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Design And Fabrication Of Platinum-Based Mems Microheater With Integrated Temperature Sensor For The Thermal Analysis Of Greenhouse Gases

Shwetha H R[1] , Rudra swamy S B[2]

[1]Department of Electronics and Communication Engineering, J.N.N College of Engineering, Shivamogga, India-577204.

[2]Department of Electronics and communication Engineering, Sri jayachamarajendra college of Engineering, Mysuru, India-570006.

Abstract

Micro electromechanical system (MEMS) microheater plays a vital role in in semiconductor based chemical sensors. In this study a platinum-based MEMS microheater is designed using CleWin 4.1mask designing tool and fabricated using surface micro machining technique. To analyze the thermal activities, thedesigned platinum microheater is integrated with temperature sensor. The thermal analysis such as of rise time and fall time of heater is performed on the selected greenhouse gases CO_2 and CH_4using a novel research equipment Sensimer. The designed microheater along with temperature sensor successfully responded to the above-mentioned gases with different temperature with less power consumption. From the Experimental results we can infer that, that power consumption and average pulsating time constant for $CO_2$and CH_4 are 2.8mW&6ms and 3.6mW & 5ms respectively, for the temperature variation from 30°C to 400°C in the fabricated microheater. The thermal analysis of above-mention edgreen housegases causing elements to have been presented in this paper.

Introduction

The upsurge in the establishment of industries has undeniably led to the economic growth of the different nations but has however also led to a rapid escalation in emission of toxic gases and other hazardous environmental pollutants. Hence, accurate monitoring of toxic gases and detection of environmental pollutants has become an inevitable concern during the recent years. In this regard, designing of robust, low cost and portable sensors is an essential requirement to develop new range of chemical sensors with enhanced sensitivity.

Sensor is the device that converts the physical phenomenon into readable form, sensors have different types viz., mechanical, electrical, optical and magnetic forms. They are classified based the type of output they generate after converting the physical phenomenon. some sensors are classified based on the type of input they sense to convert to another form they are strain gauge, chemical sensors, biological sensors etc. In this paper we have focused on the development MEMS based chemical sensor.

There are various MEMS based chemical sensors are widely used in detection of hazardous gases and each operates with different criteria like capacitive, thermal, resistive, etc., These sensors are fabricated using integrated circuit (IC) batch processing techniques and can range in size from a few micrometres to millimetres. These sensors have the ability to sense, control and actuate on the micro scale, and produce the effects on the macro scale. Gas detection sensors are developed to ensure the level of various harmful gases within in an acceptable range. A chemical sensor consists of a transducer and an active layer to convert the chemical information into electrical signal like frequency change, current change or voltage change. There are different gas sensor technologies are used to detect the toxic gases namely, catalytic sensors, thermal conductivity gas sensor, electrochemical gas sensors, optical gas sensors, infrared gas sensor, semiconductor metal oxide sensor and acoustic wave gas sensors [1]. Among these technology metal oxide semiconductors playsa vital role towards detection of concentration of target gases. In this paper we have focused on the development MEMS chemical sensor based on metal oxide semiconductor technique. The basic Structure MOS Sensor is illustrated in the Fig. 1. Structurally, a MOS sensor comprises the following key layers.

- Substrate,
- Insulating platform,
- Micro-heater,
- Integrated electrodes, and

Sensing layer

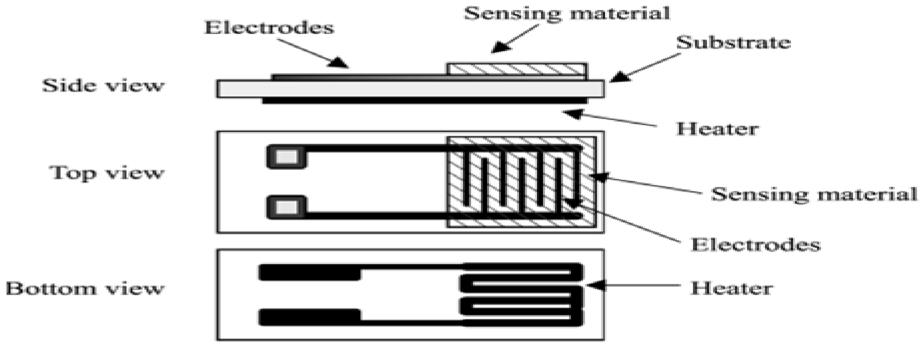

Figure 1 :Schematic structure of MOS sensor

Substrate is used as bottom layer which provide base to the heater. The most popular substrate is silicon because it has uniform mechanical properties. Insulating platform is the layer present between the substrate and micro-heater. To avoid direct damage to the substrate as well as to reduce the heat loss insulating platform is used. In order to detect the target gas effectively, micro heaters are used in the metal oxide gas sensors. Heating of the micro heater is carried on basis of joule of heating. The micro heaters are applied in humidity sensors, pressure sensors and gas sensors. Integrated electrodes are placed under the sensing layer and used to identify the variations which occur in resistance of the metal oxide sensing surface when it responds to gases. Integrated is nothing but the digit like pattern of electrodes which used in resistance measurement when the gas is absorbed on the layer. Conductivity of the material is changed when the sensor is reached with the atmospheric chemicals. Integrated chemical sensors are very popular because it is cost efficient. Metal oxide sensing layers can use many materials depending upon the gas to be sensed and have good sensitivity towards hazardous gases. The conductivity of this layer varies according to the gas concentration present around.

In metal oxide sensors, oxidation or reduction process that takes place on the sensing layer occurs at a specific temperature. Oxidation or reduction decides the number of free electrons on the layer which either increases or decreases the conductivity of the layer. Micro heater plays a significant role in achieving the adequate temperature for the reaction. The parameters like uniform heating, low power consumption and mechanical stability are necessary to consider in the design of micro heater.

Literatures reveal that in Micro heater designing it is important to consider the characteristics like heat transfer, geometry and thermal response time, which could not be addressed in their study. As stated, functionally, Micro heaters transfer the heat in three modes such as conduction, convention and radiation. At temperature less than ~700°C the conduction mode and convention mode are significant, while radiation is insignificant for Ti or Pt micro heaters [2] and therefore the selection of materials and temperature sensitiveness is of vital significance. In [3], authors applied numerical simulation to select material by calculating maximum temperature and power savings using different insulating layers. With similar effort authors [4] focused on enhancing the geometries so as to achieve power savings, reduce- stress profiles and efficient heat distribution. Emphasizing on operation of micro heater, especially to enhance the thermal response time authors [5] proposed tungsten micro heaters that enabled achieving thermal response time up to 2 ms at the temperature of 600°C. It was found exhibiting the power consumption of 12mW. Author [6] applied Pt/Ti as material to design Micro heater so as to achieve thermal response time of 1ms with temperature of 400°C using only 9mW of power, but they are not optimized for even temperature distribution. In practice even MEMS packaging material too has impact on overall sensing efficiency. Considering this fact, authors [7] selected packaging materials for a MEMS device in such manner that it could withstand the operating condition such as high temperature operation, high pressure, chemical resistance, mechanical and thermal shock and vibration. As solution, the most commonly used materials for micro heater packaging are metals, ceramics, silicon and plastics. Authors [8] concluded that Metals are good for their robustness ease of assembly, mechanical integrity and chemical inertness in harsh environments and ceramics are good for their material properties like electrically insulating, hermetic sealing, thermal conductivity and chemical inertness and to the ease in shaping. However, very few researches have been reported on micro-heater packaging. Considering structural optimization, the fact that the Micro heater is a small resistance heater and it operates by passing an electric current across a filament to generate heat, is needed to be considered. Since the response time of the micro heater is very fast, a sophisticated feedback system is used to control the temperature. The authors [9] applied Proportional-integral derivative (PID) controls in order to control the temperature of the micro heater. Similarly, feedback control for a micro heater in which the resistance of a conductor changes with its temperature; thus, the average temperature of some conductor may be determined through its change in resistance [10]. The temperature of the micro heater is usually read through the resistance change of an additional metal filament which is near the micro heater, rather than by reading the resistance of the micro heater [11]. Different researchers used different shapes of microheaters Table 1 depicts the survey of different researchers microheaters geometry and the temperature achieved for power consumed [12].

Table 1: Literature Survey of Microheaters geometry and their power consumption [13]

Geometry	Length (mm)	Average temperature achieved (K)	Power consumption in (milli wats)
Double spiral	522.9	375.65	2.2
Meander	322.7	330.9	2.9
S shape	224.9	380.7	3.6
Fan	287.9	372.7	2.7
Hilbert order 3	224.52	376.21	3.6
Hilbert order 4	455.06	360.78	3.9
Moore order 3	224.52	376.66	2.5
Moore order 4	455.06	361.66	2.0
Peano order 2	273.09	379.57	3.8
Peano order 3	927.7	317.38	2.9

As already stated, the factors like substrate materials, filament material, Micro-heater material and design, etc has always the impact on overall sensitivity or performance. Under such circumstances, identifying an optimal material set and design constructs can be of paramount significance for MEMS based gas sensor and flow rate analyser to be used for industrial or varied purposes. With this motive this paper mainly focusses on the thermal analysis of greenhouse causing gases CO_2 and $CH_4$with novel microheater structure called Labyrinth which achieves better temperature compared to previous microheater shapes for the thermal analysis of $CO_2$and CH_4 .

Design and fabrication of microheater

The microheater is designed in Cle Win designing tool and fabricated using surface micromachining technique. The schematic geometrical details of the designed microheater are shown in the Figure 3. The designed microheater is integrated with temperature sensor. It consists of two suspended platinum coils H and H^1 and RTD (temperature sensor) R and R^1. As shown in the Figure 4. The microheater die surface area is 2.5mm *2.5mm, the membrane size is 100*100µm. The fabrication of simulated MEMS microheater structure was carried out using surface micromachining technique. In this method, a covered oxide layer is used to discharge the body of the cantilever for ultra violet (UV) photolithography. The silicon substrate is cleaned using RCA1 (Radio Corporation of America standard1) and RCA2 (Radio Corporation of America standard2) protocols. The wafer was treated with RCA1 solution at 75°C for 15 min. The RCA1 and RCA2 cleaning protocol solution ratio is taken as H_2O: NH_4OH: H_2O_2 :: 10: 2:2, and H_2O: HCl: H_2O_2: 12: 2: 2, for HF (Hydro fluoric acid) HF: H_2O :: 3: 12: 0 respectively for RCA1 and RCA2 protocols. The silicon wafer is washed with distilled water after treating with RCA1 solution then the wafer is again treated with RCA2 solution to remove any contaminations on the wafer surface. Then the wafer is placed on hot plate having a temperature of 270°C to remove any residual vapours on the wafer. Then the thin films of analytes are coated on the wafer using spin coating technique. Then, the soft baking is carried out by heating the substrate in a furnace up to 120ºCfor 30 min to improve the adhesion quality of wafer. To produce pattern images, the substrate is exposed to UV radiation of 360nm with radiation energy of 40mJ/cm^2. Now, the fabricated sensor is developed using tetra methyl ammonium hydroxide (TMAH MF26A) for 45 seconds and dried with nitrogen gas. To create a physical mask that covers the wafer to protect from chemical contamination during etching the substrate is kept in hotplate for 5 min and heated at 100ºC. After soft baking, the hard baking process is carried out to increase the adhesiveness on the wafer surface. Now, the cantilever is treated with UV photo lithography to remove the photo resist on waferusing dry etching technique. For this, the wafer is then mounted on Inductively Coupled Plasma Receptive Particle Scratching (ICPRIE) instrument. In ICPRIE, aluminium nitrate base is connected for legitimate warm vitality exchange between bearer wafer and cantilever substrates, it permits effective anisotropic etching of substrate according to Bosch process. The gases viz., sulphur hexafluoride (SF_6) and octaflurocyclobutane (C_4F_8) is used for anisotropic carving. The details of the parameters used in ICPRIE anisotropic etching is shown in table 1 .

Table 1: Parameters used in anisotropic etching

Parameters	Quantity
Mask Type	AZ5214
Sulphur hexafluoride (SF_6)	30 SCCM
Octaflurocyclobutane (C_4F_8)	70SCCM
Inductively Coupled Plasma (ICP) power	900 W
Radio Frequency (RF) Power	120 W
Etch time	15 sec
Chamber pressure	15 mbar
Process temperature	15 °C

After dry etching, the residue of photo resist is burned by the ICPRIE instrument. To confirm the removal of unwanted Silicon, optical microscopic inspection was carried out, where the change in colour of substrate is observed.

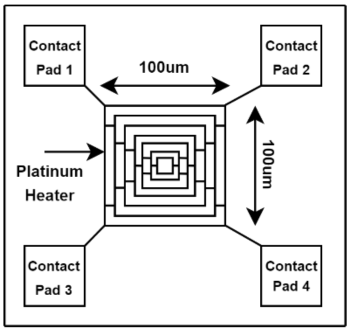

Figure 2: Schematic of proposed labyrinth designmicroheater

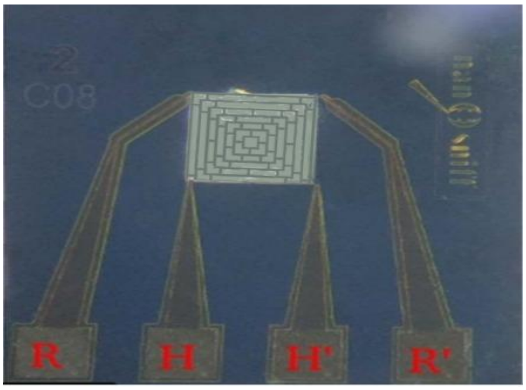

Figure 3: SEM image of designed microheater

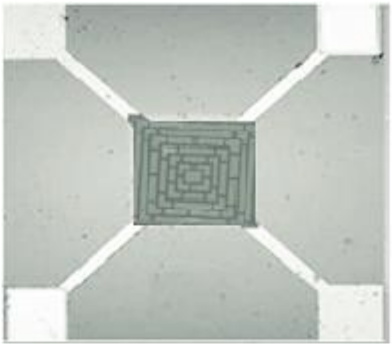

Figure 3: MEMS microheater design in Cle Win software

The microheater design in Cle Win software is shown in figure 3. The developed sensor as the die after the fabrication is shown in the figure 4.

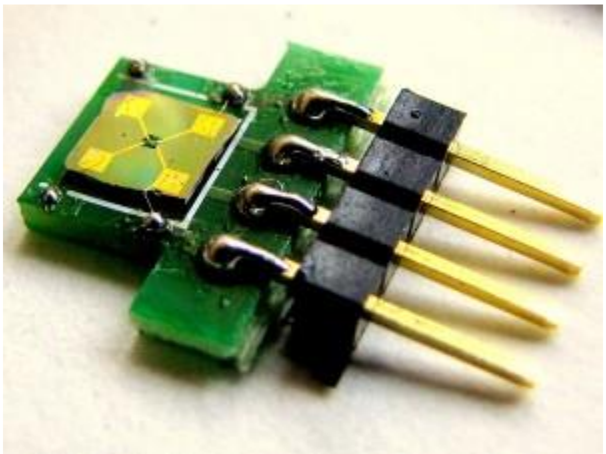

Figure 4 developed microheater die set after fabrication with four pins R, R^1 H, H^1

Experimentation in Sensimer

The developed microheater is tested in Sensimer microheater tester. Sensimer is the microheater testing tool which will helps to perform thermal analysis of testing material in power or liquid form. The developed microheater has four pins as shown in the Figure 4. The sinusoidal voltage input is given to the sensitive electrodes inside the sensimer the electrodes will elevate and reduce the microheater temperature suddenly with in the milliseconds in pulsating form. the pulse rises time of temperature and fall time of temperature is measured for all the tested materials. The rise time and fall time of the temperature response for all the materials is unique due to different coefficient of temperature for all the materials.in the experimentation the rise time and fall time of the temperature vs time is analyzed for all the selected greenhouse gases and the results are reported in the results and discussion section. The device sensimer which is used for the research is shown in the Figure 5.

Figure 5: Sensimer microheater testing machine

Sensimer is desktop experimentation device to control the temperature of microheater to analyses the thermal response. It consists of microheater plug in port with associated electronics, software to define electrical excitation. Thermal mass response of microheater for material is seen as the graph of temperature v/s time.

Results and discussion

In the current research work the selected greenhouse gases CO_2 and $CH_4$are tested in microheater in the powder form. The thermal response of CO_2 and $CH_4$is analyzed individually and then the raise time and fall time of both thermal mases has been compared to study the temperature sensitivity of selected greenhouse gases. The thermal response of CO_2 is recorded as temperature v/s time graph which is shown in Figure 6. The thermal response of CH_4 is shown in Figure 7. From the graph shown in Figure 6 we can infer that the rise time of CO_2 is 10ms and the fall time is 6ms. From the graph shown in Figure 7, we can infer that, rise time of CH_4 is 5ms and fall time is 6ms. The thermal response comparison graph is shown in the Figure 8. The demonstration image of microheater pulsating heat for sinusoidal voltage input is shown in the Figure 9.The summary of the results is depicted in Table 2. We can infer that power consumption of microheater to reach the maximum temperature for CH_4 is more compared to CO_2. The power is calculated based on the product of supply voltage and constant current of the sensimer machine.

Table 2: Rise time, fall time and power Consumed by the microheater for labyrinth design

Sl. No.	Material	Voltage Applied	Raise Time	Fall Time	Power Consumed	Average temperature achieved
1.	CO_2	3.5mV	10ms	6ms	2.8mW	390° C
2.	CH_4	4.5mV	5ms	6ms	3.6mW	410° C

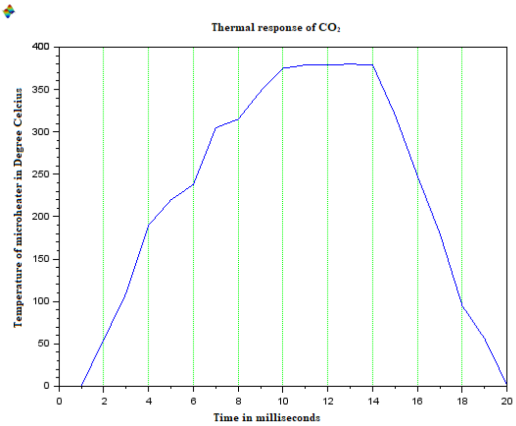

Figure 6: Thermal response of CO_2

Molecules of carbondioxide (CO_2) has special property that can absorb energy from infrared (IR) radiation. The energy from the photon causes the CO_2 molecule to vibrate. Sometime later, the molecule gives up this extra energy by emitting another infrared photon. Once the extra energy has been removed by the emitted photon, the carbon dioxide molecule stops vibrating [14].

CO_2 Molecules are constantly in motion, colliding with other gas molecules and transferring energy from one molecule to another during collisions. In the more-complex, real-world process, a CO_2 molecule would most likely bump into several other gas molecules before re-emitting the infrared photon. The CO_2 molecule might transfer the energy it gained from the absorbed photon to another molecule, adding speed to that molecule's motion. Since the temperature of a gas is a measure of the speed of the molecules in the gas, the faster motion of a molecule that eventually results from the IR photon that was absorbed by a CO_2 molecule raises the temperature of the gases [15]. Therefore, CO_2 has more heat absorbing capacity, hence it needs high temperature environment to be detected. Therefore, the designed microheater reached 390°C to break the CO_2 atoms on the sensor to as shown in the Figure 6. This temperature is very high compared to oxygen and nitrogen because they may break at 60 – 70° C [16]. It has capacity to absorb more heat if the concentration of the gas is increased.

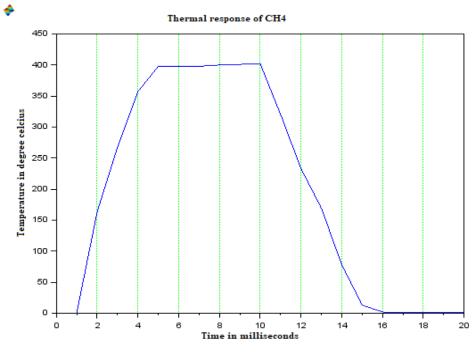

Figure 7: Thermal response of CH_4

Methane has one carbon atom and four hydrogen atoms it is the more powerful gas than CO_2 in the atmosphere. It contributes more to greenhouse problem though it is there in very less quantity compared to CO_2 [17]. Methane has very high IR radiation absorbing capacity compared to CO_2, this property makes it to trap more heat compared to CO_2 [18]. Therefore, CH_4 took more heat from the microheater than CO_2 to break the atoms of the CH_4 as shown in the Figure 7. The CH_4 reported 419° C microheater heat and CO_2 reported 390° C, the comparison graph of both CO_2 and CH_4 is shown in the Figure 8.

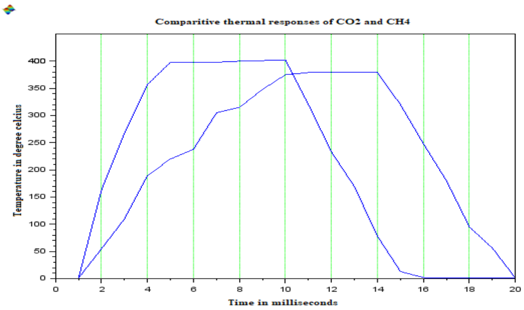

Figure 8 : Comparison of thermal responses of CO_2 and CH_4

The demonstration of the proposed labyrinth structure microheater is conducted in the sensimer device. The increased temperature of microheater to very high elevated temperature makes the microheater appears red hot structure as shown in the Figure 8. The sinusoidal voltage is applied to microheater; therefore, it is showing pulsating output. The Figure 8 shows before voltage supply and after voltage supply to microheater.

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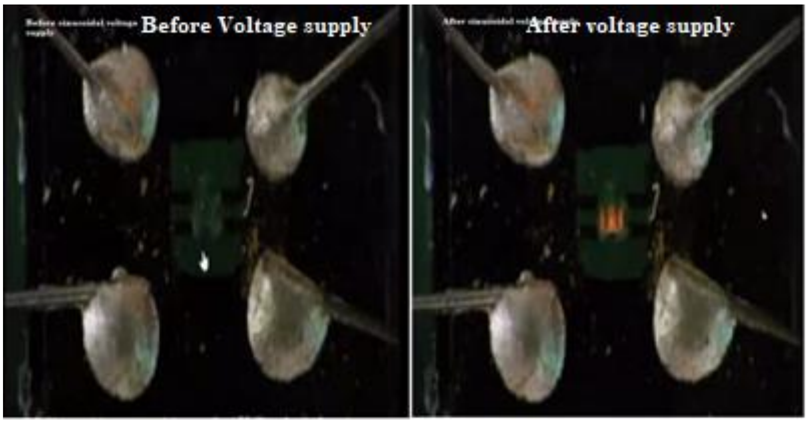

Figure 9: Demonstration imageproposed labyrinth structure microheater pulsating temperature response for sinusoidal voltage input

Conclusion

This work focused on the design of platinum based microheater with novel Labyrinth design in CleWin 4.1 and thermal analysis is conducted for both CO_2 and CH_4 gases using novel machine sensimer. it can be concluded that, the proposed labyrinth structure in microheater is having capability to reach more temperature for very less voltage supply compared other conventional microheater structure. The average temperature of microheater to break the CO2 atoms is reported 390° C and the average temperature to break the CH4 atoms reported 420° C. From the experimental results it can be concluded that, CH_4 gas is more dangerous than CO_2 though is it there in very less concentration in atmosphere compared to CO_2.

Acknowledgement

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Reference:

[1] Spruit, Ronald & Omme, J. & Ghatkesar, Murali & Perez Garza, Hector Hugo. (2017). A Review on Development and Optimization of Microheaters for High-Temperature In Situ Studies. Journal of Microelectromechanical Systems. PP. 1-18. 10.1109/JMEMS.2017.2757402.

[2] Creemer, J.F. & Briand, D. & Zandbergen, Henny &Vlist, W. &Deboer, Charles &Rooij, N.F. &Sarro, P.M.. (2008). Microhotplates with TiN heaters. Sensors and Actuators A: Physical. 148. 416-421. 10.1016/j.sna.2008.08.016.

[3] Materials selection for aerospaceEditor(s): Adrian P. Mouritz,Introduction to Aerospace Materials, Woodhead Publishing, 2012,Pages 569-600,ISBN 9781855739468,https://doi.org/10.1533/9780857095152.569.

[4] Khalid, M. & Abu Bakar, Mohamad &Miskam, Muhamad. (2011). Effect of heater geometry on the high temperature distribution on a MEMS micro-hotplate. Proceedings of the 3rd Asia Symposium on Quality Electronic Design, ASQED 2011. 10.1109/ASQED.2011.6111709.

[5] Jeroish, Z.E., Bhuvaneshwari, K.S., Samsuri, F. et al. Microheater: material, design, fabrication, temperature control, and applications—a role in COVID-19. Biomed Microdevices **24,** 3 (2022). https://doi.org/10.1007/s10544-021-00595-8.

[6] Ali, Syed Zeeshan &Udrea, Florin & Milne, W. & Gardner, J.W.. (2009). Tungsten-Based SOI Microhotplates for Smart Gas Sensors. Microelectromechanical Systems, Journal of. 17. 1408 - 1417. 10.1109/JMEMS.2008.2007228.

[7] raj S. Amiri, M.M. Ariannejad, D. Vigneswaran, C.S. Lim, P. Yupapin, Performances and procedures modules in micro electro mechanical system packaging technologies,Results in Physics,Volume 11,2018,Pages 306-314,

[8] Prasad, K., Bazaka, O., Chua, M., Rochford, M., Fedrick, L., Spoor, J., Symes, R., Tieppo, M., Collins, C., Cao, A., Markwell, D., Ostrikov, K. K., &Bazaka, K. (2017). Metallic Biomaterials: Current Challenges and Opportunities. Materials (Basel, Switzerland), 10(8), 884. https://doi.org/10.3390/ma10080884.

[9] harles Manière, Geuntak Lee, Eugene A. Olevsky,Proportional integral derivative, modeling and ways of stabilization for the spark plasma sintering process,Results in Physics,Volume 7,2017,Pages 1494-1497.

[10] Darrin, Ann &Osiander, Robert. (2011). MEMS Packaging Materials. 10.1007/978-0-387-47318-5_12.

[11] Guan, Teck & Puers, Robert. (2010). Thermal Analysis of a Ag/Ti Based Microheater. Procedia Engineering. 5. 1356-1359. 10.1016/j.proeng.2010.09.366.

[12] Vasiliev, Alexey & Nisan, Anton &Samotaev, Nikolay. (2017). Aerosol/ink Jet Printing Technology for High-Temperature MEMS Sensors. Proceedings. 1. 617. 10.3390/proceedings1040617.

[13] Toskov, Sasa& Glatz, Ronald &Miskovic, Goran & Radosavljevic, Goran. (2013). Modeling and fabrication of Pt micro-heaters built on alumina Substrate. Proceedings of the International Spring Seminar on Electronics Technology. 47-52. 10.1109/ISSE.2013.6648213.

[14] Aldoshin, G. & Yakovlev, S.. (2015). Analytic model of vibrations of a carbon dioxide molecule. Fermi resonance. Mechanics of Solids. 50. 33-43. 10.3103/S0025654415010045.

[15]Thomas R. Anderson, Ed Hawkins, Philip D. Jones,CO2, the greenhouse effect and global warming: from the pioneering work of Arrhenius and Callendar to today's Earth System Models, Endeavour,Volume 40, Issue 3,2016,Pages 178-187.

[16] Kim, J., Ha, H., Doh, W.H. et al. How Rh surface breaks CO_2 molecules under ambient pressure. Nat Commun **11,** 5649 (2020). https://doi.org/10.1038/s41467-020-19398.

[17] Darkwah, Williams Kweku & Odum, Bismark & Addae, Maxwell & Koomson, Desmond & Kwakye Danso, Benjamin & Oti-Mensah, Ewurabena & Asenso, Theophilus & Buanya, Beryl. (2018). Greenhouse Effect: Greenhouse Gases and Their Impact on Global Warming. Journal of Scientific Research and Reports. 17. 1-9. 10.9734/JSRR/2017/39630.

[18] Hdr, Zdgvbdh & Tan, Qiulin & Wendong, Zhang &Chenyang, Xue& Tao, Guo & Jijun, Xiong. (2011). Miniature low-power IR monitor for methane detection. Measurement. 44. 823-831. 10.1016/j.measurement.2011.01.021.