Sensors & Transducers, Vol. 156, Issue 9, September 2013, pp. 317-323

www.sensorsportal.com

Sensors & Transducers

http://www.sensorsportal.com

Preparation and Test of Fe/Cr/Al -Ni/Cr Composite Film Micro-heater

*** Qiaohua Feng, Yunbo Shi, Mengmeng Wang, Fang Du, Lining Sun**
The Higher Educational Key Laboratory for Measuring & Control Technology
and Instrumentation of Heilongjiang Province, Harbin University of Science and Technology,
No. 52, Xuefu Road, Harbin, 150080, China
Tel: +86 0451-86390840, fax: +86 0451-86390840
* E-mail: fengqiaohua80@126.com

Received: 18 June 2013 /Accepted: 25 August 2013 /Published: 30 September 2013

Abstract: MEMS gas sensor is one of the development directions of the sensor, and it must work at a certain temperature, so micro heater is an important part of MEMS gas sensor. In this paper, aiming at the high cost and complex process of Pt film heater, we developed the research of Fe/Cr/Al and Ni/Cr cheap metals replacing Pt metal. With Fe/Cr/Al and Ni/Cr metal as the targets respectively, Fe/Cr/Al and Fe/Cr/Al-Ni/Cr composite thin film was prepared by DC sputtering on the wafer substrate and was made into micro-heater by wet etching after heat treatment. Finally, the film was characterized by scanning electron microscope (SEM), and the performance of micro heater was test. The characterization indicated that the thickness of film was between 500 nm and 800 nm, the grain size of Fe/Cr/Al was about 40 nm. The test results showed that the change of resistance was small in the range of 0-200 ^{o}C, so the film suited to be made into micro heater. The relationship between power of micro heater and temperature of chip was linear. The ambient temperature had certain influence on the temperature of chip. In certain DC voltage conditions, when the temperature of chip was 200 ^{o}C, power consumption of 75 Ω Fe/Cr/Al-Ni/Cr composite film was less than 700 mW, and the heating rate was more than 0.5 ^{o}C/s. It can satisfy the requirement of MEMS micro gas sensor, which has research value for the substitution of Pt thin film micro heater. *Copyright © 2013 IFSA.*

Keywords: Composite film, Magnetron sputtering, Micro heater, MEMS sensor, Fe/Cr/Al.

1. Introduction

Resistive semiconductor gas sensor is highly regarded in the various types of gas sensors, after decades of change its structure develops from ceramics sintered type, thick film type to film type, array, silicon-based micro-structure type, main development trend is direction of miniaturization, integration, multi-function and low-cost.

As is well known, resistive semiconductor gas sensor needs heat treatment for a longer time before used in order to improve the stability of the sensor and to be periodically heated at work to shorten the recovery time of the sensor, a gas usually reacts with the catalyst at a specific temperature, and a large area of the uniform temperature district is needed to ensure its selectivity and sensitivity. Metal oxide gas-sensitive element normally operates in high temperature (200 to 450 ^{o}C). The most commonly used sintered tin oxide (SnO_2) gas-sensitive element works generally at 200 to 300 ^{o}C. Zinc oxide (ZnO) thin film gas sensor works at a higher temperature of 400-500 ^{o}C. The usually working temperature of the organic polymer semiconductor gas sensor is

100-200 °C. Therefore, the micro-heater is an important part of the MEMS gas sensor.

At present different material micro-heaters are extensively researched at home and abroad. A heater of Al-doped polysilicon was developed by Rong Hao and suitable for performance requirements of thermo-mechanical micro-nanofabrication of polymer materials [1]. Ni/Cr micro-heater array was manufactured on the Si substrate by Chen Sihai and the resistance was about 10 Ω, when the voltage was 10 V, the temperature could reach thousands of degrees, which could be used in MEMS chip level hermetically sealed package [2]. Cr and Ni-Cr metal film micro-heater was developed and able to meet the requirements of PCR biochip and silicon thermal distributed micro flow sensor by Professor Yan Weiping [3]. Permalloy micro heater was designed based on the principle of induction heating by Liu Bedong and had advantages of easy processing and convenient for seal [4]. RuO_2 heater was integrated on the aluminum oxide ceramic substrate by Bo Zikui through titration covering film formation method with a pulsed YAG laser micro-machining, the average consumption was less than 500 mW, the thermal response time was less than 20 s, and the temperature gradient was less than 5 °C/mm, which could fully collect the gas sensitive information of gas-sensitive materials [5]. SiC micro heater was used in NO gas sensors by Jae-Cheol Shim, power consumption was 13.5 mW when the chip temperature was 300 °C, and had an advantage in the high-voltage and high temperature [6]. Ni-Cr-Pt three layer metals were used to make hot-film velocity sensor by Que Ruiyi [7]. But the most widely used micro-heater was polycrystalline silicon thin-film micro-heater [8-10] and Pt film micro-heater [11-17].

Platinum is expensive, has poor adhesion of the platinum film and the silicon or silica, and needs titanium or other film as an intermediate layer, which makes the platinum resistance temperature characteristic complex [18]. Platinum is a difficult corrosive metal, the lift-off process instead of the normal wet etching process is used in the preparation of the Pt resistance, and the preparation process is complicated [19].

Fe/Cr/Al and Ni/Cr films have the advantages of easy manufacture, the relatively mature process, the small temperature coefficient of resistance, cheap and good adhesion with the silicon or silicon dioxide, can be prepared through a normal wet etching process, which has great significance to the research of micro-heater. While compared with the nickel-chromium alloy, Fe/Cr/Al alloy has the advantages of a long life, high surface load, good oxidation-resistance, small specific gravity, less influence of cold working and heat treatment on resistivity and cheap, so research of Fe/Cr/Al thin film heater is more practical.

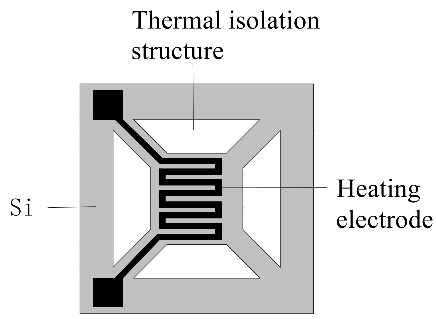
Because the resistivity of Fe/Cr/Al was higher than Ni/Cr, in order to reduce the resistance of the film of the same film thickness and improve the heating efficiency, in this paper the Fe/Cr/Al thin film was sputtered on the surface of nickel-chromium thin film to form Fe/Cr/Al-Ni/Cr composite film and made into micro-heater, and heating performance of micro-heater was test.

2. Experimental Details

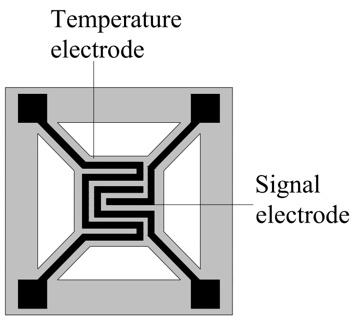
2.1. Structure Design of the Micro-Heater

In this paper, the metal thin film heater developed was mainly used for MEMS organic semiconductor gas sensor, design of the micro-heater should make the temperature distribution as uniform as possible and the working temperature of the gas-sensitive film as 100 to 200 °C.

The structure of indirectly heated semiconductor gas sensor was shown in Fig. 1.



(a) The front of the chip



(b) The back of the chip

Fig. 1. The configuration diagram of micro-heater.

The configuration diagram of micro-heater herein was shown in Fig. 1 (a), the metal thin film was designed into serpentine in order that the thermal field distribution was uniform [20]. The substrate was monocrystalline with the crystal orientation (100), a thickness of 200 μm and the size of 3 mm × 3 mm, and the surface of the wafer was deposited silicon dioxide with a thickness of 300 nm. The heating electrode was Fe/Cr/Al and Fe/Cr/Al-Ni/Cr composite film respectively, each of the strip width was 50 μm, the spacing was 50 μm, the size of the line was 1 mm × 50 μm. The thermal isolation structure was isosceles trapezoid, the upper base was 0.5 mm, and the lower bottom was 1.4 mm, and the height was 0.5 mm. The thermal isolation structure was cut by micro-laser processing and could reduce the heat exchange between the micro-heater and the substrate, energy loss and power consumption. In order to test the working temperature of the chip, temperature sensor was produced in the back of the chip and made of Pt, as shown in Fig. 1 (b). The type of temperature sensor was Pt1000. Signal electrode was used to test the resistance of sensitive material.

2.2. Heater Preparation Process

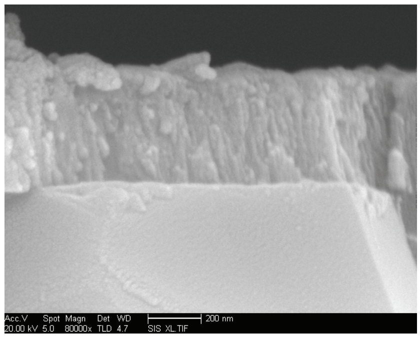
In the experiment iron-chromium-aluminum alloy (82 % iron, 14 % chromium, 4 % aluminum) and nickel-chromium (80 % nickel, 20 % chromium) alloy as targets were made into thin film with JGP560C ultra-high vacuum sputtering mechanism by magnetron sputtering method, magnetron sputtering could ensure alloy material component unchanged during deposition, to obtain a dense film structure and metal film was firmly bonded with the substrate.

The steps of thin film resistor production process were as follows: at first, the wafer was pretreated with acetone, ethanol, deionized water for 10 min respectively through ultrasonic cleaning and dried in oven to remove the contaminants on the substrate, which enhanced the adhesion between substrate and film; then thin film of 500 nm to 800 nm was plated on a silicon wafer with a DC magnetron sputtering method, the main production process conditions of thin film were that the body vacuum was 2×10^{-5} Pa, striking pressure was 1.6 Pa, the working pressure was 0.5 Pa, sputtering power was 50 W and sputtering time was about 1-2 h; Fe/Cr/Al and Ni-Cr thin film needed to be heat-treated at 400 °C for 3 hours, components of the alloy were diffused each other and lattice defects could also be eliminated, which improved the thermal stability of thin-film, eliminated internal stress, and enhanced the adhesion of the film and the substrate, eliminated the adsorption of the gas molecules in the film and protected the film from contamination and corrosion; after that the resistance pattern was made through the wet etching process with the homemade etching liquid and thermal isolation structure on the chip was cut by micro-laser processing; finally leads were welded and heating performances were test.

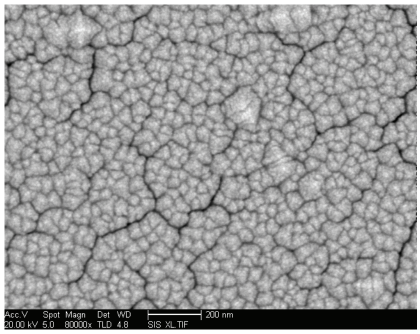
3. Results and Analysis

3.1. Analysis of Film Performance

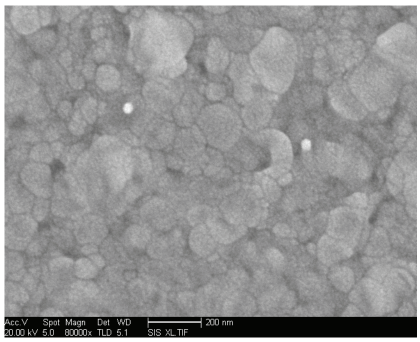
The scanning electron micrographs were obtained by Quanta 200 scanning electron microscope. Fig. 2 was a scanning electron micrograph of the Fe/Cr/Al film (sputtering power of 50 W, the sputtering time of 2 h). Film thickness was about 600 nm and particles arranged neatly as shown in Fig. 2 (a). Film surface was made of particles arranged compactly and uniformly, the particle was about 40 nm (the sizes of Fe, Cr and Al particles were different), there were no voids, high flatness and cracking stripes on the film surface as shown in Fig. 2 (b); the size of grain in the film grew after the heat treatment, cracking of film surface was improved, and the flatness of the film was also improved, as shown in (c), at the same time, after the annealing treatment, the resistance of the metal thin film was the stable which was conducive to the performance test of the heater.



(a) Fracture



(b) Surface



(c) After heat treatment

Fig. 2. The SEM pictures of Fe/Cr/Al film.

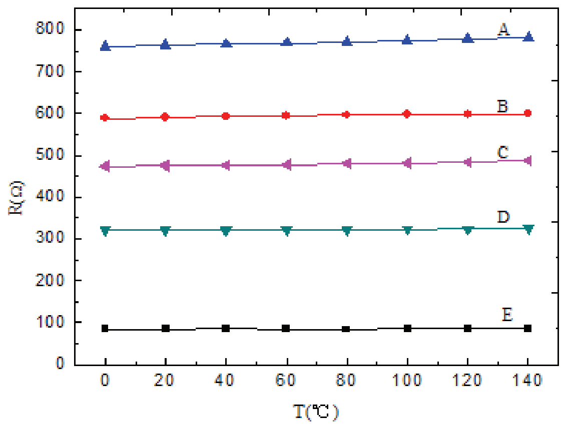
3.2. Analysis of Heater Performance

In order to understand whether the Fe/Cr/Al and Fe/Cr/Al-Ni/Cr composite metal thin-film heater prepared in this paper could meet the requirements above, the heaters with different resistances were put into TEMI880 high and low temperature alternating experimental box to test the heating effect. In the experiment the different resistances of heaters were obtained by changing the thickness of Fe/Cr/Al film or Ni/Cr film.

3.2.1. Temperature Stability of Film Resistivity

Temperature stability of the film resistivity was discussed from the two aspects of the characteristics of resistance with the change of temperature and the relationship between the heating voltage and heating current, respectively.

Metal film heaters required the resistivity varied as little as possible with the temperature and had high stability [3]. Fig. 3 was a characteristic curve of the heater resistance with the change of temperature, A, B, C were Fe/Cr/Al thin-film resistors, and D, E were the Fe/Cr/Al-Ni/Cr composite thin film resistors. Within 0-150 °C the resistance value of the all heaters changed little and was relatively stable. The highest test temperature of high and low temperature alternating experimental box used in this experiment was 150 °C, therefore the resistance change within 150 °C was only test.



A, B, C: Fe/Cr/Al; D, E: Fe/Cr/Al - Ni/Cr

Fig. 3. The curve of resistance changing with temperature.

Fig. 4 was the relationship curve between the heating voltage and heating current of the four different resistance heaters working properly at constant ambient temperature of 25 °C. As can be seen from Fig. 4, the relationship between the heating voltage and heating current was approximately linear, i.e. when the heater was loaded the input voltage, the resistance change was little and relatively stable; and under the same voltage, smaller the resistance of the heater, the greater the heater power consumption increased, and the faster the temperature rose.

From above, the resistivity of Fe/Cr/Al thin film and Fe/Cr/Al-Ni/Cr composite thin film was stable, and varied little with the change of temperature of environment and the heating voltage loaded, therefore, they were suitable for heater.

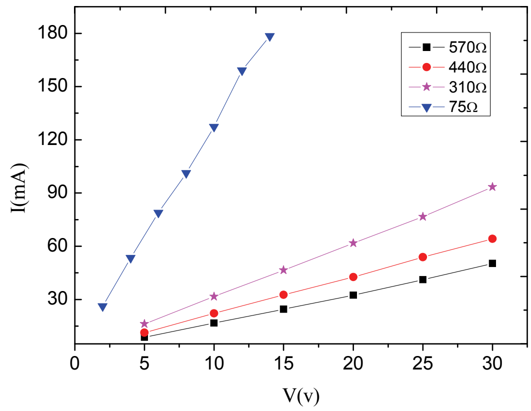


Fig. 4. The relation of voltage and current.

3.2.2. Thermal Power Characteristics of the Thin Film Heater

When the thermal power of the heaters was different, the highest temperature of the chip was test in order to control the temperature of chip through the heater power. Thermal power characteristics of the thin film heater was discussed from the three aspects of the relationships between the heating voltage and the chip temperature, power consumption and chip temperature, the influence of ambient temperature on the chip temperature, respectively.

Fig. 5 was a relationship curve of the heating voltage and chip temperature at constant ambient temperature of 25 °C, the resistor of 75 Ω was Fe/Cr/Al-Ni/Cr composite film resistor, and the rest were Fe/Cr/Al thin film resistors. As shown in Fig. 5, all the heaters could reach 200 °C under a certain voltage, while at the same voltage, the smaller the resistance, the higher the temperature of the heater would be. Therefore, in order to meet the requirements of the MEMS gas sensor working at a low voltage, the resistance value of the heater was reduced. Because the resistivity of the Fe/Cr/Al thin film was relatively large, on the basis of without increasing the thickness of the film, the Fe/Cr/Al-Ni/Cr composite film was used to achieve a low resistance value. The rate of chip warming up was greater than 0.5 °C/s.

Fig. 6 was the relationship curve of the heater power of different resistance and chip temperature at constant ambient temperature of 25 °C, the temperature of the chip increased gradually with the increase of power consumption of the heater, the relationship between the heater power and the chip temperature was approximately linear; under the same power consumption the lower the resistor value, the higher the temperature was; when the chip temperature needed to achieve 200 °C, power consumption of 75 Ω Fe/Cr/Al-Ni/Cr thin film was less than 700 mW, while the power consumption of 310 Ω Fe/Cr/Al film was nearly 800 mW, moreover, in order to achieve the same working temperature, the power of both heater was greater than the Pt thin-film heater [6], which because the Fe/Cr/Al film resistor value was larger.

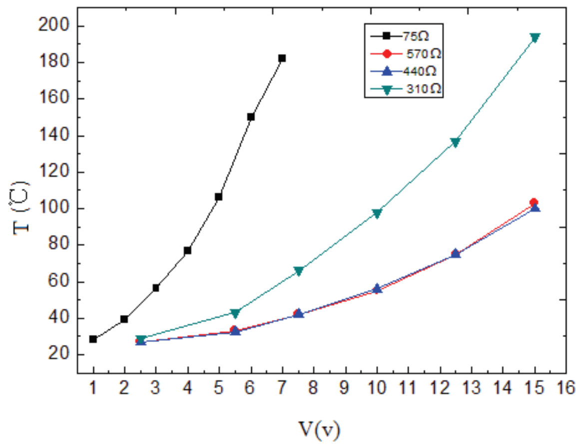


Fig. 5. The relation of voltage and temperature.

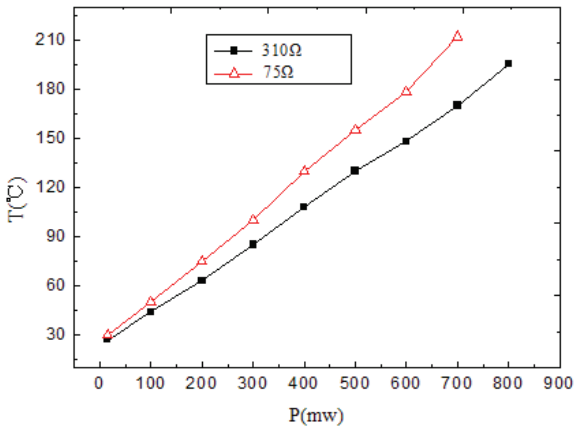


Fig. 6. The relation of power and temperature.

Fig. 7 was the relationship curve of the 570 Ω Fe/Cr/Al thin film heater power and chip temperature at ambient temperature of -20 °C and 25 °C respectively. As shown in Fig. 7, the influence of ambient temperature on the heater was great, under the same power consumption, chip temperature at 25 °C of ambient temperature was higher than the chip 45 °C at -20 °C of ambient temperature, which was just the temperature difference of the two environments; at the different ambient temperatures, the relation of the heater power and the chip temperature was still linear, and with the increase of power consumption, two heater temperatures increased at the same rate. Therefore, when the micro-sensor worked in different environments, we must take into account the impact of ambient temperature on the heater, that is to say we must take into account the temperature difference between the environment and at room temperature in order to adjust the power consumption of the micro heater to achieve the desired operating temperature.

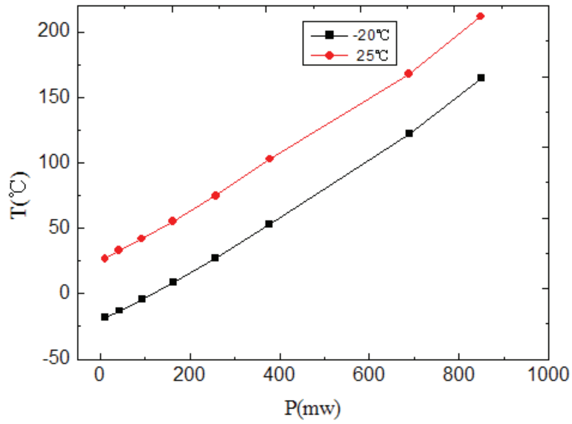


Fig. 7. The relation of power and temperature under different ambient temperature.

4. Discussion

As shown from temperature stability of the composite film resistivity discussed above, for the thin film process, micro heater film was doped low-resistance metal to form composite film, temperature stability of the composite film resistivity varies little, which can reduce resistivity of micro heater film, and reduce the thickness of micro heater film, which has practical significance for reducing costs of micro heater and simplifying preparation process of micro heater.

As shown from the thermal power characteristics of the thin film heater discussed above, the temperature of chip was related to the environment temperature, the resistance of the chip and the heating voltage. According to the energy conservation, in order to maintain a system a certain temperature the power consumption of heater is determined by heat dissipation. Therefore, the environment temperature is different, the heat dissipation system is different, and therefore power required is different in order to maintain the same temperature. While the voltage and the environment temperature are constant, the smaller the resistance, the greater heating power and the higher the equilibrium temperature reached.

It was tested in the experiments that the resistance of the heater did not vary with temperature and the heating voltage, so as long as the heating voltage was constant, power consumption of heater was constant, and the relationship of the thermal power and chip temperature was established by experiments, thus the relationship curve between the heating voltage and the chip temperature can be established, so we can easily know the temperature of the chip through the heating voltage level.

5. Conclusions

In this paper, aiming at the high cost and complex process of precious metal Pt thin film heater, we selected the Fe/Cr/Al and Ni-Cr cheap metal to prepare micro-heater. The Fe/Cr/Al thin film resistivity varied little with temperature and the heater could reach 200 ^{o}C in a certain DC voltage conditions, but because of the large resistance value, high voltage was needed to reach the desired temperature. Therefore, in order to reduce the film resistance, on the basis of without increasing the thickness of the film, the Ni-Cr film was coated on the surface of Fe/Cr/Al film to form the Fe/Cr/Al-Ni/Cr composite film, whose resistivity varied little with temperature either and was suitable for micro-heater. The experimental results were as follows: 75 Ω Fe/Cr/Al-Ni/Cr composite thin-film heater in 700 mW power consumption could reach 200 ^{o}C, and the heating rate was greater than 0.5 ^{o}C/s; the influence of ambient temperature on the micro-heater temperature was great, so the temperature difference between the environment and room temperature should be considered to adjust the power consumption of the micro-heater to achieve the desired operating temperature at the non-room temperature. In general, the Fe/Cr/Al and Ni-Cr cheap metal thin-film heater could meet the requirements of the MEMS micro gas sensor and had certain significance for replacing precious metals Pt thin-film heater.

Acknowledgements

Thanks the support of Key Projects from Natural Science Foundation of Heilongjiang Province (No. ZD201217), Technological Innovation Talent Research Special Funds of Harbin (No. 2010RFXXG009), and Youth Scientific Research Fund of Harbin University of Science and Technology (No. 2011YF028).

References

[1]. Rong Hao, Zhao Gang, Chu Jiaru, Al doped poly-Si micro-heater for thermomechanical fabrication of micro/nano structure, *Optics and Precision Engineering*, Vol. 19, Issue 1, 2011, pp. 124-131.

[2]. Chen Sihai, Chen Mingxiang, Yi Xinjian, et al., Design and fabrication of microheater array for MEMS wafer-level scale hermetic package, *Micronaonoeletronic Technology*, Issue 7/8, 2003, pp. 249-250.

[3]. Yan Weiping, Zhu Jianbo, Ma Lingzhi, et al., Research of Metal Membrane Heater, *Chinese Journal of Sensors and Actuators*, Issue 4, 2004, pp. 615-618.

[4]. Liu Bendong, Jiao Bingfeng, Li Desheng, Design of Micro Heater Based on Induction Heating, *Chinese Journal of Scientific Instrument*, Vol. 31, Issue 8, 2010, pp. 48-51.

[5]. Bai Zikui, Wang Aihua, Xie Changsheng, Experimental Study of the Integrated Nano Gas Sensor Arrays Fabricated by Laser Micro-Machining and Their Corresponding Characteristics, *Chinese Journal of Sensors and Actuators,* Issue 4, 2004, pp. 655-659.

[6]. Jae-Cheol Shim, Gwiy-Sang Chung, Fabrication and Characteristics of Pt/ZnO NO Sensor Integrated SiC Micro Heater, in *Proceedings of the Conference on'9th IEEE Sensors Conference (SENSORS 2010)',* Waikoloa, HI, United States, November 1-4, 2010, pp. 350-353.

[7]. Que Ruiyi, Zhu Rong, Liu Peng, et al., Combined hot-film anemometers for measuring flow speed vectors, *Optics and Precision Engineering*, Vol. 19, Issue 1, 2011, pp. 103-109.

[8]. P Kahroba, I Mirzaee, P Sharifi, et al., The microcavity-based micro-heater: an optimum design for micro-heaters, *Microsyst. Technol.*, Issue 14, 2008, pp. 705–710.

[9]. Wen-Chie Huang, Chung-Nan Chen, Shang-Hung Shen, *et al.,* Study of the Annealing Effect of Low-Temperature Oxide on the Etch Rate in TMAH Solutions for Micro-Heater Applications, in *Proceedings of the IEEE Conference on 3rd International Conference on Nano/Molecular Medicine and Engineering (NANOMED 2009)'* Tainan, Taiwan, October 18-21, 2009, pp.175-179.

[10]. Ching-Liang Dai, Mao-Chen Liu, Nanoparticle SnO_2 Gas Sensor with Circuit and Micro Heater on Chip Fabricated Using CMOS-MEMS Technique, in *Proceedings of the IEEE Conference on' the 2nd International Conference on Nano/Micro Engineered and Molecular Systems',* 2007, pp. 959-963.

[11]. Wang Junqing, Wu Cinan, Zhang Pu, et al., Study on Characterization of Micro Heater Used for Micro PCR Chip, *Semiconductor Technology*, Vol. 37, Issue 6, 2012, pp. 442-447.

[12]. Bobby Reddy, Oguz H. Elibol, Pradeep R. Nair, *et al.*, Silicon Field Effect Transistors as Dual-Use Sensor-Heater Hybrids, *Anal. Chem.*, Issue 83, 2011, pp. 888–895.

[13]. S. E. Moon, J.-W. Lee, N.-J. Choi, *et al.,* High-response and Low-power-consumption CO Micro Gas Sensor Based on Nano-powders and a Micro-heater, *Journal of the Korean Physical Society*, Vol. 60, Issue 2, 2012, pp. 235-239.

[14]. Hyung-Kun Lee, Seung Eon Moon, Nak-Jin Choi, et al., Fabrication of a HCHO Gas Sensor Based on a MEMS Heater and Inkjet Printing, *Journal of the Korean Physical Society*, Vol. 60, Issue 2, 2012, pp. 225-229.

[15]. Prasad Mahanth, Yadav R. P., Sahula V., *et al.*, Design and simulation of double-spiral shape micro-heater for gas sensing applications, *Sensors & Transducers*, Vol. 129, Issue 6, June 2011, pp. 135-141.

[16]. Liu Zewen, Tian Hao, Liu Chong, Experiment and thermal calculation of micro heater, *Optics and Precision Engineering*, Vol. 19, Issue 3, 2011, pp. 612-619.

[17]. Xue Yanbing, Tang Zhenan, Gas sensor array based on ceramic micro-hotplate for flammable gas detection, *Optics and Precision Engineering*, Vol. 20, Issue 10, 2012, pp. 2200-2206.

[18]. Ting Xu, Hui Li, Zhihong Wang, et al., A Novel Method of Growing Aligned Carbon Nanotubes at Low Temperature by Using Integrated Micro-Heater,

in *Proceedings of the 58th Conference on Electronic Components and Technology Conference (ECTC)'*, Lake Buena Vista, FL, United States, May 27-30, 2008, pp. 1395-1399.

[19]. Zhao Wenjie, Zhou Zhen, Shi Yunbo, *et al.*, Study on Si-based Plate Microstructure Gas Sensor Integrated with Temperature Sensor, in *Proceedings of the 4th Conference on International Symposium on Computational Intelligence and Industrial Applications (ISCIIA 2010)'*, Harbin, China, August 2-8, 2010, pp. 279-282.

[20]. Sidek, M. Z. Ishak, M. A. Khalid, et al., Effect of Heater Geometry on the High Temperature Distribution on a MEMS Micro-hotplate, in *Proceedings of the 3rd Asia Symposium on Quality Electronic Design (ASQED 2011)'*, Kuala Lumpur, Malaysia, July 19-20, 2011, pp. 100-104.
