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LETTER

Fabrication of the Pt Microheater Using Aluminum Oxide as a Medium Layer and Its Characteristics

Gwiy-Sang Chung and Sang-Soo Noh

Division of Electronics and Mechanical Engineering, Dongseo University,
San 69-1, Churye-Dong, Sasang-Gu, Pusan 617-716, Korea

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The electrical and physical characteristics of aluminum oxide films, onto which Pt thin films are deposited by reactive sputtering and DC magnetron sputtering, respectively, were analyzed by increasing the annealing temperature (400 ~ 800°C) by a four-point probe method and by SEM and XRD. Below the annealing temperature of 600°C, aluminum oxide films improved Pt adhesion to silicon thermal oxide films and acted as insulators without chemically reacting with Pt thin films. Also, the resistivity of Pt thin films was improved. However, these properties of aluminum oxide films with deposited Pt thin films were degraded at annealing temperatures above 600°C because aluminum oxide films were changed into aluminum metal which reacted with the deposited Pt thin films. The thermal characteristics of Pt microheaters were analyzed using Pt-RTD temperature sensors integrated on the same substrates. In the analyses of the properties of the Pt microheater, a Pt microheater with the smaller active area had better thermal characteristics. The temperature of the Pt microheater with an active area of 200 μm × 200 μm was 400°C with a heating power of 1.5 W.

1. Introduction

The research on microsensors utilizing Si micromachining technology has recently been of great interest.[1] Among these mechanical, chemical and radiant sensors, gas sensors, vacuum sensors and flow sensors are based on chemical reactions between gas

molecules and a sensitive film to produce a detectable electrical signal.[2,3] These chemical reactions, such as gas adsorption and desorption and diffusion effects, significantly depend on the operating temperature, which is an important factor for the optimization of the sensor properties such as sensitivity, selectivity and response time. Therefore, microheaters must be implanted with these sensors to maintain the operating temperature. Up to now, the research on microheaters (SiC thin films,[4] poly Si,[2] NiFe alloy,[5] NiCr,[6] Pt/Ti[7] and Pt/Cr[8]) using micromachining technology has been actively carried out, and these heaters have been found to have the following advantages: low power consumption, accurate temperature control, low heat capacity and easy realization of sensor arrays.

Along with the fabrication of Pt microheaters based on Si substrates, adhesion layers have also been studied because of the poor adhesion of Pt thin films to silicon thermal oxide films. If certain metallic materials were used as adhesion layers, the material of the adhesion layers would react with Pt thin films during the annealing treatment at high temperature, and the typical characteristics of the Pt material and the Pt adhesion to silicon thermal oxide films would be degraded. Pt thin films must be treated at a high annealing temperature (over 1,000°C) to obtain bulk characteristics because Pt has a high melting point (1,780°C).[9] Therefore, dielectric thin films are safer than metallic thin films electrically under the condition of a high annealing temperature. In this paper, we present not only the fabrication process of a microheater based on the Si substrates using aluminum oxide films as a medium (adhesion) layer and Pt as a heater material, but also its thermal characteristics. Aluminum oxide films improve Pt adhesion to silicon thermal oxide films without undergoing any reactions with Pt thin films at a high temperature. In addition to the linear response, Pt thin films gain the advantages of chemical and thermal stability so that its resistor has no variations of resistance values in spite of being heated at a high temperature for a long time (over 600 min). The electrical and physical characteristics of aluminum oxide films with Pt thin films were analyzed using a four-point probe method and by α-step, SEM (scanning electron microscope) and XRD (X-ray diffraction) while increasing the annealing temperature. The thermal characteristics of Pt microheaters were analyzed using Pt-RTDs (platinum resistance thermometer devices) integrated on the same substrates, and the resistance ratios of the heater and the RTD resistor were also investigated.

2. Materials and Methods

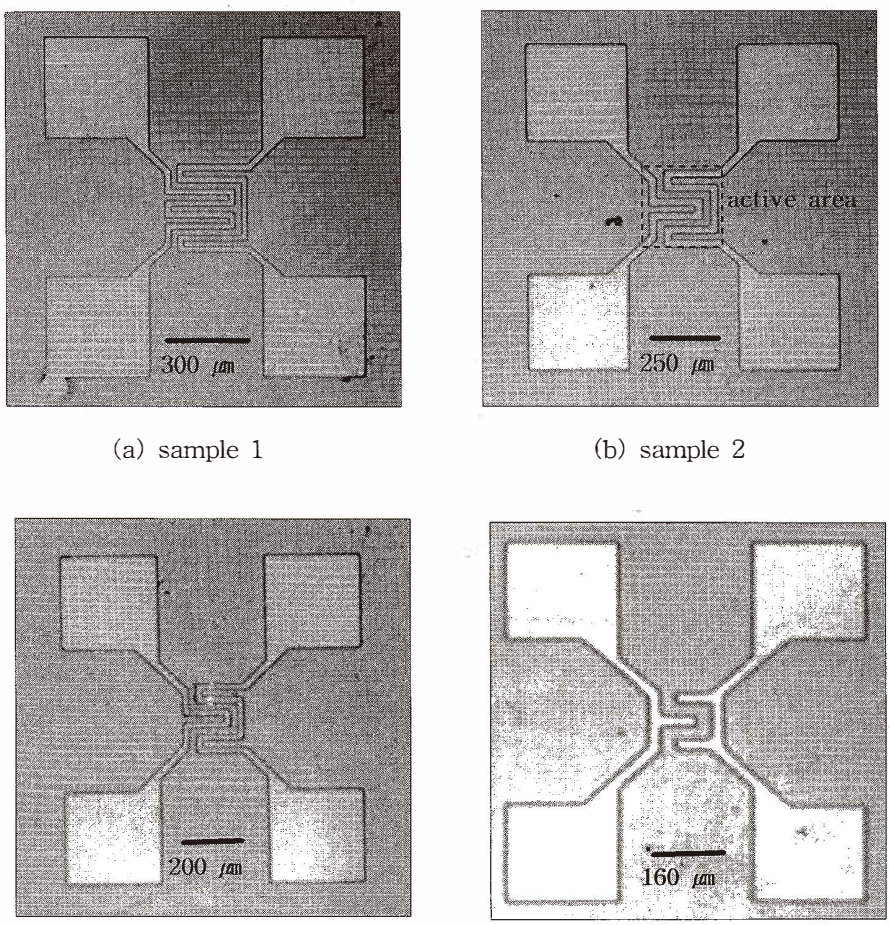
2.1 *Preparation for annealing the sample*

The N-type (100) Si wafer with a thickness of 530 μm and thermal oxide films with a thickness of 3,000 Å were used as substrates after the standard cleaning process, and their resistivity was 4 ~ 5 $\Omega \cdot$ cm. Aluminum oxide films with a thickness of 1,500 Å deposited by reactive sputtering (working vacuum (in mixtures, Ar : O_2 = 1 : 1): 10 mTorr, input power density: 7 W/cm^2, substrate temperature: 25°C) were used to improve Pt adhesion to silicon thermal oxide films, and Pt thin films were deposited by DC magnetron sputtering under the following conditions: working vacuum 5 mTorr; input power density 7 W/cm^2; substrate temperature 300°C. After the annealing treatment of aluminum oxide films in the

temperature range of 400 ~ 800°C in N_2 atmosphere using a quartz tube furnace, the annealing effects and the influences on the Pt thin films were investigated.

2.2 *Fabrication of microheaters*

Microheaters were fabricated by the photolithography process and the lift-off method. Figure 1 shows the micrographs of Pt microheaters and Pt-RTD temperature sensors, which were integrated on the sample substrates. Pt microheaters and Pt-RTD temperature sensors differ in both structure and size. The characteristics of Pt microheaters and Pt-RTD



(a) sample 1 (b) sample 2

(c) sample 3 (d) sample 4

Fig. 1. Micrographs of Pt microheater and Pt-RTD fabricated on aluminum oxide films.

temperature sensors were investigated after the annealing treatment at 600°C for 60 min in N_2 atmosphere. Spin-on-glass (SOG) was used for passivation to protect devices from external particle contaminants and dirt after being baked at 100°C and 500°C sequentially, and then Pt wire was bonded with silver epoxy. The properties of both devices were analyzed in a closed system to control the conditions of atmospheric gas and vacuum for the purpose of minimizing the convection effects of atmospheric gas.

3. Results and Discussion

3.1 *Analysis of aluminum oxide components*

The EDS (electron diffraction spectroscopy) spectram of aluminum oxide films, deposited to a thickness of 1,500 Å by reactive sputtering in oxygen-argon mixtures, is shown in Fig. 2. It is noted from the analysis of the components of aluminum oxide films deposited on the Si wafer that aluminum has been oxidized by reaction between Ar and O_2, and the

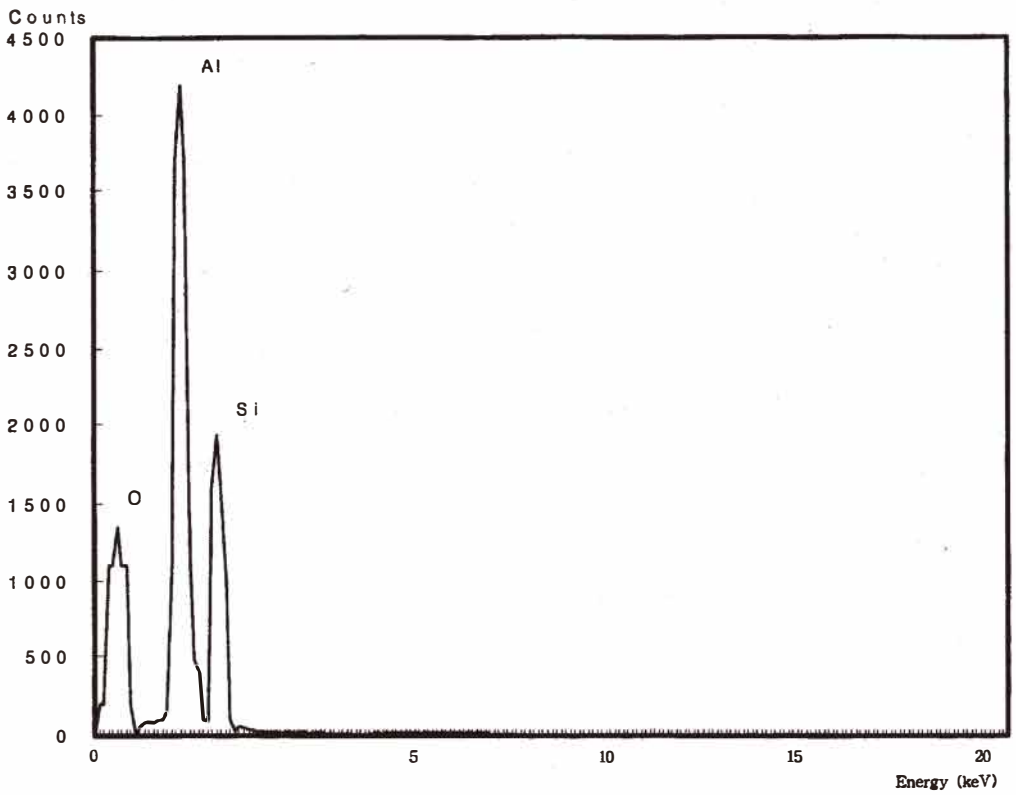


Fig. 2. EDS spectrum of aluminum oxide films deposited by reactive sputtering.

component of Si is considered to be detected from substrate materials due to the thin aluminum oxide films. The proportions of Ar, O_2 and Si were 45.30%, 39.41% and 15.29% in weight, respectively.

3.2 *Annealing characteristics of aluminum oxide and Pt thin films*

The variations in electrical resistivity and sheet resistivity of aluminum oxide films with deposited Pt thin films are shown in Table 1 and Fig. 3, respectively. The electrical resistivity of Pt thin films is the product of the thickness measured by α-step multiplied by the electrical sheet resistivity measured by a four-point probe method. Below the annealing temperature of 600°C, aluminum oxide films improved the Pt adhesion to silicon

Table 1
Variations of electrical sheet resistivity of aluminum oxide films with annealing temperature.

Annealing Temperature (°C)	400	500	600	700	800
Sheet Resistivity (Ω/□)	∞	∞	∞	10.47	3.13

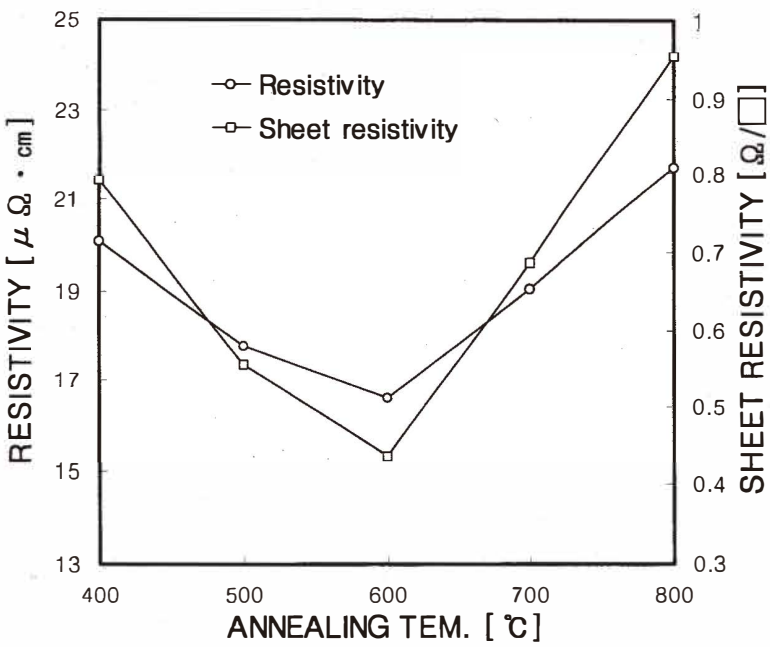


Fig. 3. Electrical resistivity and sheet resistivity of Pt thin films deposited on aluminum oxide films as a medium layer with annealing temperature.

thermal oxide films and insulation without undergoing any chemical reactions with Pt thin films, and the electrical characteristics of deposited Pt thin films were improved. However, the properties of both of these layers were degraded above the annealing temperature of 700°C because aluminum oxide was changed into aluminum metal which then reacted with the deposited Pt thin film.

Figure 4 shows SEM images of Pt thin films deposited on aluminum oxide films at different annealing temperatures. Sample (a) has a uniform surface before the annealing treatment and the characteristics are preserved to 600°C, but the characteristics of samples (c) and (d) are suddenly degraded. The degradation of the uniform surface above the annealing temperature of 700°C is regarded to result from the chage of aluminum oxide to

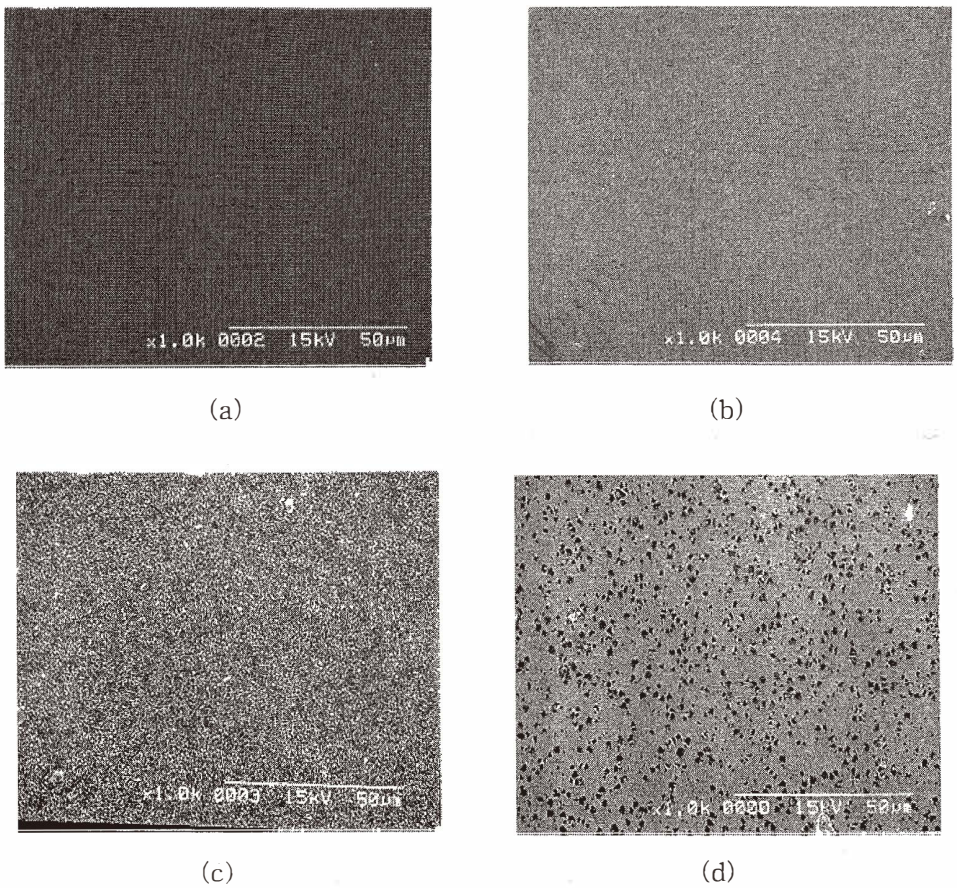


Fig. 4. SEM images of Pt thin films deposited on aluminum oxide films (a) without annealing, and with annealing at (b) 600°C, (c) 700°C and (d) 800°C .

aluminum metal which then reacted with the deposited Pt thin films. The characteristic of Pt adhesion was severely degraded above the annealing temperature of 800°C. The XRD patterns of aluminum oxide film at different annealing temperatures are shown in Fig. 5. In the samples without the annealing treatment and with the annealing treatment below 600°C, only the aluminum oxide peak appears at $2\theta = 28.5°$, while it disappears and new peaks, which have been identified as aluminum peaks based on JCPDS (Joint Committee on Powder Diffraction Standards) data, appear with increasing annealing temperature above 600°C. Resistance ratios of Pt-RTD resistors were measured from 25°C to 450°C in a constant-temperature (25°C) and constant-humidity (40%) system. The resistance ratios

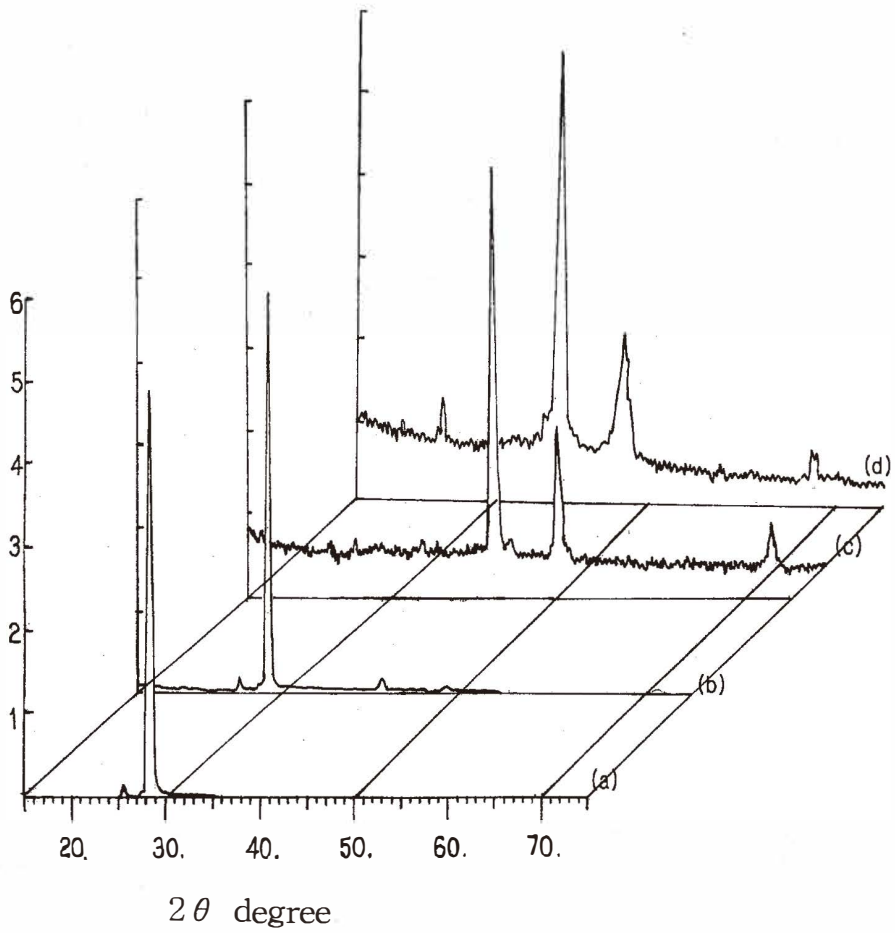


Fig. 5. XRD patterns of aluminum oxide films (a) without annealing and with annealing at (b) 600°C, (c) 700°C and (d) 800°C.

of samples (1 ~ 4) were found to be similar, and the resistance ratios of samples 1 and 3 are shown in Fig. 6. The TCR (temperature coefficient of resistance) values of samples 1 and 3 were 2,712 ppm/°C and 2,685 ppm/°C, respectively, which are much different from that (3,920 ppm/°C) of bulk Pt. The cause of the poor TCR values was the influence of the impurities originating from the sputtering system which was used for other materials in addition to Pt. The poor TCR would be improved if an exclusive sputtering system for Pt is used and the annealing treatment is carried out at a higher temperature.[10] Resistance ratios of Pt microheater resistors due to self-heating are shown in Fig. 7. At the same heating power, the heater with a smaller active area has a larger resistance ratio than one with a larger active area for reducing external thermal loss.

3.3 *Thermal characteristics of Pt microheaters*

Figure 8 shows thermal characteristics in relation to the heating power of Pt microheaters annealed at 600°C for 60 min. Similar to the results in Fig. 7, the Pt microheater with the smaller active area had better thermal characteristics because it had less convection of air and conduction of the Si substrate which causes the external thermal loss. The Pt microheater which was fabricated with an active area of 200 μm × 200 μm emitted heat to 400°C with the heating power of 1.5 W and the thermal characteristics were superior to those of previous Pt microheaters which were fabricated using the dielectric membrane

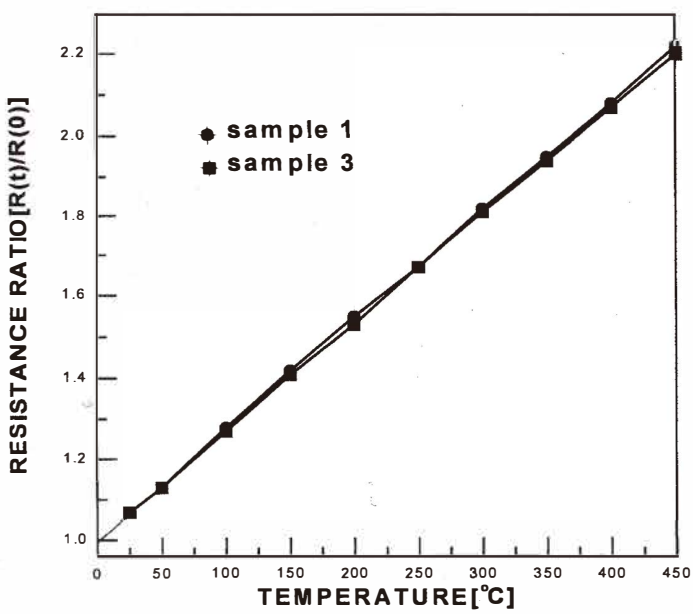


Fig. 6. Resistance ratios of Pt-RTD with increasing temperature.

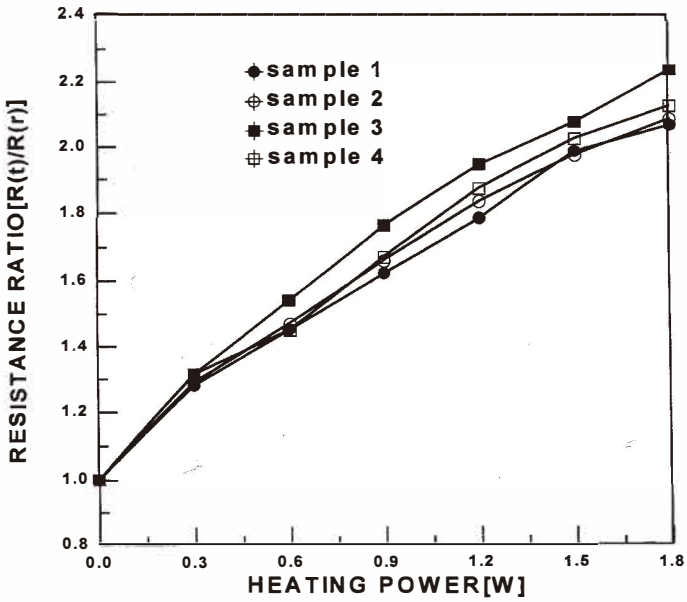


Fig. 7. Resistance ratios of Pt microheaters on aluminum oxide with increasing heating power.

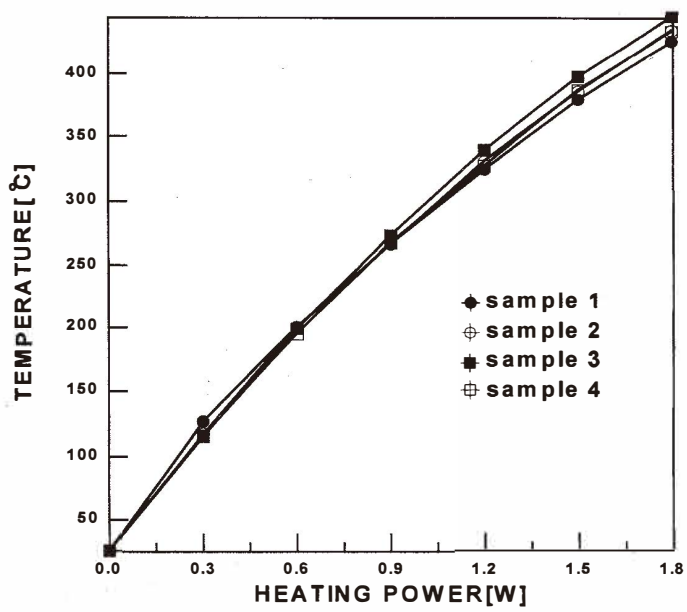


Fig. 8. Thermal characteristics of the Pt microheater fabricated on aluminum oxide films.

structure and Cr as the adhesion layer (heating power: 1.7 W, heater temperature: 420°C). The superior characteristics of our device were a result of the reduction of external thermal loss due to the minimization of the device size, and the typical characteristics of the Pt material were maintained using dielectric aluminum oxide films as a medium layer during the annealing treatment at high temperature. On the other hand, the thermal characteristics of sample 4 , which had the smallest active area of 160 μm × 160 μm, did not fulfill our expectations due to the structural and geometrical problems of the microheater and the RTD.

4. Conclusion

In this study, the electrical and physical characteristics of aluminum oxide films and Pt thin films, deposited by reactive sputtering and DC magnetron sputtering, respectively, were analyzed in relation to the annealing temperature. The properties of microheaters and RTD were also analyzed.

Aluminum oxide films deposited by reactive sputtering in oxygen-argon mixtures improved Pt adhesion and insulation without undergoing any chemical reactions with Pt thin films below the annealing temperature of 600°C. However, the film properties were degraded above 600°C due to the imperfect combinations of Al and O_2 during reactive sputtering. In the analyses of the thermal characteristics of Pt microheaters using Pt-RTD fabricated on the same substrates, the Pt microheater with the smaller active area had the better thermal characteristics due to the lower thermal loss caused by the heat convection of air and the conduction of the Si substrate. The fabricated Pt microheater with the active area of 200 μm × 200 μm emitted heat to 400°C with the heating power of 1.5 W. The degradation of aluminum oxide characteristics over the annealing temperature of 700°C will be improved by modifying the deposition conditions, such as increasing the substrate temperature, varying the input power and adopting the proper ratio of Ar to O_2. If the bridge structure of the dielectric membrane for the thermal isolation of the device from external conditions were applied, the thermal characteristics of the microheater would be further improved.

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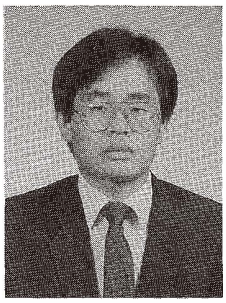
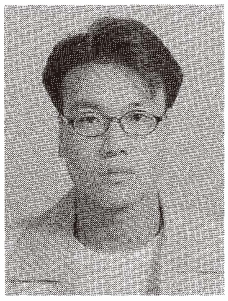


Gwiy-Sang Chung received his B.E. and M.E. degrees in Electronic Engineering from Yeungnam University, Kyongsan, South Korea, in 1983 and 1985, respectively, and his Ph.D. degree from Toyohashi University of Technology, Toyohashi, Japan, in 1992. He joined the Electronics and Telecommunications Research Institute (ETRI), Taejon, South Korea, in 1992, where he worked on silicon-on-insulator materials, MOS devices and microtransducers. He is now an assistant professor in the Division of Electronics and Mechanical Engineering, Dongseo University, Pusan, South Korea. His research interests include silicon-on-insulator materials and MOS devices, power devices, microsensors and microactuators. Dr. Chung is a member of the Korea Institute of Telematics and Electronics, Korean Sensors Society and the Institute of Electronics, Information and Communication Engineers in Japan.



Sang-Soo Noh received his B.E. and M.E. degrees in Electronic Engineering from Dongseo University, Pusan, South Korea, in 1996 and 1998, respectively. He is currently pursuing his Ph.D. degree in Material Engineering from Pusan National University, Pusan, South Korea. His research interests include design, materials and technical development of microsensors and microactuators.