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Micro-hotplates on polyimide for sensors and actuators

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Abstract

In this communication, we present the fabrication and the characterisation of micro-heating elements made on polyimide (PI) sheets and on spin coated polyimide membranes. Different types of polyimide and heater materials were investigated to realise micro-hotplates for gas-sensing and thermal actuating applications. The effects of the type of polyimide used and of the different annealing performed on the mechanical and electrical properties of the metallic heaters were characterised. Using an optimised combination of materials and processes, flexible micro-hotplates on polyimide sheets and on polyimide membranes on silicon were realised and their thermal properties evaluated. Platinum and aluminium micro-heating elements on polyimide exhibited promising characteristics for their integration in low-power gas sensors and thermal actuators.

Keywords: Micro-heaters; Polyimide; Platinum; Anodic bonding; Gas sensors

1. Introduction

There is a need in the field of microsystems for low-cost micro-heating elements made in a polymeric technology. Compared to micro-hotplates on silicon with their membranes made of dielectric layers, their fabrication on polymers brings the advantages of simplified processing and an improved robustness and flexibility. These advantages are of interest for the integration of micro-heating elements in devices made of polymers such as in the field of micro-fluidics and for the realisation of flexible and low-cost thermal microsystems such as gas sensors, flow sensors and actuators. In the case of thermal actuators based on the thermal expansion of an actuating material, the use of low-cost robust polymeric micro-heaters would ease the integration of the thermo-expandable material and make the actuating device more compatible with micro-fluidics technology. Finally, flexible low cost gas sensors would be interesting for the integration of gas sensors in radio-frequency identification flexible tags or in textiles. Polyimide (PI) has been already used several times in different type of Microsystems [1–4]. Some work has been previously performed on the integration of heating elements on polyimide, but it was limited in terms of characterisation and gas sensing integration or concentrated on the development of hot anemometers and finger print sensors [5–8]. Indeed in the article of Aslam et al. [5], the development of micro-hotplates using polyimide for gas-sensing applications was limited to the integration of the micro-heating element on a polyimide membrane. The integration of the transducing electrodes and the compatibility of the hotplate with the gas-sensitive film were not addressed.

In this work, we have fabricated and characterised micro-heating elements made on different types of polyimide for applications in the gas-sensing and thermal actuating fields. The evaluation of the processing and the adhesion of different metals on several types of polyimide has been performed. The effect of annealing on the mechanical and electrical properties of the metallic heaters was investigated. From the results of these tests, a set of polyimides and metals have been selected for the realisation of micro-hotplates for gas-sensing and thermal actuating applications. For the gas-sensing applications (operation up to 350 °C), micro-hotplates with platinum heaters and electrodes were made on polyimide sheets and on spin coated polyimide membranes on silicon substrates. In the case of the thermal paraffin actuator (operation up to 200 °C), micro-hotplates with an aluminium heater and aluminium layer for anodic bonding of a Pyrex cavity (to store the paraffin) were made on polyimide sheets. The robustness of these micro-hotplates has been char-

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Table 1
Properties of the polyimide sheet and of the spin coated polyimide film used in this work

Properties	SiO_2	Upilex-S	PI2731
Manufacturer	Thermal	UBE Industries Ltd.	HD Microsystems
λ (W/m K)	1.4	0.28	–
CTE (ppm/°C)	0.55	12	
T_g (°C)	–	500	>350
Form	Thin film	Sheets	Liquid

acterised by ramping up the power dissipated by the heater of the device until its breakdown. Platinum and aluminium micro-heating elements exhibited promising characteristics for their integration in low-power gas sensors and thermal actuators.

2. Experimental

2.1. Design

There were several designs of micro-hotplates considered in this work. They were based on polyimide sheets (Upilex-S, T_g at 500 °C, 50 µm-thick) and on spin coated photosensitive polyimide films (PI 2731 from HD MicroSystems, $T_g > 350$ °C). Table 1 presents the properties of the PI in comparison with silicon oxide thin films. The objective was to develop polymeric hotplates made of PI to replace standard hotplates on silicon made of a thin dielectric membrane [9]. These polymeric hotplates were designed to be used in two different types of devices, in resistive type gas sensors and in thermal actuators. The resistive type gas sensors, such as metal-oxide gas sensors or chemoresistors made of a polymeric gas sensitive film, consist of a heating element passivated by an insulator layer with on top two metallic electrodes to measure the variation of the electrical properties of the films when exposed to gases. The thermal actuator developed at IMT is based on the solid to liquid phase transition of paraffin encapsulated in a cavity made in glass located on top of a heating element. A summary of the main characteristics of the devices presented in this communication is given in Table 2.

2.1.1. Hotplates made of PI films on silicon

For both types of devices, the sensor and the actuator, a micro-hotplate made of a Pt heating element suspended on a 9 µm-thick polyimide membrane (PI 2731, HD Microsystems) was processed on silicon substrates (A1Si, S1Si, see Fig. 1a). Devices with two different membrane dimensions, 1.0×1.0 mm^2 and 1.5×1.5 mm^2 were realised. The cavity in the silicon substrate thermally insulates the heating element and can be used as a chamber to store the paraffin (A1Si). Finally, a photosensitive polyimide film (PI 2731) was also added on top of the heating element of the structure (Fig. 1b) to act as an inter-dielectric layer in between the heater and electrodes to realise complete low-power gas sensor structures (S1Si).

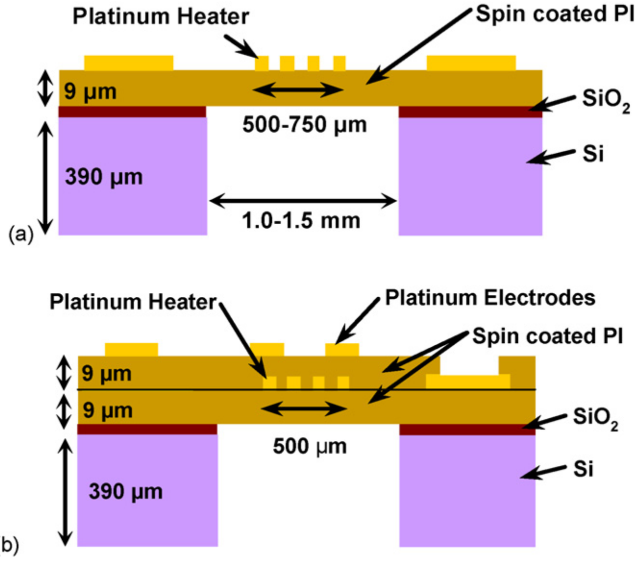

Fig. 1. (a) Design of the micro-hotplate structure with a polyimide membrane made on a silicon substrate for actuating application (A1Si), (b) with a thin spin coated polyimide film patterned on top of the heater + the platinum electrodes (S1Si).

2.1.2. Hotplates on PI sheets

There were two specific designs for the micro-heating elements realised on polyimide sheets, one for a resistive gas sensor (S2U, S3U) and the other one for the thermal actuator (A2U). Fig. 2a illustrates the design for a resistive gas sensor with the platinum electrodes and the heater patterned on the top side and on the bottom side of the 50 µm-thick polyimide sheet, respectively (S2U). This design involves simplified processing steps to realise fully flexible micro-hotplates for resistive gas sensors. However, some changes have to be brought to the standard packaging procedure of the chips on TO headers to ensure thermal insulation (by suspending the chip in air) and to be able to contact the heater on the backside of the chip. This design was fabricated but not tested due to the constraints that were just mentioned

Table 2
Summary of the main characteristics of the different hotplate designs (A = actuator, S = sensor, Si = silicon, U = polyimide sheet from Upilex)

Names	Substrate	Application	Heater area (mm^2)	Heater type	PI membrane size (mm)	Notes
A1Si	Silicon	Actuator	0.5×0.5	Pt	1.0	Heater only
			0.75×0.75		1.5	
S1Si	Silicon	Gas sensor	0.5×0.5	Pt	1.0	Heater and electrodes
			0.75×0.75		1.5	
S2U	Upilex sheet	Gas sensor	0.5×0.5	Pt	PI sheet	Electrodes opposite side of heater
S3U	Upilex sheet	Gas sensor	0.5×0.5	Pt/Cr	PI sheet	Electrodes same side as heater
A2U	Upilex sheet	Actuator	0.75×0.75	Al	PI sheet	Al rim for anodic bonding

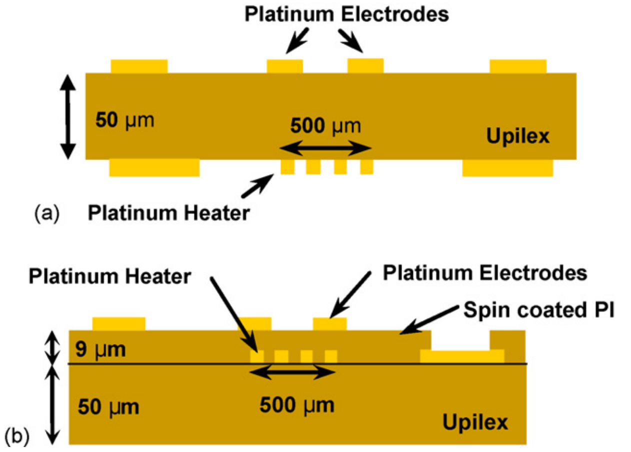

Fig. 2. (a) Design of the gas sensing structure realised using both sides of a polyimide sheet (S2U), (b) design of the gas sensing structure realised only on the top side of the polyimide sheet (S3U).

before. Another design proposed and presented in Fig. 2b consists of an Upilex sheet on which is patterned a heating element (500 × 500 µm^2) covered by a 9 µm-thick spin coating layer of photosensitive polyimide with on top platinum electrodes. This design allows having the electrical contacts, both for the heater and the electrodes, on the same side, and therefore this design was chosen to realise polyimide hotplates for resistive type gas sensor.

The micro-hotplate design for the thermal actuator (A2U) is presented in Fig. 3. An aluminium film is patterned to define the heating element (750 µm-wide) and the rim for the anodic bonding of the Pyrex cavity, used to store the paraffin, on the top side of a 50 µm-thick polyimide sheet. Metal to glass anodic bonding was the technique chosen to fix the Pyrex chip on the polyimide hotplate [10]. To achieve at the same time the anodic bonding of the Pyrex chip on the Al rim and on the two interconnections of the Al heater, an Al line linking both structures has been added to the design to electrically connect them during the anodic bonding. This line was cut afterwards to allow the independent electrical operation of the heater afterwards.

2.2. *Fabrication*

2.2.1. *Hotplates made of PI films on silicon*

Concerning the hotplates made on silicon, a 0.5 µm-thick thermal silicon oxide film was first grown on 390 µm-thick double face polished 100 mm silicon wafer. The PI 2731 used as membrane was spun over the oxide film and pre-bake on a hotplate (3 min: 65 °C + 3 min: 95 °C) and cured in an oven using the temperature ramp (up to 350 °C) suggested by the supplier. The e-beam evaporated 0.2 µm-thick Pt heater was patterned using a lift-process (AZ-1518 in acetone) and annealed at 350 °C to stabilise its microstructure. No adhesion layer was used in between the platinum and the PI film. For the thermal actuator (A1Si) the membrane was released at this point using deep reactive ion etching of silicon (DRIE, AZ-4562 as resist mask) with the oxide film acting as an etch-stop. To obtain the gas sensor structure, a second PI film was spun over the substrate, pre-baked with the same parameters as presented before, exposed to UV light and developed to open windows for the electrical contact of the heater and then cured at a temperature up to 350 °C. Electrodes were patterned by e-beam evaporation using the same lift-off process as for the heater. Finally the membrane release was performed as for the actuator using DRIE of silicon. Pictures of the hotplate structures realised on silicon (S1Si) are presented in Fig. 4. The polyimide membrane could also be used in itself as an etch-stop, nevertheless an oxide layer was used to avoid the plasma to be in contact with the polyimide to avoid possible contamination of the DRIE reactor.

2.2.2. *Hotplates on PI sheets*

The processing was simple for the devices realised on polyimide sheets. Fifty micrometer-thick wafers with a diameter of 100 mm were cut in a sheet of Upilex-S. The PI wafers were handled as silicon wafers on the spinner, in the UV mask aligner, being compatible with the vacuum systems, and in the chemical baths. For the deposition of metals by e-beam evaporation, the PI wafers were fixed on dummy substrates.

The aluminium and platinum e-beam evaporated heaters ($\sim$0.2 µm-thick) were patterned using wet chemical etching (Al etch solution) and lift-off (AZ-1518 in acetone), respectively. The platinum film was deposited on top of thin chromium film (15 nm) used to improve the adhesion to the polyimide sheet. Cr improved the adhesion of the film but induced a significant variation of the heater resistance during the thermal steps involved in some of the processing steps. Annealing of the substrates was performed to stabilise the microstructure of the metallic films to be used as heating elements.

In the case of the aluminium heaters (A2U, Fig. 3), the structures were annealed at 200 °C, corresponding to the maximal temperature of operation, during 30 min in air. Fig. 5a presents

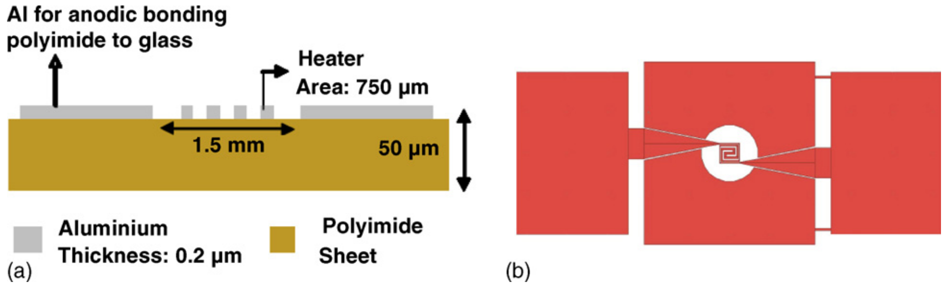

Fig. 3. Design of the micro-heating element made of aluminium realised on a polyimide sheet for a thermal actuator (a) cross-section view (b) top view (A2U).

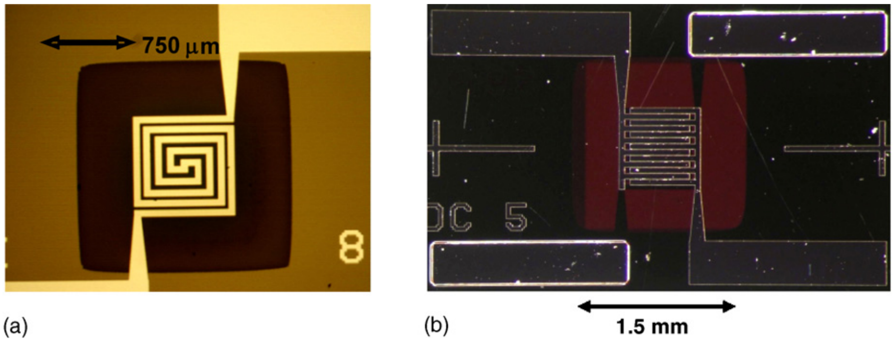

Fig. 4. (a) Micro-hotplate structure with a platinum heater on a polyimide membrane made on a silicon substrate (A1Si), (b) with the added spun PI and the Pt electrodes (S1Si).

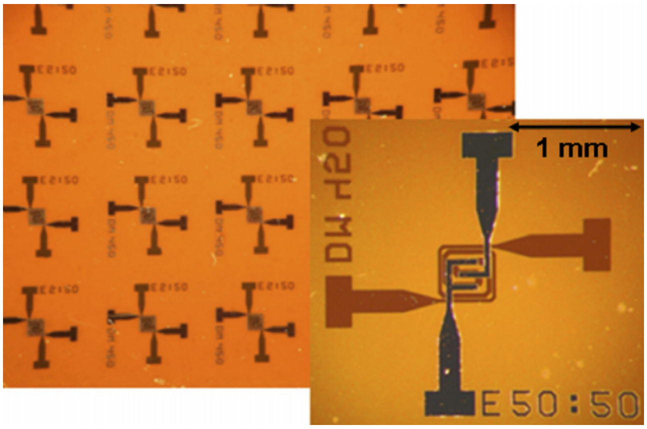

Fig. 5. Micro-hotplate structure on a polyimide sheet for gas sensing applications with the Pt electrodes patterned on one side of the PI sheet and the heater patterned on the other side (S2U).

the picture of a micro-hotplate with aluminium heater and rim fabricated on PI sheets to be used in a thermal actuator. When integrating this micro-heating in the thermal actuating device, an anodic bonding was performed in between the aluminium layer structured on the polymide sheet ($4 \times 12\,\mathrm{mm}^2$) and a Pyrex chip ($2 \times 3\,\mathrm{mm}^2$) with a micromachined cavity to store the paraffin (Fig. 5b). The anodic bonding was carried out at a temperature of 320 °C and with a voltage of 1 kV between the Pyrex and the aluminium layer on the PI sheet.

Concerning the gas sensor structure, the annealing was performed in an oven when there was no subsequent PI film spin coated. This was the case for the simplified micro-hotplate designs presented in Fig. 2a (S2U), annealing of the wafer was performed in an oven at 200 °C during 30 min in air. Photos of the structure are presented in Fig. 6. When the spin coated PI (9 μm-thick) was applied over the heater/PI sheet to realise the complete gas sensor structure as presented in Fig. 2b (S3U), the platinum was annealed during the curing of the second PI layer. In that case, before curing, the polymer was exposed to UV light and developed to pattern windows to access the heater contact pads. The curing of the PI involved temperature ramps from room temperature to a plateau of 200 °C and then from 200 °C to a plateau at 375 °C instead of 350 °C, to improve PI robustness at high temperature according to manufacturer recommendations. Platinum electrodes were then patterned on top of the spin coated PI by lift-off using the same process as described for the hotplates on silicon. Fig. 7 shows a gas sensor chip (S3U) and the complete wafer after the completion of the process.

3. Results

3.1. Heater resistance

The Pt heater resistance of the micro-hotplates made of a spin coated PI membrane on a silicon substrate varied upon the design of the device, 1.0 or 1.5 mm wide membrane and coated or not with an extra spin coated PI. The heater values for the Pt heater of the complete gas sensing structure made of a PI membrane on Si (S1Si), which was stabilised by the curing of the spun PI film, were of 90 and of 100 Ω, respectively, for the 500 μm and 750 μm wide heater designs.

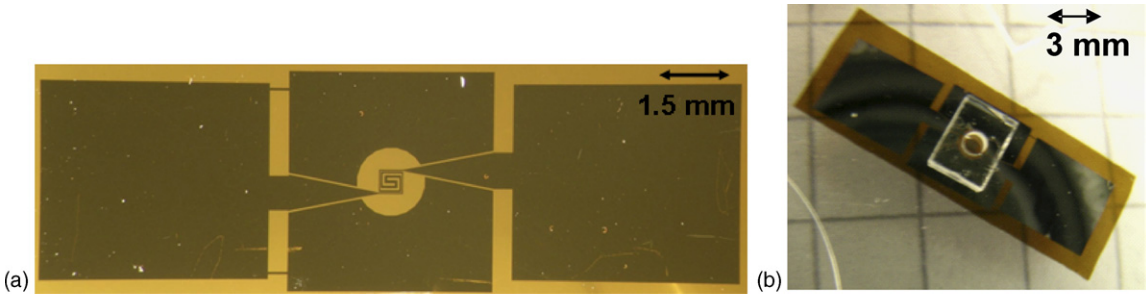

Fig. 6. (a) Micro-heating element (750 μm wide) made of aluminium on a polyimide sheet. (b) The same type of micro-hotplates anodically to a Pyrex chip (A2U).

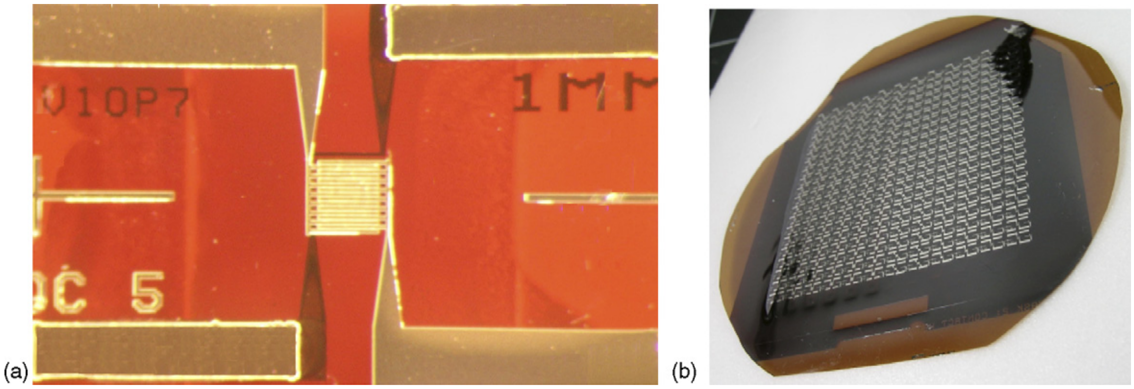

Fig. 7. (a) Micro-hotplate with a platinum heater + spun PI + Pt electrodes on a polyimide sheet, with a 500 μm wide heating area (b) picture of the processed polyimide wafer cut in a polyimide sheet (S3U).

After being patterned on the polyimide sheets and annealed at 200 °C in air for 30 min, the electrical resistance of the aluminium heater was of 27 Ω for the thermal actuator design (A2U). Concerning the gas sensing structures made on polyimide sheets (S3U), the Pt/Cr heaters (measured to be 0.15 μm-thick) covered with a spin coated PI film had a resistance of 190 Ω. The use of Cr improved the Pt adhesion during the wire bonding of the contact pads.

3.2. *Temperature as a function of temperature*

Temperature measurements were performed using a micro-thermocouple with an estimated error of 2% on the measured temperature (Type S: Pt/Pt-10% Rh with a diameter of 1.3 μm) [11]. The temperature as a function of the dissipated electrical power in the heater for the polyimide hotplates made on silicon is presented in Fig. 8. The polyimide micro-hotplates on silicon for the thermal actuator (A1Si, without a 2nd film of spin coated PI on top of the heater) required less power to reach a given temperature. With the 1.5 mm membrane structure, a power of 40 mW was needed to reach the 200 °C required for the thermal actuating application. The complete gas sensing structures (S1Si) with the same membrane size reached 200 °C at a power of 45 mW and 325 °C at a power of 80 mW. Decreasing the size of the membrane from 1.5 to 1.0 mm and respectively the heater width from 750 to 500 μm reduced the power to a value of 60 mW to reach a temperature of 325 °C.

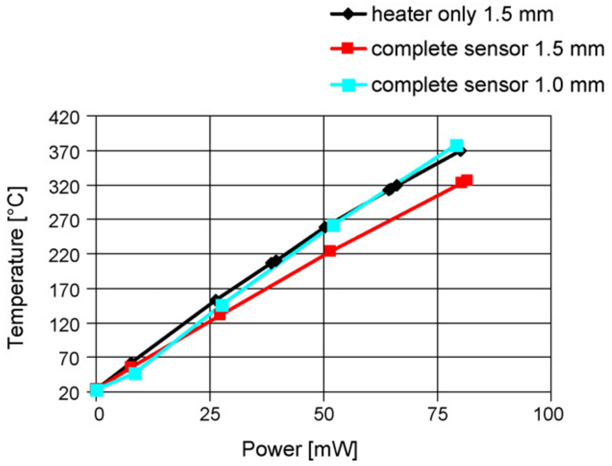

Fig. 8. Temperature at the centre of the membrane as a function of the input power for Pt heaters on polyimide membrane spin coated on silicon; heater only (A1Si), complete sensor (S1Si).

As shown in Fig. 9, the complete gas sensor structure realised on a polyimide sheet (S3U) consumes more power than the polyimide hotplates on silicon to reach a given temperature, for instance 110 mW is needed to reach 325 °C. On suspended Upilex sheets with Al heaters (A2U, 750 μm wide heating area), the maximum temperature of operation of 200 °C required for the thermal actuator was reached at about 130 mW.

3.3. *Effect of a post-annealing on the gas sensor structures*

To look at the thermal stability of the device and to be able to realise complete metal-oxide gas sensors using these hotplate technologies, annealing tests were performed at different temperatures on the PI gas sensing structures made on silicon substrates and on polyimide sheets. Fig. 10 illustrates the effect of an annealing at 450 °C, 10 min in air, on PI hotplates on silicon. One can notice the plastic deformation of the PI membrane. Deformation of the membrane also occurred for the same type of

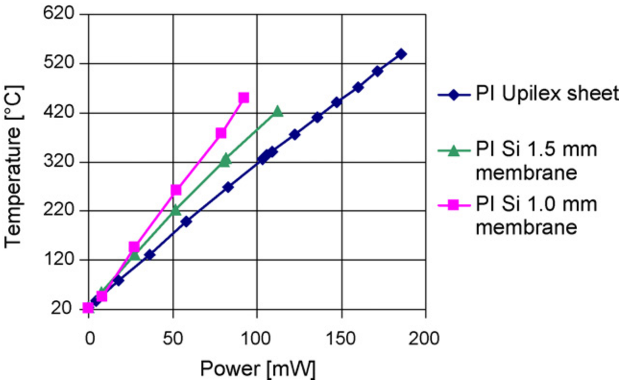

Fig. 9. Temperature at the centre of the membrane as a function of the input power for polyimide micro-hotplates with platinum heater and electrodes made of a spin coated polyimide membrane on silicon (S1Si) and made on a polyimide sheet (S3U).

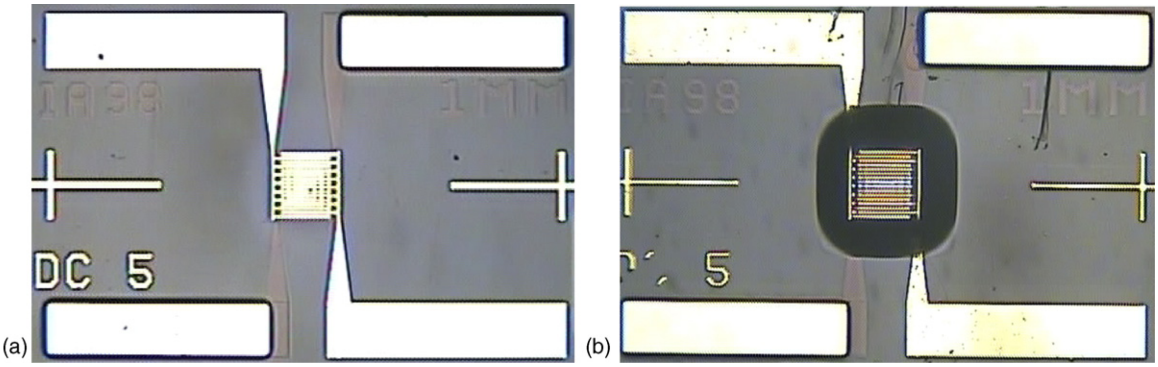

Fig. 10. Photos of polyimide micro-hotplates with a platinum heater made of a spin coated polyimide membrane (1 mm wide) on silicon (S1Si) (a) after processing (b) after annealing at 450 °C, 10 min in air showing the membrane deformation.

annealing performed at 400 °C. The maximum annealing temperature to keep the mechanical stability of this type of device corresponds to the curing temperature of the polyimide (350 °C). The different annealing performed did change slightly the heater resistance, with a decrease of the heater value between 1 and 5%.

The polyimide micro-hotplates on Upilex withstood an annealing at 450 °C, 10 min in air without any modifications observed under an optical microscope. However, the heater resistance increased by a factor of about 15% due to the instability of the Pt and Cr interface. At 400 °C, the resistance increased by a smaller factor of 5%. At an annealing temperature of 500 °C, high stress occurred in the structures and the PI substrate bent over. This was probably caused by the spin coated PI layer being not stable at this temperature when coated on the Upilex sheet. The use of the Upilex sheet (thicker than the spin coated PI film and higher T_g) improved the mechanical stability of the devices compared to the hotplates with membrane made only of PI spin coated films on silicon substrate.

3.4. Maximum power leading to breakdown

By ramping up the device voltage by 50 mV every 100 ms until rupture, a maximum power leading to breakdown was measured. The experiment was performed two to three times per type of device, before and after annealing. It was observed that this maximum power value is significantly higher than the power required for the operation of the devices in the applications targeted in this paper. The maximum power values recorded correspond to temperatures higher than the glass transition temperature of the two different polyimide materials used.

Concerning the Pt heater sitting on suspended spin coated polyimide membrane on silicon (A1Si), the breakdown occurred at a power in of 155 and of 222 mW, respectively for the 1.0 and 1.5 mm PI membranes. The annealing of these devices, as described in the previous section, had a little influence on the maximum power values obtained for these devices, modifying them just by few milliwatts. After processing, the complete gas sensing structures (S1Si) integrated on PI membranes on Si, 1.0 and 1.5 mm-wide, exhibited a higher maximum power of 163 mW and of 229 mW. Annealing of these micro-hotplates at 400 or 450 °C increased their maximum power values by 30 to 45 mW, improving their robustness in that type.

For the gas sensor structures made on the PI sheets (S3U), due to their smaller heating area and therefore to a higher temperature of operation for a given power, the maximum power to breakdown was higher at 308 mW. On suspended Upilex sheets with Al heaters (A2U), the maximum temperature breakdown power was of 213 mW. The annealing did not have an influence

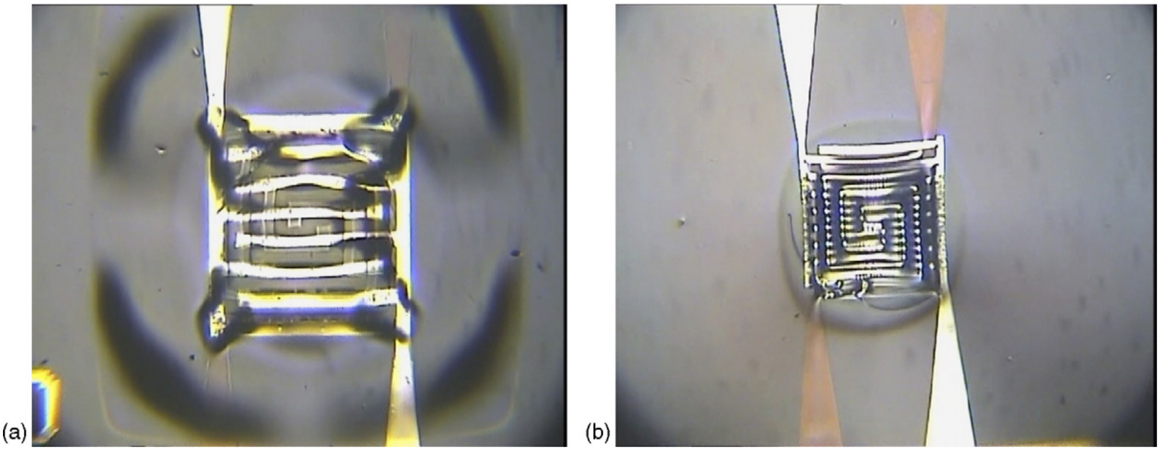

Fig. 11. (a) Image of the Pt heater on a 1.5 mm spin coated polyimide membrane on Si (S1Si) when it broke down at 270 mW (b) image of the Pt heater on polyimide sheet (S3U) when it broke down at 275 mW, the test was performed with the device suspended in the air to improve the thermal insulation of the device when heated.

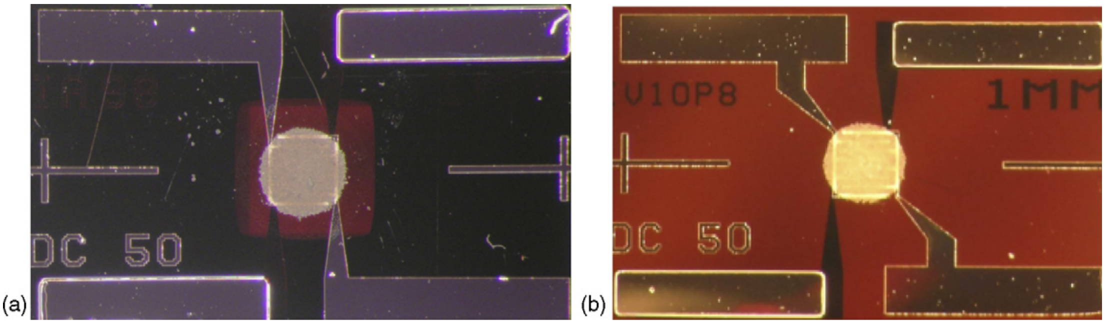

Fig. 12. Polyimide hotplates (a) on silicon (S1Si) and (b) on a polyimide sheet (S3U, 500 μm wide active area) coated with a drop of SnO_2 annealed at 450 °C, 10 min in air.

on the maximum power values leading to the breakdown of the micro-hotplates made on Upilex PI sheets.

Fig. 11 presents pictures of these devices after breakdown, showing that there is a PI film deformation leading to a degradation of the platinum films on both types of devices, on Si substrates and on PI sheets. The main failure mechanism was related to the breakdown of the heating element due to the mechanical deformation of the membrane and to the higher mobility of the polymeric chains at temperatures closed to the glass-transition temperature of the polyimide film. More extended and systematic experiments and failure analysis are needed to define the breakdown mechanisms and to evaluate the long-term stability of these devices under operation.

3.5. Towards applications

Complete gas-sensing structures were drop coated with SnO_2 and annealed in air at 450 °C during 10 min (Fig. 12). Packaging of the devices to perform gas measurements is under progress. Upilex sheets with aluminium heaters had their bonded Pyrex cavity successfully filled with paraffin and actuators with large deformations (>100 μm) were realised. The heater has been proven to work out for operating temperatures not more than 200 °C in the case of this application. The design, fabrication and characterisation of these thermal actuators will be presented elsewhere. For applications for which higher dissipated power is required, the heating material would have to be changed from aluminium to platinum.

4. Conclusion

Platinum and aluminium micro-heating elements on polyimide exhibit promising characteristics for their integration in low-power gas sensors and thermal actuators. Relatively high temperature can be reached with low-power consumption. The robustness of these micro-hotplate has been evaluated. The main failure mechanism of non post-annealed devices was related to the breakdown of the heating element due to the mechanical deformation of the membrane and to the higher mobility of the polymeric chains at temperatures closed to the glass-transition temperature of the polyimide film. From the experiments performed, the hotplates made on a polyimide sheet (Upilex) were the most robust and the most suitable for our applications. The micro-hotplate made of aluminium has been successfully integrated in a thermal actuator operating at a relatively low temperature (100–200 °C). In the case of the gas sensing structures on PI sheets, a high operating temperature was obtained at a relatively low-power and the thermal stability of the structure allowed the annealing of a metal-oxide film to realise gas sensors. Work is in progress to package properly the devices and to characterise their gas sensing performances.

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Biographies

Philippe Dubois graduated in electrical engineering from the Neuchâtel University of applied science, Switzerland, in 1991, and he received an electronics/physics diploma from the University of Neuchâtel, in 1998. In 2003, he obtained his Ph.D. on micromachined active valves and tribological studies in the group of professor de Rooij, from the Institute of Microtechnology, University of Neuchâtel. He is finishing a post-doctoral work on liquid valves, directional acceleration sensors, and polymer based microactuators. Currently in the group of professor Shea from the EPFL, he leads researches on microfabricated active polymers for space applications.

Anupama Gangadharaiah currently pursuing her Masters in Micro and Nanotechonolgy at the Institute of Microtechnology, University of Neuchâtel, Switzerland, received her B.Eng. degree from M.S. Ramaiaha Institute of Technology, Bangalore, India. Her research interests include Micromachining and Nanofabrication.

Sylvain Colin obtained a Diplôme d'Etude Approfondie (MSc.) in Optronics from University of Rennes 1, France in 1996. He worked in the optical field with Blue Sky Research Inc. and Intel Corp. Optical Platform Division from 1997 to 2003. He is currently a student in the MSc. Micro and Nanotechnology, from Institute of Microtechnology, University of Neuchâtel, Switzerland.

Emir Vela received his MSc. degree in microengineering with emphasis in micro and nanosystems from the Swiss Federal Institute of Technology in Lausanne, Switzerland, in 2005; his Master thesis was realized at the Institute of Microtechnology of the University of Neuchâtel, Switzerland.

Danick Briand received his B.Eng. degree and M.A.Sc. degree in engineering physics from École Polytechnique in Montréal, in collaboration with the Laboratoire des Matériaux et du Génie Physique (INPG) in Grenoble, France in 1995 and 1997, respectively. He obtained his Ph.D. degree in the field of micro-chemical systems from the Institute of Microtechnology, University of Neuchâtel, Switzerland in 2001, where he is currently a project leader. He is in charge of European and industrial projects and of the supervision of doctoral students. His research interests in the field of microsystems include PowerMEMS, polymeric MEMS, the integration of nanostructures on microsystems, and the development of micro-analytical instruments for gas-sensing applications.

Nicolaas F. de Rooij received a Ph.D. degree from Twente University of Technology, The Netherlands, in 1978. From 1978 to 1982, he worked at the Research and Development Department of Cordis Europa N.V., The Netherlands. In 1982, he joined the Institute of Microtechnology of the University of Neuchâtel, Switzerland (IMT UNI-NE), as professor and head of the Sensors, Actuators and Microsystems Laboratory. Since October 1990 till October 1996, he was acting as director of the IMT UNI-NE. Since 1987, he has been a lecturer at the Swiss Federal Institute of Technology, Zurich (ETHZ), and since 1989, he has also been a professor at the Swiss Federal Institute of Technology, Lausanne (EPFL). His research activities include microfabricated sensors, actuators and microsystems.