Fast Transient Temperature Operating Micromachined Emitter for Mid-Infrared for Optical Gas Sensing Systems

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ABSTRACT

A novel micromachined thermal emitter for fast transient temperature operation is presented. Compared to most commercial available thermal emitters, the one here presented, is able to operate in a pulsed mode. This allows the use of lock-in techniques or pyrodetectors in the data acquisition without the use of an optical chopper for light modulation. Therefore, these types of thermal emitters are very important for small filter photometers. Several spider type hotplate concepts were studied in order to find a design with excellent mechanical stability and high thermal decoupling. The thermal emitters are fabricated using silicon on insulator (SOI) technology and KOH-etching. The emitters are heated with Pt-meanders. For temperature determination an additional Pt-structure is deposited onto the hotplates. The emitters are mounted in TO-5 housings using a ceramic adhesive and gold wire bonding. The used operation temperature is 750°C. In pulsed operation it’s important to have a large modulation depth in terms of thermal radiation intensity in the needed spectral range. The maximal reachable modulation depth ranges from ambient temperature to steady state temperature. A modulation frequency of 5 Hz still allows using nearly the maximum modulation depth.

Keywords: thermal emitter, microhotplate, MEMS, filterphotometer, high temperature, thermal radiation, infrared, thermal source

1. INTRODUCTION

A rising number of process, safety and environmental applications require measurement systems for gas detection as well as for contamination monitoring of liquids. Examples for such applications are the monitoring of toxic gases and early detection of leakages. Another field is the chemical industry which needs sensor systems for process control. In these applications absorption measurements are an important detection technology which combines high sensitivity, fast response time and high reliability.

In particular infrared spectroscopy facilitates the selective and sensitive measurement of various molecules by their specific absorption. Infrared spectroscopy uses the characteristic absorption of the molecules in the mid infrared and allows the estimation of the species and its concentration. Especially by the absorption at longer wavelengths between 8 and 12 µm, the so called fingerprint region, the molecules can be measured with highest selectivity. In the last years infrared detection and measurement technologies gained more and more importance.

One key component of a spectroscopic gas measurement system is the light source. For the use in FTIR spectrometers or multi channel filter photometers a light source with a broad optical emission spectrum is required. Typically thermal emitters are used in such systems. For low-end photometers filament sources with glass bulbs can be used up to 4.5 µm wavelength. For wavelengths above 4.5 µm filaments emitter without glass housing and a reduced operation temperature are commonly in use. Due to the high thermal mass of the heated emission region, these emitters can only operate in a steady state mode.

However, the modulation of the optical emission allows techniques for noise reduction or the use of pyroelectric detectors. Mechanical modulation with a chopper is a possibility, but it has the disadvantage of moving mechanical components and at least it’s an additional component which increases the system costs. The commercial available emitters for pulsed operation use thin dielectric membranes to achieve thermal isolation of the microhotplate (the...
emitting area) from the supporting frame of the silicon chip [1, 2]. Nevertheless, the performance of this “standard” MEMS based emitters suffers from inadequate mechanical robustness and low emission of radiation in the upper part of the MIR, due to the use of a closed dielectric membrane for thermal insulation.

In contrast micromachined IR emitters carried out as a suspended membrane type (e.g. spider type) enable fast transient temperature operation, higher operation temperatures and as a consequence a better emission characteristic in the MIR. In [3, 4] SOI-based spider type hotplates for high temperature applications were investigated. Therefore, a hotplate approach based on suspended membrane type has been chosen and different suspension shapes (incl. spider type) were investigated and compared.

2. EXPERIMENTAL

2.1 Design

The basic chip size of each hotplate emitter is 3 mm edge length. Different shapes of the suspension bars – the mechanical connection between hotplate and frame – were integrated in the mask in order to investigate the thermal coupling, the heating and cooling time constants as well as the mechanical stability. Additional, the distance between hotplate and frame were varied. The hotplate sizes were kept constant at an edge length of 500 µm. The three main designs of the hotplate suspension bars are shown in figure 1.

![Figure 1: A representative choice of the basic suspension layout. (a) Shows a variant with a direct corner to corner connection with roundings in the corner of the suspension bars and an additional temperature sensor (ST42RT). Figures (b) and (c) show a concept to transform the deflection caused by thermal expansion in a rotary movement (STAL1, STAL2). Figure (d) shows a variant with shorter suspension bars (ST42R2).](image)

It is important to have a heater design with large radius of curvature in order to ensure a minimum of electrical current density in the meander curve. An increased current density can result in electromigration effects. Therefore, the heater designs were checked using FEM analysis concerning the maximum current density and two different heater designs were implemented on the mask.

For thermal investigations it is necessary to have temperature sensors on several chips. Temperature sensors were implemented on the hotplate as well as on the silicon frame. The temperature increase on the silicon frame is indicator for the quality of thermal decoupling of the hotplate.
2.2 Hotplate Fabrication Process

The thermal emitter hotplate is based on standard 4 inch SOI wafer with 15 µm Si, 1 µm SiO2 and 380 ± 15 µm Si (figure 2a). The SiO2 layer acts as an etch stop for the KOH-etch process. The bulk silicon has a (100) orientation and is p-doped with Boron. On both sides a 400 nm thick LPCVD Si3N4 layer is deposited as passivation for the further processes.

The first part of the process embraces the front sided structuring of the platinum heating elements and the platinum temperature sensors. An aluminum layer with a thickness of 280 nm is deposited and acts a sacrificial layer in the platinum structuring process. The layout of the Pt-structure is transferred in the following photolithographical process in the photoresist. The resulting photoresist structure is used for wet etching of the beneath aluminum layer. The wet etching of the aluminum yields in an undercut of appr. 1 µm. This undercut is important to create a homogeneous border area of the sputtered Pt-elements. The aluminum layer has the negative shape of the final Pt-structures. Before the deposition of the 200 nm thick platinum, a thin, 20 nm tantal layer is deposited by sputtering in order to increase the adhesion of the platinum. After the Pt-deposition, the photoresist is stripped with an aceton-propanol-cascade and the aluminum layer removed by wet etching. The cross section of the structured platinum elements is shown in figure (figure 2 b).

![Fabrication of the thermal hotplate](image)

Figure 2: Fabrication of the thermal hotplate (cross section view). (a) SOI-substrate with a thickness of 380nm. Si, 1µm SiO2 and 15µm silicon. (b) Both side deposited Si3N4 layer with Pt-heater and Pt-T-sensor elements. (c) Bottom side KOH-etch. (d) Top side structuring of the suspension bars.

For the backside definition the Si3N4-layer is photolithographical structured. The remaining photoresists acts as a negative mask for the following reactive ion etch step. After stripping of the photo resist with an aceton-propanol-cascade the wafer is prepared for the KOH-etch of the backside cavity. The bare silicon is anisotropic etched in a potassium hydroxide etch solution at a temperature of 80°C (figure 2 c). In order to protect the front side, a special wafer holder which allows to seal one side of the wafer is used. This is necessary because the potassium hydroxide etch solution reacts with the platinum structures. The KOH-etch has a much slower etch rate at the Si3N4 mask, at the (111)-surfaces in the silicon bulk material and at the SiO2 layer. This results in cavities with a shape of truncated pyramids. The
angle between the backside surface and the side walls is 54.7°. The SiO₂, which is the etch stop for the KOH-etch is finally removed by a backside reactive ion etch process. This is done uniformly without any additional passivation.

In the current state, silicon membranes with platinum structures are fabricated. Now, these membranes have to be structured. Once again this is done with an aluminum sacrificial layer process (thickness of 500 nm). The aluminum is structured using a photolithographic process and a wet etch. The aluminum hard mask protects the hotplate region with the platinum structures and the suspension bars from the dry etch process. Using reactive ion etching the 400 nm Si₃N₄ as well as the 15 µm Si between the hotplate, the rim and the suspension bars are removed. Finally, the remaining aluminum hard mask is removed by a wet etching process (figure 2 d).

The dicing of these fragile devices is done using a wafer saw. The wafer is applied with its bottom side to an adhesion foil. The top side is protected with a viscous photoresist which also fills the cavities beneath the hotplates. After an outgassing of 2 h the photoresist is baked for 15 min at a temperature of 50°C. Additional, this leads to an improved adhesion between wafer and foil. The wafer is cut along dicing marks, which were deposited together with the platinum heaters and sensors on the front side. After the dicing process, the adhesion foil with the diced chips and the photoresist is soaked in acetone. The photoresist is in solution and the chips can be removed from the adhesion foil. After a cleaning in propanol and deionized water the chips are prepared for the packaging.

The chips are glued with a ceramic adhesive on TO-5 sockets. The connection of the IR-emitter and the pins of the housing is done with gold wire bonding. Figure 3 shows a mounted hotplate emitter.

Figure 3: Micromachined IR-emitter mounted in a TO-5 housing. At the outer rim an additional Pt-temperature sensor is integrated to check the substrate temperature during operation.

2.3 Optimisations During the Fabrication Process

Quality of the Platinum Structures

The fabrication of the platinum structures was a critical process during the emitter fabrication. After removing the second aluminum sacrificial layer a change in the platinum quality was observed. Figure 4 (left) shows a microscopic picture of Pt-structures after the first run. The aluminum is not lightish and glossy anymore, but dark and soft. With SEM and EDX investigations aluminum residues were found in the platinum. There was also a blistering in the Pt-structures on the hotplate observed during operation. The into the platinum diffused aluminum has a significant lower melting point than the platinum. The overall temperature stability is not given anymore. Figure 5 (left) shows SEM pictures of the blisters in the platinum. The blistering can also yield in complete crack through the platinum structure, which yields in a breakdown of the emitter (Figure 5, right).
Figure 4: Microscope pictures of the platinum structures on the micro hotplates. (left) Platinum structures after the first run. High temperatures during the front side RIE etch caused in a bad platinum quality and aluminum contamination. (right) Pt-structures after the process optimisation.

Figure 5: SEM pictures of the platinum structures after operation. (left) The aluminium causes blustering on the heater. (right) In some cases the blusters can form a crack through the complete Pt-structure. A break down of the device is the result.

The origin of the high temperature on the hotplate during the fabrication process is the front side RIE etch. This process requires due to the layer thickness (400 nm Si₃N₄ + 15 µm Si) and the used RIE system 60 min. The ion shelling results, especially in the thermal decoupled hotplates, in a high temperature increase. The temperature increases in the deposited platinum as well as on the aluminum mask. Aluminum molecules from the mask can deposit on the growing platinum structure, too.

A solution has to be found without changing the deposition equipment. A splitting of the process into two parts with each 30 min etch time and a break of 15 min for cooling already results in an improvement. Successive continuation of this approach allows the fabrication of the platinum structures. The etching process comprises 12 RIE etch steps with a break of 15 min for cooling down. Figure 4 (right) shows the successful fabricated hotplate with excellent platinum structures.

**Dicing of the IR-emitters**

A further challenge is the decollating of the microhotplates. The standard process in the clean room is developed for bulk devices and makes use of a dicing saw. The wafer is mounted on an adhesion foil and covered with a photoresist in order to protect the devices from the water jet of the dicing saw. The photoresist covered wafer was for a drying process of 30 min at 90°C in the oven.
First, this process was directly applied to the microhotplate devices. The devices could not be removed from the adhesion foil with an acetone dip. Even after many hours only several chips could be removed from the foil. In the next step an UV-sensitive adhesion foil was tested. Here, the adhesion effect decreases with UV-illumination. The result was the same as in the test with the standard adhesion foil. It seems that the photoresist protection or the treatment of it is the key of the delocating challenge. An optimization is required.

If a photoresist with a low viscosity is used (e.g. Microchemical AZ 5214, like in the standard process), the photo resist can flow into the small cavities. During this flowing process small air bubbles are injected in the photoresist. During the drying process in the oven, the outgasing of these bubbles can yield in movement of the photoresist and the enclosed microhotplate. This can also damage the hotplates or the suspension bars.

The use of a more viscose photoresist (Mircochemicals AZ 4533) improves the handling of the dellocation process. The photoresist seals the hotplates surfaces without flowing in the cavities. There were no moving bubbles during the drying process observed. A photoresist thickness of 1µm was used. The baking temperature was reduced to 50°C and the baking time to 15 min in order to reduce the adhesion quality and to simplify the dissolution process.

An alternative and promising solution is laser cutting. The disadvantage here is the availability of a laser cutter which is able to cut silicon. Silicon has high transmission in a wide spectral band in the IR region. However, a dicing saw is a standard equipment in almost every clean room.

3. CHARACTERIZATION

The characterization of the thermal emitter is divided in electrical and optical characterization. The platinum heater is used for the determination of the operation temperature. The additional integrated platinum temperature sensor is required for tasks, where a voltage measurement is necessary to monitor the temperature signal (i.e. in a pulse mode). In that case, a small constant current has to be applied to the sensor. Both platinum structures are located on the silicon rim and the hotplate region. That means that these structures are partly at room temperature as well as at operation temperature during operation. The standard formula for platinum temperature sensors, which depends on a basis electrical resistance and the temperature coefficient, will result the medium temperature somewhere between the rim and hotplate temperature. The hotplate temperature as a function of the heating resistance is required.

The hotplate temperature in dependence of the heating resistance is calculated using finite element software COMSOL. For the platinum elements the electrical resistance and the temperature coefficient of the own material were measured and implemented in the model. For the other components material data from literature were used. The model consider heat transfer to the ambient and radiation. Figure 6 shows the calculated hotplate temperature as a function of the heater resistance for the three different hotplate layouts.

![Figure 6: Hotplate temperatures as a function of the heater resistance for the three hotplate types. The characteristics were calculated using finite element analysis.](image-url)
3.1 Electrical Characterisation

Static Investigations

The basis of the electrical characterization is the determination of the U-I-characteristics. The results are shown exemplarily one curve per design - in figure 7. Due to the inhomogeneous temperature along the heater, the U-I-characteristics is clearly nonlinear. The platinum regions at the rim are at or close to room temperature, whereby the platinum regions on the hotplate are at several hundred degree Celsius and the platinum structures on the suspension bars have a temperature distribution between rim and hotplate temperature. The electrical characteristic of the platinum heater can be assumed as a serial connection of resistors which are changing their resistance in dependence of the hotplate temperature. The hotplate temperature can be determined with the calculated resistance and the results from the finite element analysis shown in figure 6.

The U-I-characteristics of the emitters with the design ST42 and STAL2 (long suspension bars) showed a more nonlinear behavior compared to ST42R2 (short suspension bars). On the one hand, this is caused by the larger rim region where the platinum heater is kept at a lower temperature and on the other hand by the better thermal coupling of the hotplate to the rim. The better coupling leads to a lower hotplate temperature at a given operation voltage and thus to the more homogenous temperature distribution along the heater in the suspension bars and the hotplate. That means also, that the emitters with the design ST42R2 need an higher operating power to reach the same temperature.

![Figure 7: U-I-characteristics of the three different microhotplate emitters.](image)

Transient Investigations

The microhotplate emitters are designed for a direct modulation of the optical output by the modulation of the heating power. The response time of the hotplate temperature – the time span from room temperature to operation temperature in steady state condition – gives information of the modulation behavior during operation. Typically, a voltage step is applied to the heater. That implies that the heater current - and thus, the heating power - is changing during the transient process, because of the temperature dependence of the electrical resistance of the heater. The response characteristic has an exponential shape. It is very improper to determine the end value during the transient temperature measurements, because the temperature change at the end of the heating or cooling process is relative small. For a better comparison of different emitters it is typical to use the $t_{90}$-time. The $t_{90}$-time is the time span during the heating process from the static ambient temperature to 90% of the static operation temperature and the time span during the cooling process from the static operation temperature down to 10% of it, respectively.
For the $t_{90}$-time measurements hotplate devices of the type ST42R were used. These emitters have an integrated Pt-temperature sensor for the temperature determination in modulated operation. A constant current of 1.59µA was applied to the sensor and the voltage was measured with an oscilloscope during the heating and cooling process, respectively. The sensor current has to be chosen under considering that the power loss through the temperature shouldn't yield in hotplate heating of several degree Celsius. With the FEM results of the electro thermal simulation it is possible to calculate the hotplate temperature on the basis of the temperature sensor voltage.

The used heater voltage was chosen to reach a hotplate temperature of 800°C. Figure 8 shows the transient heating and cooling characteristic. During the heating process the hot plate reaches 90% of the steady state operation temperature after 39 ms. The cooling process down to 10% of the operation temperature lasts 45 ms.

The next transient characterisation determines the frequency dependence of the hotplate maximum and minimum temperature. The operation conditions - heater voltage, temperature sensor current - are the same than in the $t_{90}$-time measurement. The modulation frequency was varied from 0.1 Hz to 100 Hz and the maximum and minimum temperature per frequency was measured with the oscilloscope. Figure 9 shows the maximum and minimum temperature as a function of the modulation frequency. The emitter reaches the maximum temperature up to a frequency of 8 Hz and for frequencies over 10 Hz the maximum temperature decreases strongly. At a frequency of 30 Hz the maximum
temperature is still 600°C. The minimal temperature already increases slightly for modulation frequencies less than 1 Hz, but significant increase of the minimum temperature occurs for frequencies greater than 8 Hz. At a modulation frequency of 30 Hz the minimum temperature increases up to 200°C. The resulting temperature difference at a modulation frequency of 30 Hz is reduced to 400°C.

3.2 Optical Characterization

The spectral emission was measured using a Bruker IFS 66v/S FTIR. The measurements were performed at 600°C and 800°C. Figure 10 (left) shows the spectral emission of the microhotplate emitters. A broad emission range is observable. Compared with a black body emitter at the same temperature, the optical power is reduced and the maximum is shifted towards shorter wavelengths. The emission maxima for the 600°C curves are nearly at the same wavelength as these from the 800°C curves. A possible explanation for the enhanced thermal emission is the influence of the platinum structures on the silicon hotplate. The important information of the FTIR measurement is the appearance of strong interferences on the emission spectra. The amplitude of the interferences increases with rising temperature. The origin of the interferences are internal reflections in the microhotplate. The photons are mainly generated in the silicon hotplate and the platinum structures. Some of the photons from the silicon hotplate can partly coupling out of the silicon, the others are internal reflected. This yields in interference pattern on the emission spectra. A decreasing of the amplitude towards shorter wavelengths could be explained by the influence of the platinum structures. Probably, the thermal emission caused by the platinum structures dominates the characteristic. The influence of the Si₃N₄ layer between the platinum and silicon on the thermal emission is considered as insignificant.

For a verification of the dependence of the interference and the thickness of the silicon hotplate some hotplates were thinned uniformly from the backside with reactive ion etching. The period of the interference pattern increases with a decreasing of the hotplate thickness. These measurements confirm the assumption that the interferences are caused by the thin silicon region of the microhotplate.

Figure 10: (left) Spectral emission of three different types of the emitter. The three spectra with a lower intensity are taken from the emitters operated at 600°C and the other three from the emitters operated at 800°C. (right) Spectral emission of microhotplate emitters with different hotplate thicknesses operated at 800°C. The microhotplates were uniformly thinned from the backside with reactive ion etching. The period of the interference pattern increases with a decreasing of the hotplate thickness. These measurements confirm the assumption that the interferences are caused by the silicon region of the microhotplate.

For a verification of the dependence of the interference and the thickness of the silicon hotplate some hotplates were thinned uniformly from the backside with reactive ion etching. A significant change in the interference period is expected. The FTIR-measurements were performed using the heater resistances of the 15 µm-thick standard emitters for 600°C and 800°C. A calculation of the hotplate temperature using the FEM results is here not recommendable, because the changed hotplate thickness influences also the dependence of the heater resistance from hotplate temperature. Figure 10 (right) shows the spectra of the several thicknesses. The period of the interferences increases with a decrease in the microhotplate thickness.
4. CONCLUSION AND OUTLOOK

Novel hotplate designs for the use as modulate able thermal emitters were developed and in detail investigated. Finite element analyses were performed for each design in order to calculate the hotplate temperature as a function of the electrical resistance of the heater. These data was used for the static electrical characterization. Further, the transient characteristic were investigated. The emitter can be operated with high efficiency up to 8 Hz. For higher frequencies the effective temperature difference between minimum and maximum temperature increases strongly. The emission characteristics were investigated using a FTIR. The emitters showed a broad spectral emission with an inference pattern. Tests with different hotplate thicknesses showed a dependence to the period of the interferences.

A new run with a redesign based on different hotplate sizes and two chosen suspension layouts are the focus of the current work. The larger hotplates are interesting for the integration in filterphotometers together with multi channel detectors.

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REFERENCES