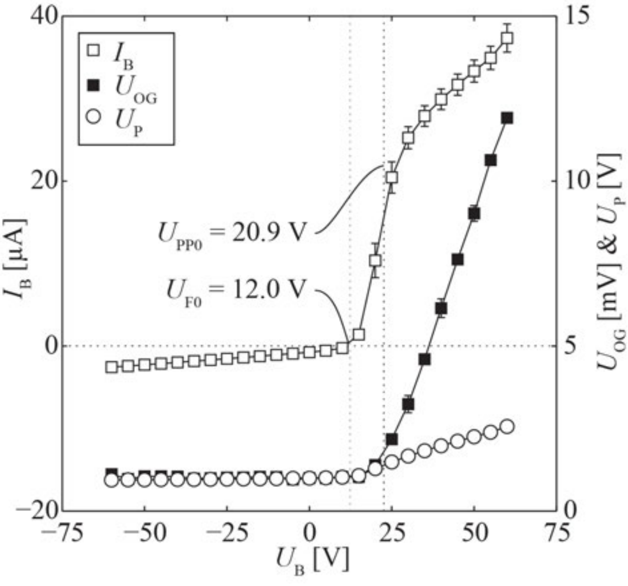

Figure 3.10. Langmuir curve of a pure CO_2 plasma at 4.5 Torr (white squares). The OG signal, U_{OG} (black squares), and the floating potential, U_P (white circles), are also shown at different scales on the right axis.

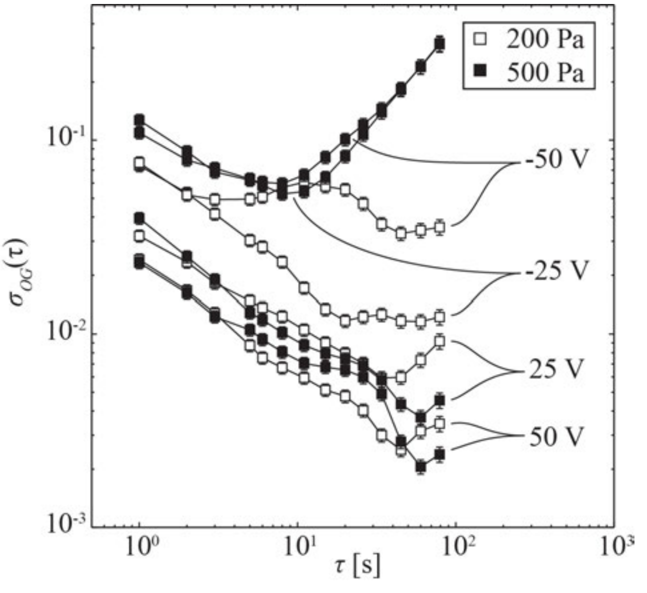

Figure 3.11. Allan variance,σ_{OG} of normalized OG signal at different probe bias and pressures.

In addition to measuring the OG signal, the Langmuir probes were also used to characterize the plasma parameters by I-V measurements. That is, measuring the current as a potential was applied on one of the probes. The results were presented as Langmuir curves, figure 3.10.

During I-V characterization, the electrode that was not biased was connected to electrical ground. If the electrode instead is left floating, I-V characterization can not be made. Instead, the OG signal can be measured on that probe while biasing the plasma with the other probe. In Paper V, it was found that when the biased electrodes potential passed the plasma potential, the OG SNR started to increase linearly, figure 3.10, to about one order of magnitude above its initial value, after which the plasma was quenched. In addition to a better SNR the stability of the plasma was improved. In addition to these empirical observations, a theory of what caused this very desirable effect was presented. Using basic plasma theory, a model is presented in Paper V, to try to explain the effect. In principle, as the bias probe potential increases, it forces the plasma to increase its potential, because of the fact that the plasma potential must be the highest potential in the system. As the bias potential becomes larger than the initial plasma potential, the plasma potential is approximated to be equal to the bias potential, even though the plasma potential in reality will be slightly higher. Figure 3.10 shows how the floating potential of the unbiased probe too increases linearly with the bias/plasma potential, but not as fast. Therefore, the sheath potential must also be increasing with the bias.

In Paper V the parameter K is expanded to include the electron temperature and the plasma potential of the unperturbed plasma. It also included the parameter α, a constant depending on the probe geometry and the mass of the ions. This new formula was shown to fit well with measurements performed in Paper V.

Even though additional effects need to be incorporated into a complete model, the model presented in Paper V explained the observed phenomena, and gave a clearer understanding of the OGE in a microwave plasma. For example, as the bias potential increases, the plasma loses its slow-moving electrons more easily than faster ones, causing an indirect heating of the electrons. It tells us that a cooler initial plasma benefits more from probe biasing, which is also supported by the measurements performed in Paper V.

In Paper V, the long-time stability of the OG SSRR cell was evaluated by the Allan variance at different biases, figure 3.11. As can be seen, the stability improved with increasing bias voltage, and compared with Paper IV a maximum improvement of 40% was achieved, correlating to a significant improvement to the SNR of the entire system.

3.3 Integration

After having achieved such improvements in SNR, stability, reproducibility and EMC it was time to return to integrating the SSRR into a system. The final integrated system should include the plasma source, plasma probes, temperature sensors, pressure sensors, flow sensors, valves, filters, a fluidic system and the combustion chamber mentioned earlier. Left as external devices are a QCL laser, some optics, a vacuum pump, the power supply and control electronics.

Of all the instruments to be integrated, the plasma source and the combustion chamber dictate the choice of material. Silicon would once again be of interest as it is inert, has high ε_r and integrates well with other microsystems. However, silicon does not handle temperatures up to 1000 °C, produced in the combustion chamber. Instead, high-temperature co-fired ceramics (HTCC),

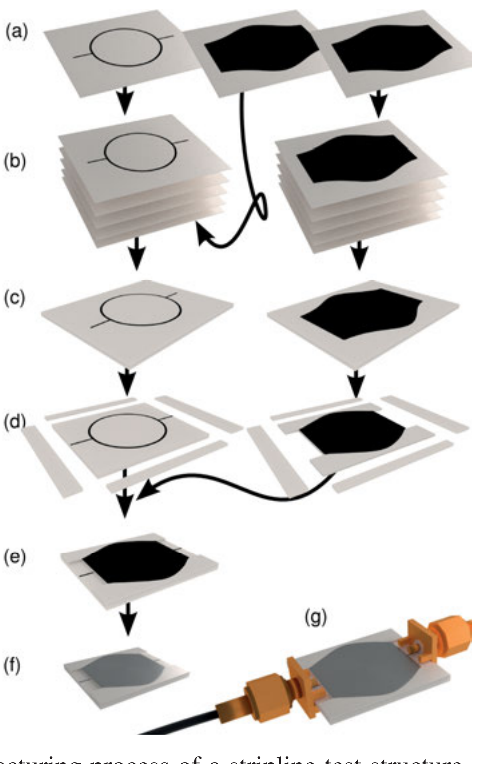

Figure 3.12. Manufacturing process of a stripline test structure. (a) Three platinum structures are screen printed on individual alumina green tapes. (b) From these and blank alumina tapes, two stacks of 5 tapes each are formed. (c) The stacks are laminated. (d) The two halves are cut to fit each other, and openings in the top ground plane are milled to expose the conductors. (e) The two halves are laminated together to a single stack. (f) This is sintered at 1550 °C, causing the device to shrink (and the platinum to change color). (g) Finally, the stripline test structure is mounted with SMA connectors and cables. Courtesy of Karin Berglund

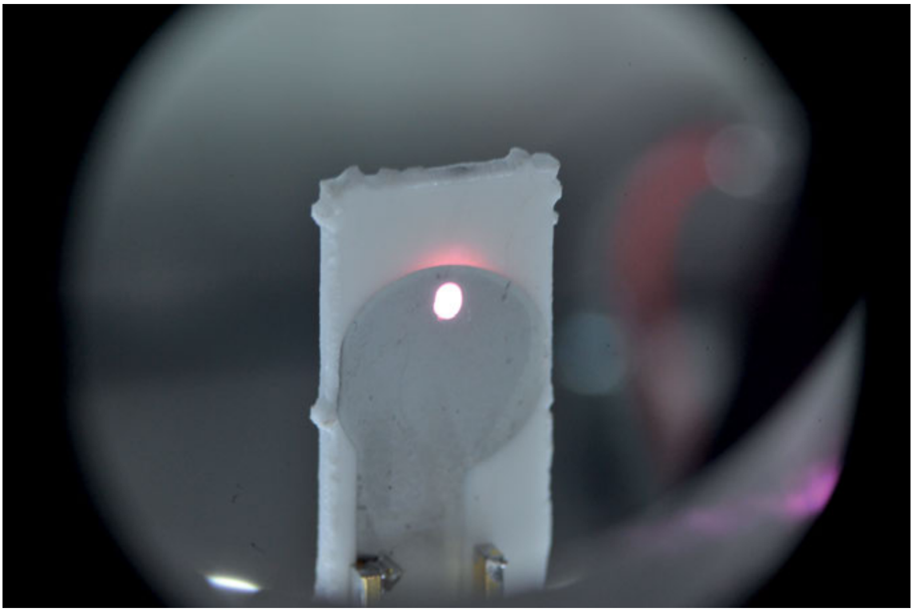

Figure 3.13. Alumina SSRR plasma source, ignited in 25 Torr air.

alumina, was a more logical choice. Alumina too has a high ε_r, making the SSRR smaller, and the inert nature of alumina prevents contamination from the substrate from affecting the measurements.

Co-fired ceramics systems are combinations of dielectric ceramics and conducting metals that are fired together into a monolithic device. HTCC uses tungsten or platinum as the conductive material and pure ceramics as the dielectric [39]. In Paper VI, HTCC alumina with platinum conductors were used to manufacture SSRR microplasma sources. Initially, alumina is mixed with a plastic that is burnt away in the early stages of the firing, to enable structuring the devices in a soft material. The alumina material is usually bought in the form of thin tapes that are machined and then laminated together to form the desired structure. The platinum conductors are screen printed onto the alumina before lamination.

HTCC has primarily been used for packaging of integrated circuits, and has only recently started to attract attention as a platform for MST development [39]. This means that the dielectric properties of HTCC alumina is still not very well investigated [40].

Using a resonator test structure, Paper VI investigates the relative permittivity, ε_r and the dielectric losses, $\tan\delta$, of HTCC alumina, and the resistive losses of two different screen-printed platinum pastes.

The method of manufacturing a stripline structure in HTCC alumina is described in figure 3.12 and Paper VI.

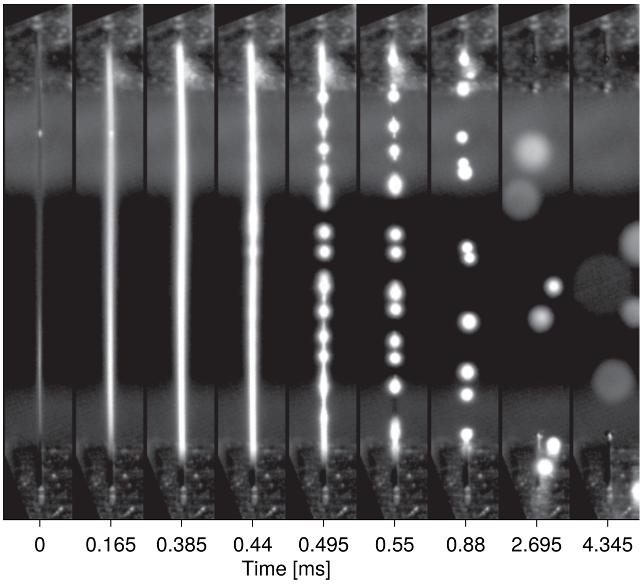

Figure 3.14. High speed camera footage of a platinum wire fused using 5 A. Notice how the entire wire melts into small droplets.

The RF properties of a number of test structure devices were measured in Paper VI, using a network analyzer, X-ray, scanning electron microscopy, and four-point sheath resistance probes. The data from these measurements were then used to design RF components in HTCC alumina.

In Paper VI, the ε_r of the alumina was found to be 9.65 ($\pm$0.14) and $\tan\delta$ 0.0011 ($\pm$0.0007), and the bulk resistivity of the most favourable platinum paste was 227 ($\pm$8) nΩm. These parameters were used to manufacture SSRR microplasma sources, figure 3.13.

With working plasma sources built in alumina, the intention is to manufacture all the other parts of the integrated systems in HTCC ceramics, and ultimately integrate them all into a single chip.

3.4 Plasma probes

As a first step of this integration, focus was directed to the Langmuir probes which were one of the most important parts of the OGS concept in this thesis.

The two metal probes used as plasma probes are inserted into the plasma from pads in the same plane as the resonator ring of the SSRR, and were usually made from bonded gold wires, figure 3.7.

After switching material system to HTCC, this method was no longer feasible, since it is nearly impossible to bond wires onto soft tapes and platinum not yet hardened by the firing process. Also, gold was no longer a good material

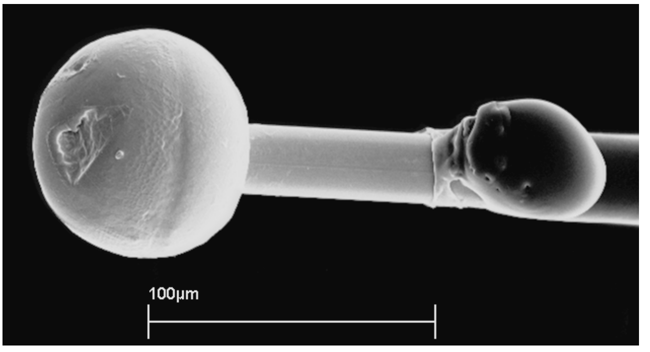

Figure 3.15. Platinum bond wire fused with a thin coating of Parylene for insulation of the stub.

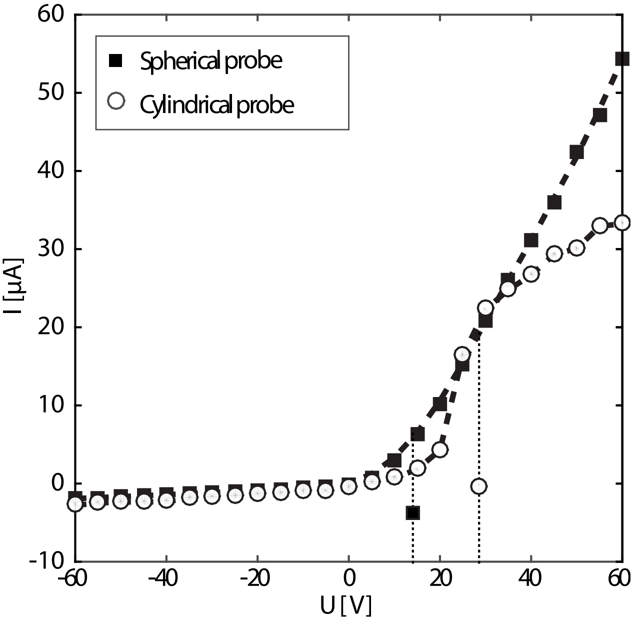

Figure 3.16. Comparison of the shape of Langmuir curves captured using the original, cylindrical, probe and the Parylene insulated spherical probes. The two measurements were performed at different pressures and with two different SSRR PCB plasma sources, therefore only the shapes of the curves are comparable.

for the probes, since it would not survive the firing. Hence, a new method was needed to form the probes.

Instead, the possibility of stretching a platinum bond wire across the gap of the SSRR before lamination, and then fusing it into two probes after firing, was investigated. Fusing is the process of conducting current through the wire until it melts, leaving two stubs at each end, in the same configuration as the bonded plasma probes. One added benefit would be if droplets formed at the endpoints of the stubs and if the metal stubs up to the droplet could be isolated, so that spherical Langmuir probes could be realized. The benefit of having isolated wires with spherical ends inserted into the plasma instead of fully exposed cylindrical wires is that a change in the plasma distribution does not affect the collecting area. Additionally, the collection area of the probe would expand spherically when the probe is biased.

In Paper VII, an investigation was made to characterize the fabrication of such probes using fusing. Initially, only the bond wires were of interest and the study was preformed on PCB. Further on, however, the intention is to incorporate the probes into the alumina HTCC SSRR.

After fusing more than 200 platinum wires, response surfaces for different parameters such as the length of the stubs, and the surface area and shape conformity of the droplets, as a function of current and voltage limits of the power supply used, were created. In addition, the dynamics of the fusing were investigated using a high-speed camera, figure 3.14.

Experiments with platinum wires coated in Parylene were also carried out with some success, figure 3.15. A comparison between the shape of a Langmuir curve captured using an original, cylindrical, probe and a Parylene insulated spherical probes, similar to the one in figure 3.15 was also performed. The result from this comparison, figure 3.16, shows the expected characteristic curve shape of a spherical probe, indicating that the fabrication process described in Paper VII can be used to make well-functioning spherical Langmuir probes. Their performance in OGS will now be investigated.

4. Conclusions and outlook

In this thesis, the first microplasma source ever used in OGS has been developed, providing better SNR and reproducibility than the macroscopic plasma source traditionally used in this field.

The plasma source is based on a MSRR plasma source equipped with an extra ground plane, forming a SSRR. The SSRR is equipped with bond wire Langmuir probes, creating robust and stable plasma sources, and constitutes an excellent platform for further OGS development.

Using the SSRR instead of the traditional RF plasma tube in an OG spectrometer provides far greater long-term stability for the system. The increase goes from an original few seconds up to about 100 seconds of time over which the signal can be averaged before drifts in the system makes further measurements pointless. Furthermore, SSRR OGS cells provide much better reproducibility compared with their traditional counterparts.

The SNR of the system has been continuously improved by, among other things, enclosing the sample cell, employing a more stable laser, and by biasing the counter electrode during measurement. The total SNR improvement exceeded 40 dB.

The stripline design of the plasma source provides improved EMC, making the resonator virtually unaffected by its electromagnetic surrounding, in opposite to its microstrip counterpart. The power radiated from the resonator was also reduced from $\sim$7.5%, to $\sim$1.3%.

The sample cell formed by the hole through the stripline, i.e., the gap where the plasma is ignited, provides a very compact sample cell that can be filled to the operational pressure using only nanograms of sample.

One of the major benefits of SSRR OG spectrometers, used as IR detectors, are their inherent simplicity. The out-signal is a slow AC signal, easily collected by simple electronics. The power supply required to ignite and maintain the plasma is made out of readily available, off-the-shelf, RF parts. Using multiple lasers to detect different molecules, modulated with different frequencies provides simultaneous detection of several species.

Switching the building material to HTCC alumina and platinum provides an inert plasma environment, free from contamination, in addition to great heat tolerance from nearby components, figure 4.1.

The new building material required a new method of manufacturing the two Langmuir probes. The new method of fusing a platinum wire into two probes provides a useful way of manufacturing miniature spherical Langmuir probes. These probes worked well and produce the characteristic Langmuir curve of a spherical probe.

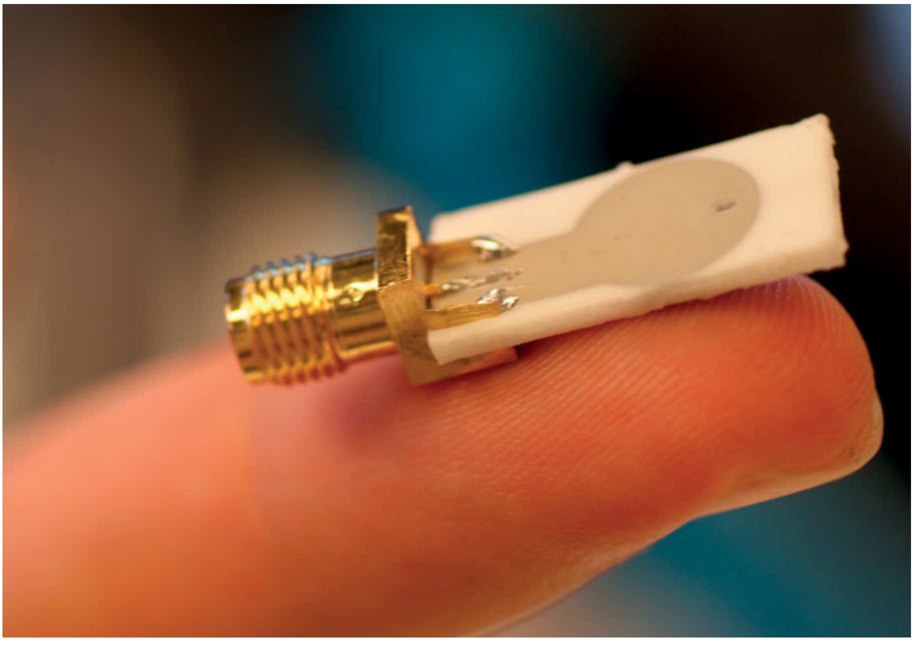

Figure 4.1. SSRR microplasma source manufactured in HTCC alumina with the author's finger for scale.

In addition to an improved plasma sample cell for OGS, the work done in this thesis provides a better understanding of the OG effect by the investigation of some of the mechanisms previously hidden in the empirical constant *K* in Eq. (3.2).

As the SSRR microplasma source starts to mature as an OGS detector, the different systems surrounding it, need to be miniaturized, integrated, and tested, to form a complete miniaturized instrument.

This far, the setup has included two or more gas lasers that are impossible or at least impractical to integrate. Hence, a tunable quantum cascade laser needs to be installed instead. Moreover, an optical cavity could be a convenient way of improving the performance of the system even further.

A rough estimate of the final system would be the size of a standard milk carton weighing less than 3 kg, where more than 2/3 of the volume is taken up by the QCL laser and a vacuum pump. The lab-on-a-chip module incorporating the SSRR detector and its supporting sensors and subsystems should not become larger than a few credit cards stacked on top of each other, and the rest of the volume is taken by electronics, optics, and thermal control. This should be compared to the SAM system, on the Curiosity rover, that is the size of a microwave oven weighing 40 kg.

In the near future the SSRR detector will be integrated with a high temperature combustor, for studies of solid samples, into an HTCC alumina lab-on-a-chip.

Integration of the fused platinum bond wires in the HTCC SSRR needs to be demonstrated. New materials for insulating the Langmuir probes will also be investigated, e.g. silicon dioxide, to avoid contamination of the chamber with carbon. An investigation into how the new probes affect the OG signal also needs to be carried out.

The artificial heating of the plasma from pushing the plasma potential with a biased probe will be further investigated, from which a more precise model of the OGE can be constructed and its limits tested. It would also be interesting to further study the mechanisms of the OGE by studying different gases and driving conditions.

All in all, this new SSRR microplasma source for molecular OGS provides a robust platform for further development and integration with new instruments for space exploration. It is not only helpful in lightening the load of future rovers but has a clear use in many ground-based applications for example in medical diagnostics, greenhouse gas monitoring, archaeology, and much more.

5. Sammanfattning på svenska

Få frågor är nog lika fantasieggande som de om utomjordiskt liv. Finns det? Hur ser det i så fall ut? Och om det inte finns, uppstår snart frågan: Varför inte?

Tack vare rymdteleskopet Hubble vet vi att universum består av åtminstone 100 miljarder galaxer. I en av dessa galaxer, vår egen, finns det uppskattningsvis 400 miljarder stjärnor. I vårt eget solsystem finns åtta planeter och 168 månar. Vore vårt solsystem och vår galax representativa för världsalltet, har vi mer än 7 000 000 000 000 000 000 000 himlakroppar att beakta. Bortsett från detta ofattbara antal och den hisnande snabba grovgallring som skulle behövas för att avfärda eller bekräfta förekomsten av liv på dessa, är det ett obestridligt faktum att inte ens en bråkdels bråkdel är tillgängliga för någon form av närmare inspektion. Vi kommer därför aldrig att kunna konstatera, varken med lättnad eller fasa, att vi är ensamma i universum. Emellertid kan vi möjligen konstatera att vi inte är det.

Ett första steg i detta arbete, är att definiera vad vi letar efter. Redan på vår egen planet finns en fantastisk mångfald av liv. Till och med den lilla underavdelningen däggdjur bjuder på stor variation, och man behöver inte ta sig särskilt många meter under havsytan för att hitta djur som lika gärna kunde höra hemma på en annan planet, eller rent av vara uppdiktade. Och då har vi nog ändå inte ägnat de märkliga extremofilerna – mikroorganismer som trotsar allt vad en dräglig tillvaro är – en tanke. I ett vidare perspektiv, finns det dock gemensamma drag att ta fasta på. Liv, som vi känner det, är nämligen kolbaserat och beroende av vatten, åtminstone i någon mån.

Sökandet efter liv på andra planeter reduceras därmed till att leta efter spår av vatten och efter förekomst av kol. Hittar man väl detta – och det kan man faktiskt göra lite på håll – är det dags att göra en närmare undersökning. Var kostnaderna och besvären stora för att konstatera att det åtminstone funnits vatten på Mars eller att det faktiskt finns vatten på till exempel en av Jupiters månar, är de gigantiska för den närmare undersökningen. Denna kräver nämligen att atmosfärs- eller markprover tas och att dessa antingen transporteras till jorden för analys eller att de analyseras på plats. Om än i andra syften, har det förstnämnda redan praktiserats när flera hundra kilo mineral har tagits hit från månen. Även så kallade *in situ*-analyser har gjorts och vi vet därför en del också om gruset på Mars, även om ingen någonsin har hållit i ett prov därifrån. Visserligen kan den apparatur som vi kan landa på någon himlakropp aldrig mäta sig med den vi har tillgång till på jorden, men det är en nöjaktig kompromiss – åtminstone i det avseendet.

Snarare består svagheten i det bristande urvalet. Med de fjärrstyrda och otympliga fordon som trafikerar till exempel Mars yta, skrapar vi både bokstavligt och bildligt talat bara på ytan, och till råga på det bara på en yta som är tillräckligt lätt och säker att rulla på. NASAs Curiosity, som väger 900 kg och är den mest välutrustade *rovern*, behöver nästan två (jord-) år bara för att korsa sin ganska släta "landningsbana". Med den farten tar det onekligen lång tid att få en representativ uppfattning om planetens sammansättning och att hitta något av de eventuella spår av liv som kan tänkas finnas på den kraftigt eroderade ytan. Man kan ganska lätt, och bara lite orättvist, föreställa sig det omvända: Någon betraktar jorden på håll och identifierar Saharaöknen som en lämplig landningsplats för ett ganska känsligt fordon, som till slut landas och pressas till genomsnittsfarten 0,14 km/h så länge terrängen tillåter. Skulle en sådan undersökning, vad jakt på liv beträffar, ens ertappa en förskrämd skalbagge?

Naturligtvis är det här inget lättlöst problem, men kunde man, utan att tumma på kvalitet och säkerhet, krympa tekniken, skulle saken underlättas. För det första är kostnaden för att skicka upp saker i rymden direkt relaterad till vikten; man får räkna med några hundra tusen kronor per kilo bara för själva uppskjutningen. För det andra skulle man kunna utnyttja ett begränsat *bagage* bättre. Alltså, om man kunde realisera nämnda Curiosity i en tiondel så tung version, skulle man antingen kunna spara åtskilliga miljoner kronor eller, bättre, kunna landsätta tiotalet små *rovrar* för att täcka planeten snabbare. Med en tillräcklig miniatyrisering av tekniken, kan man till och med tänka sig andra fordon, till exempel hoppande farkoster, som kan ta sig fram i oländigare terräng. Dessutom borde själva landsättningen underlättas.

För att krympa något en faktor tio eller mer, krävs vanligtvis något radikalt. (Dessutom krävs det att man lurar systemet, som paradoxalt nog egentligen bara tillåter "ny teknik" om den redan är "gammal", för att hårdra branschens Moment 22. Men det är en annan fråga.) *Mikrosystemtekniken* är ett exempel på något sådant radikalt. Den har sitt ursprung i halvledartekniken, som, till exempel, gör att en radio inte behöver vara en hel möbel eller att en dator, hur lågpresterande den än är, inte behöver inhysas i en maskinhall, men är så mycket rikare. Mikrosystemtekniken flyttar nämligen inte bara ettor och nollor eller hanterar elektriska signaler, utan används för att känna av magnetiska fält, reglera vätskeflöden, styra ljus, mäta luftfuktighet, manipulera celler, med mera, med mera. Typiskt handlar det om millimeterstora komponenter som färdigställs med mikrometerprecision. Ibland blir komponenterna, eller åtminstone de system de utgör, centimeterstora, och ibland tillåter processtekniken bearbetning på nanometernivå. Till skillnad från halvledartekniken, som ibland benämns mikroelektronik, är *mikrotekniken* i allmänhet ingen estradör. Det är därför betydligt lättare att till exempel peka ut platsen för lite mikroelektronik i en modern bil, än att peka ut ens en av fordonets åtminstone hundra mikrosystemtekniska komponenter.

I det här avhandlingsarbetet har det undersökts om funktionen hos ett av de sofistikerade analysinstrumenten på till exempel Curiosity skulle kunna realiseras i ett avsevärt mycket nättare format med hjälp av denna teknik. Valet föll på en spektrometer.

I en spektrometer kan man studera hur ett ämne tar upp eller avger strålning. Eftersom olika ämnen växelverkar på olika sätt med strålning, kan man få en unik signatur från varje sorts atom eller molekyl som ingår i ämnet, antingen som några diskreta bortfall eller som några diskreta tillskott i ett spektrum – en räcka av ljus- eller strålningsvåglängder. Spektrometrar är vanligtvis mycket känsliga, men också ganska skrymmande, särskilt om ljus måste separeras noggrant med linser, bländare och prismor. Någonstans mot slutet av spektrometern placeras en detektor som omvandlar strålningsintensiteten till en elektrisk signal för filtrering, lagring och tolkning. även denna del är känslig, fast i ordets negativa bemärkelse. Varje signalomvandling medför förluster och risk för förvanskning, till exempel genom yttre störningar. Slutligen, eller inledningsvis snarare, måste provet prepareras, vanligtvis förgasas, för att det ska kunna analyseras i en spektrometer.

Spektrometrar finns av många slag. Därför gällde det att först identifiera en princip som dels lät sig “förminskas” utan fundamentala hinder, dels en typ som faktiskt kan identifiera spår av liv. Dessutom handlade det om att hitta en sort som är tillräckligt robust för att verka i ett sammansatt system, som det i en *rover*. Efter några inledande analyser och försök, framstod den så kallade optogalvaniska effekten som en god kandidat. I denna nöjer man sig inte med att förgasa provet. Man joniserar det också. En joniserad gas kallas för ett plasma, och är något som vi ser i stort format i neonskyltar och brasor, och i kolossalformat som norrsken. Poängen med den optogalvaniska effekten är att vi kan bygga en spektrometer utan att behöva bry oss om vad provet sänder ut för strålning. Istället exponerar vi provet, eller plasmat, för ett väldefinierat ljus och mäter hur det reagerar elektriskt. Ett plasma är nämligen elektriskt och reagerar snabbt och kraftigt i det avseendet när dess atomer och molekyler växelverkar med det inkommande ljuset.

På detta sätt hade mycket vunnits, och arbetet kom att fokuseras på att utveckla och förstå en så kallad mikroplasmakälla kapabel att jonisera en liten mängd förgasat prov och samtidigt medge att elektriska mätningar gjordes och att en laserstråle fick passera genom. Ganska snart valdes en så kallad *split-ring resonator*, som kan liknas vid en liten antenn som böjs så mycket att den bildar en ring men att ett litet gap lämnas mellan antennens två ändar. När antennen driftsätts ungefär som en radiosändares antenn, uppstår ett elektriskt fält i gapet. Detta fält är starkt nog att slita bort några elektroner från gasens molekyler och skapa det eftersträvade plasmat i mikroformat.

En stor del av avhandlingsarbetet handlade sedan om att lära sig hur plasmakällan kunde skyddas från yttre störningar, något som löstes genom att lägga till tunna metallager, hur det gasformiga provet och ljuset kunde ledas genom plasmat, vilket löstes genom en uppsättning av hål och kanaler i godset till plasmakällan, och hur den elektriska responsen bäst skulle kunna mätas. Det sistnämnda låter sig i och för sig göras genom drivelektroniken till plasmakällan, alltså genom den signal som matas till den böjda antennen, men här var ambitionen större. Med inspiration från studier av plasman i sig, inkluderades mikroskopiska elektroder, så kallade Langmuir-prober, i plasmakällan. Med dessa kunde dels en mer oberoende mätning av plasmats respons göras, dels kunde en elektrisk spänning appliceras på elektroderna för att “tränga” plasmat och justera dess respons på exponeringen för ljus en smula.

Resultatet blev en mycket liten och konkurrenskraftig spektrometerdetektor med enastående god känslighet och precision. En preliminär mätning visar att skillnad kan göras på koldioxid som framställts från liv, som vi vanligtvis definierar det, och koldioxid som framställts på konstgjort vis, något som skiljer sig bara i precis hur tung molekylens kolatom är – vilken kolisotop det handlar om. Ödmjuk inför faktumet att mycket arbete återstår, har det likväl visats att ett mycket sofistikerat instrument för jakten på molekylära spår av liv skulle kunna ersättas med ett bråkdelen så tungt instrument, troligen utan annat än vinster i funktion och prestanda. Instrumentet har inte bara potential att minska lasten på framtida Marslandare, utan har dessutom tydliga användningsområden här på jorden, till exempel, i medicinsk diagnostik, övervakning av växthusgaser, arkeologi och mycket mer.

6. Tack

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