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Characteristics of Chromium Nitride Thin-Film Strain Gauges

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The physical, electrical and piezoresistive characteristics of chromium nitride (CrN) thin films on silicon substrates have been investigated for use as strain gauges. The thin-film depositions were carried out by DC reactive magnetron sputtering in an argon-nitrogen atmosphere (Ar-(5~25%)N_2). The deposited CrN thin films with a thickness of 3500 Å and annealing conditions of 300°C for 48 h in Ar-10% N_2 deposition atmosphere have been selected as the ideal piezoresistive material for the strain gauges. Under optimum conditions, the CrN thin films for the strain gauges have a high electrical resistivity, $\rho = 1147.65\ \mu\Omega$ cm, a low temperature coefficient of resistance (TCR) = –186 ppm/°C and a high temporal stability with a good longitudinal gauge factor, $GF = 11.17$.

1. Introduction

As silicon planar processing and micromachining technologies advance, solid-state pressure sensors using the excellent elastic properties of silicon are rapidly developed. These sensors offer high performance, sensitivity and accuracy in combination with small dimensions, low power consumption, good linearity, no hysteresis, good stability, and suitability for batch fabrication.(1) However, the use of silicon is restricted to temperatures below 120°C and low dynamic pressure ranges.(2)

Recently, the demand for pressure sensors with wide dynamic pressure ranges which can be used in environments of high temperature, pressure, humidity and vibration has increased in automotive, aircraft-engine, industrial and laboratory pressure-sensing instruments.(3) Pressure sensors are electromechanical devices with a variety of applications. A pressure sensor essentially consists of a diaphragm which undergoes deformation due to applied pressure. This mechanical deformation of the diaphragm is converted into an electrical response by strain gauges bonded to or piezoresistors diffused into it.

To overcome the shortcomings of silicon, thick films and metal alloys are used as materials for high-temperature or corrosive applications.(4–9) However, they have low sensitivity because of their low gauge factor and low electrical resistivity. They are difficult to miniaturize. Conventional diffused semiconductor strain gauges have a high gauge factor and high electrical resistivity, but their use is limited above 120°C. Other semiconductor materials such as polysilicon,(10) diamond,(11) and SiC(12) have been developed as strain gauges which can be used under high-temperature conditions, but they are expensive to produce.

In order to develop strain gauges with wide dynamic pressure ranges which can be used in harsh environments, thin-film strain gauges have been investigated. Compared with conventional sensors, these devices have advantages such as a wide temperature range, excellent thermal compatibility with their substrates, and long-term stability, and they are suitable for manufacturing miniaturized sensors with high internal resistance. In addition, they are characterized by precisely controlled technology and the advantage of batch manufacture. Distinct advantages are the absence of adhesive material, and flexibility in tailoring the properties of the sensing film.

Several ceramics have comparatively higher electrical resistance, stress sensitivity and gauge factors than metals. It is possible to use them at high temperatures, and they are also available as materials for thin-film strain gauges.(13)

In this paper, we describe the physical, electrical and piezoresistive characteristics of CrN thin films on silicon substrates for use as strain gauges. The CrN thin films are deposited by DC reactive magnetron sputtering in an argon-nitrogen atmosphere (Ar-(5~25 %)N_2). In order to improve the piezoresistive properties of the sensing element, deposition parameters and postdeposition thermal treatments were varied. The thin-film deposition and masking methods for the development of a sensing element based on piezoresistive CrN thin films are also presented. Finally, under optimum deposition and annealing conditions, this study examines the I (current)/V (voltage)/T (time) curves of CrN thin-film strain gauges, hysteresis characteristics for changes in temperature and resistance, and the effect of aging on the ceramic thin-film strain gauges.

2. Experimental

CrN thin films were deposited onto thermally oxidized 500-μm-thick silicon substrates by DC reactive magnetron sputtering in an argon-nitrogen atmosphere (Ar-(5~25 %)N_2). Prior to the deposition, the silicon substrates were cleaned in an ultrasonic degreasing bath in a clean-room environment. The purity of the metallic Cr target with a 2-inch diameter was 99.9%. The residual gas pressure was less than 5×10^{-6} Torr and the total gas pressure of the Ar-N_2 mixture during CrN deposition was held constant at 0.9 Torr. The Ar gas flow was 60~76 sccm and the N_2 gas flow was 4~20 sccm. Deposition rates from 350 to 400 Å/min were achieved. A 7 W/cm^2 rf bias was applied to the substrate during the deposition. Thin films between 1500 Å and 5000 Å in thickness were measured using a profilometer. A post-deposition thermal treatment (temperature: 100~300°C, time: 24~72 h) in an N_2 atmosphere was also carried out to investigate the effect of annealing on the CrN thin-films. The structural and compositional properties of the CrN thin-films were evaluated by scanning electron microscopy (SEM), X-ray diffraction (XRD) and electronic diffraction spectroscopy (EDS). All the electrical measurements were obtained by the four-point probe method.

The CrN thin-film resistors, 35 μm wide with a 32-mm-long meandering path, were patterned using photolithographic techniques. An automatic data-acquisition system controlled by a personal computer was used for the TCR measurement in the range 25~150°C. Accelerated life tests at 150°C were performed to study the long-term stability of the CrN thin-film resistors. I/V/T characteristics were used to analyze the electrical conduction mechanism of the CrN thin-films. The hysteresis effect due to strain cycling was also examined.

The sensitivity of a strain gauge is expressed by a dimensional number called the gauge factor, which is given by $GF = \Delta R/R/\varepsilon$,where R is the initial resistance of the gauge and ΔR the change in the resistance due to the strain ε. The longitudinal (direction of current and strain parallel) gauge factor of the CrN thin-films was determined using the cantilever beam method.(6) The set-up used was located in a vibration-free air-conditioned room, in order to ensure that error due to vibrational strains and ambient temperature fluctuation would not interfere with the measurements. A high-speed steel beam was mechanized. Connecting leads are bonded to the gauge using quick drying silver paste. The strain was applied by adding precision weights at the free end. The relative change in resistance was measured using a digital multimeter with four probe and zero-setting arrangements. The value of strain ε was calculated using the relation $\varepsilon = 6WL/Ebd^2$, where E is Young's modulus for the materials of the beam, b the breadth of the beam, d its thickness, W its linear specific weight, and L the distance from the centre of the gauge to the point of application of load W.

3. Results and Discussion

3.1 *Electrical properties*

Electrical measurements were performed after the annealing treatments. Figures 1 and 2, respectively, show the variations of electrical resistivity (ρ) and TCR as a function of the Ar:N_2 flow ratio during the DC reactive magnetron sputtering and annealing.

Figure 1 shows the variations of electrical resistivity with post deposition thermal treatment conditions of CrN thin-films (heating temperature: 100~300°C and heating time: 24~72 h) when the flow rate of N_2 gas is 4~16 sccm. Before annealing, as the N_2 flow rate increased, the electrical resistivity of the CrN thin films increased. At the N_2 flow rate of 20 sccm, Cr underwent nitrification and then became insulated. When the N_2 flow rate was low, the Cr almost never became nitrified. As the N_2 flow rate increased, Cr which had been prepared

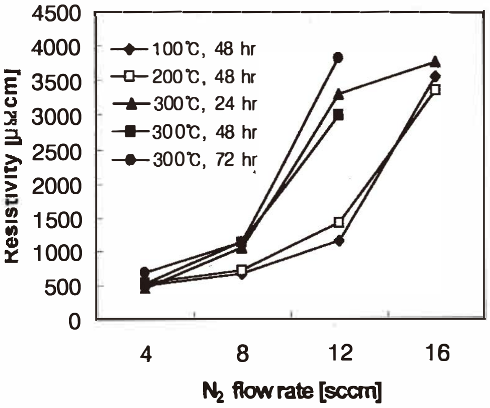


Fig. 1. Variations of electrical resistivity according to annealing conditions of CrN thin film (temperature: 100~300°C, time: 24~72 h).

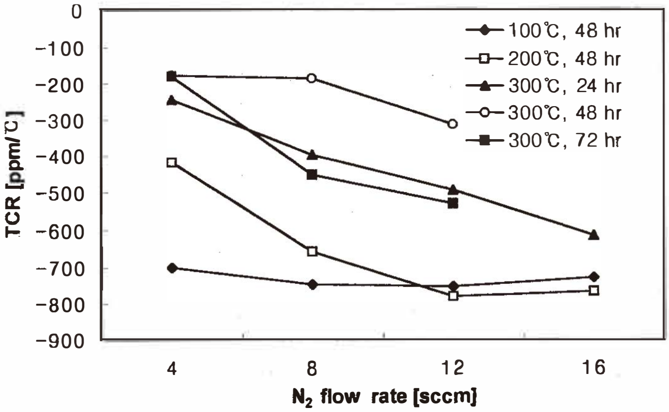


Fig. 2. TCR values according to annealing conditions of CrN thin-film strain gauges (temperature: 100~300°C, times: 24~72 h).

in a metal phase by the sputtering method proceeded with nitrification. Consequently, the high electrical resistivity seemed to control the characteristics of the thin films.(14) When the annealing temperature was increased, there was almost no variation of electrical resistivity values at N_2 flow rates under 8 sccm, but electrical resistivity values increased at N_2 flow rates above 12 sccm. In particular, at the N_2 flow rate of 16 sccm with annealing at 300°C for 48 h, wide variations of electrical resistivity were observed. It appears that oxidation occurred upon the annealing of nitrified CrN thin-films.

Figure 2 shows TCR values according to the annealing conditions of CrN thin-film strain gauges for different N_2 flow rates. As the annealing temperature increased, the TCR values decreased. When the N_2 flow rate was 4~8 sccm, the variation was the greatest, that is, the lowest TCR values appeared to be reached at a N_2 flow rate of 4 ~ 8 sccm, the annealing temperature of 300°C and the annealing time of 48 h. When the N_2 flow rate was more than 16 sccm, with an annealing temperature of more than 300°C, the TCR could not be measured because the resistance increased. Therefore, a comparatively high electrical resistivity value of 1147.65 $\mu\Omega$cm and a low TCR value of −186 ppm/°C correspond to a N_2 flow rate of 8 sccm, an annealing temperature of 300°C, and an annealing time of 48 h.

Figure 3 shows the hysteresis characteristics of the variation rate of resistance according to the variation of temperature of CrN thin-film strain gauges in the temperature range of 25~150°C when the N_2 flow rate is 8 sccm and the annealing conditions are a temperature of 300°C and a time of 48 h. The figure shows a nonlinearity and a hysteresis of less than 1.65% full scale and 2.27% full scale, respectively. The TCR shows high linearity and low hysteresis under these conditions. The variation rate of resistance according to temperature was very linear, and its characteristics seemed to become more stable with annealing time.

Figure 4 shows the long-term stability over time for an accelerated life test of CrN thin-film strain gauges at 150°C, when the N_2 flow rate is 8 sccm and the annealing conditions are a temperature of 300°C and a time of 48 h. Owing to the increase of ambient temperature to 150°C in 30 min, the initial resistance variation rate of the CrN thin-film strain gauges is increased. However, the resistance variation rate at a constant temperature of 150°C after 30 min is very small for CrN thin-film strain gauges, $\Delta R/\Delta t = \pm 6$ ppm/h. This implies that the temporal stability is good.

Figure 5 shows I/V/T characteristics of CrN thin films deposited with an N_2 flow rate of 8 sccm and annealed at 300°C for 48 h. The resistance remains constant during the test. This phenomenon is in agreement with a metallic conduction mechanism. The high resistivity and the negative TCR values of CrN thin films indicate that the conduction electron mean free path is very small.(15) It is believed that the high scattering is caused by a large amount of disorder due to the amorphous structure of these CrN thin-films. The metallic conduction mechanism indicates the existence of a continuous metallic phase.

3.2 *Physical properties*

Figure 6 shows SEM micrographs of CrN thin films in the annealing temperature range from 100 to 300°C, when the N_2 flow rate is 8 sccm. In the temperature range from 100 ~ 200°C, there was no variation, and at 300°C crystal grains were formed by increasing the annealing temperature; the border of the particles in the CrN thin films was remarkable. The station was electronically unstable, but the gaps in the structure appear to have closed. Consequently, the values of electrical resistivity of CrN thin-films and the TCR became stable, and the physical and electrical characteristics of CrN thin-films were improved by annealing.

Figure 7 shows SEM micrographs of CrN thin films for various annealing times with an N_2 flow rate of 8 sccm and an annealing temperature of 300°C. As the annealing time increased, the border of the particles in the CrN thin films became clearer. The film was

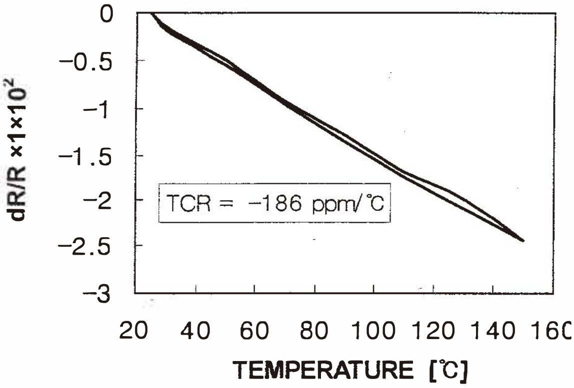


Fig. 3. Variation rate of resistance according to temperature of CrN thin-film strain gauges (N_2 flow rate: 8 sccm, annealing conditions: 300°C, 48 h).

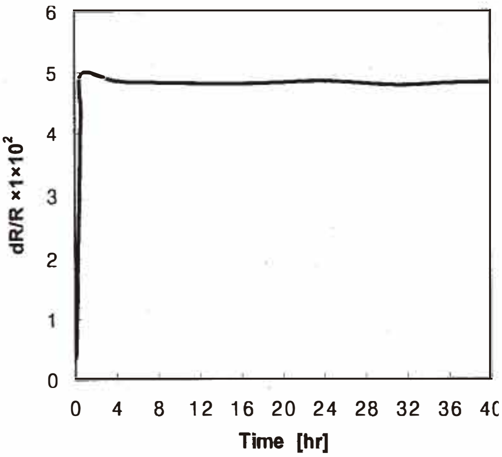


Fig. 4. Effect of aging on CrN thin-film strain gauges (N_2 flow rate: 8 sccm, annealing conditions: 300°C, 48 h).

electrically unstable, but the gaps in the structure appear to have closed. As the number of crystal grains increased, they conglomerated. However, no islands formed, even with annealing at 300°C for 72 h.

Figure 8 shows XRD patterns of CrN thin films for various annealing conditions in which the N_2 flow rate is 8 sccm. The peak values presented are for Cr crystal, which did not yield high peak values with nitrification. As the annealing temperature and time

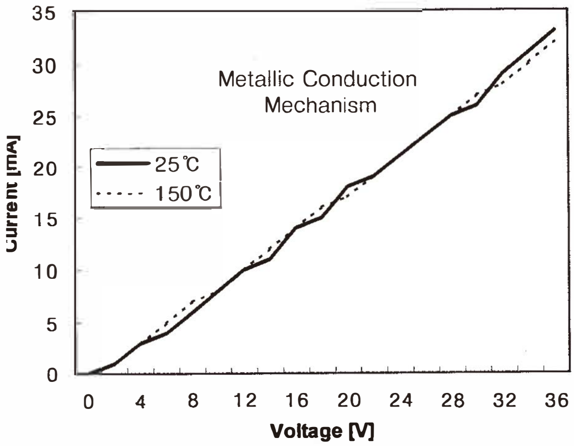
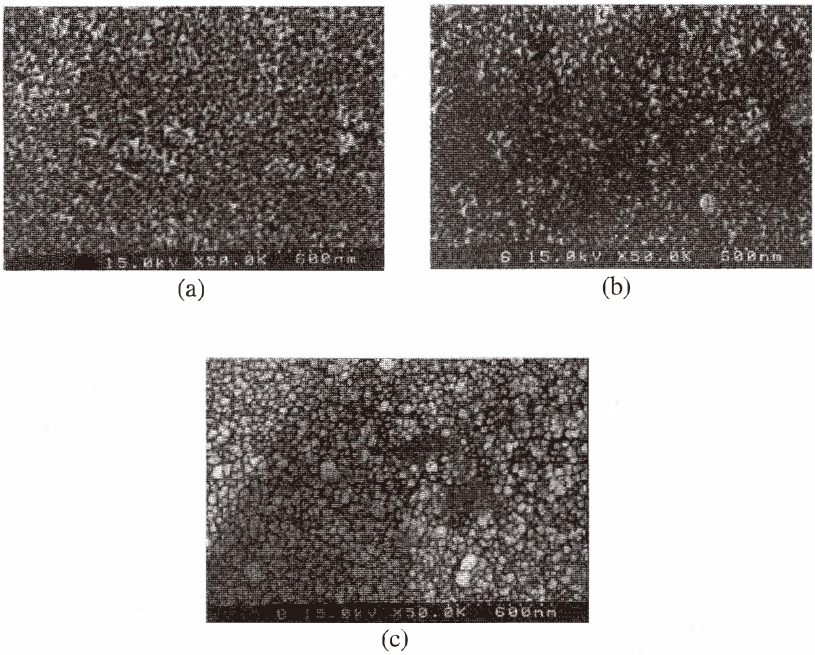


Fig. 5. I/V/T characteristics of CrN thin-films (N_2 flow rate: 8 sccm, annealing conditions: 300°C, 48 h).

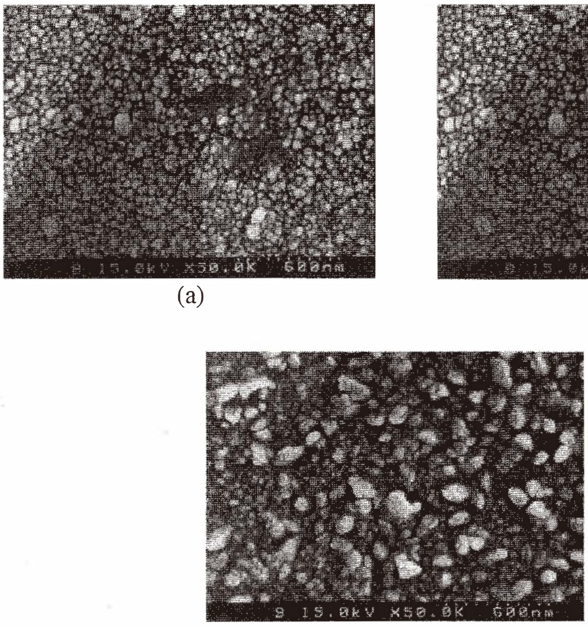


(a)

(b)

(c)

Fig. 6. SEM micrographs of CrN thin-films with various annealing temperatures: (a) 100°C, (b) 200°C and (c) 300°C (N_2 flow rate: 8 sccm, annealing time: 48 h).



(a) (b)

(c)

Fig. 7. SEM micrographs of CrN thin-films with various annealing times: (a) 100°C, (b) 200°C, and (c) 300°C (N_2 flow rate: 8 sccm, annealing temperature: 300°C).

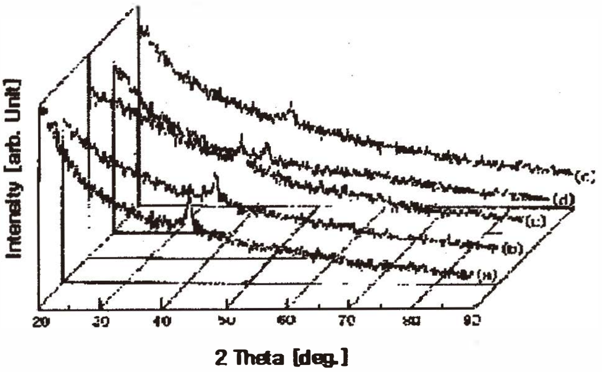


Fig. 8. XRD patterns of CrN thin films with various annealing conditions: (a) 100°C, 48 h; (b) 200°C, 48 h; (c) 300°C, 24 h; (d) 300°C, 48 h; (e) 300°C, 72 h.

increased, the peak value exhibited no significant variation. The metal phase became smaller, and nitride was formed. The results suggest that the metal phase is kept and a mixture of phases exist in an amorphous structure.

3.3 *Piezoresistive properties*

Figure 9 shows variations of the longitudinal gauge factor of CrN thin-film strain gauges according to annealing conditions. As the annealing temperature and time increased, the gauge factor remained stable without significant variation. As N_2 flow rate increased, the gauge factor of CrN thin-film strain gauges increased. Annealing does not appear to affect the sensitivity of strain gauges.

Figure 10 shows the response characteristics of the longitudinal gauge factor according to the stress on the CrN thin-film strain gauges, which were fabricated with an N_2 flow rate of 8 sccm and annealing conditions of 300°C and 48 h. A longitudinal gauge factor of 11.17 is obtained under these conditions. This value is slightly higher than the theoretical predictions for a piezoresistive metallic thin film.(16) Significant linearity in the piezoresistive behaviour is observed. These characteristics are suitable in a pressure-sensing element. Under optimum deposition and annealing conditions, the variation rates of resistance of CrN thin-film strain gauges changed almost linearly according to the externally supplied stress.

4. Conclusions

The physical, electrical and piezoresistive characteristics of CrN thin films on silicon substrates for use as strain gauges were investigated. The CrN thin films were deposited by DC reactive magnetron sputtering in an argon-nitrogen atmosphere (Ar-(5~25 %)N_2). The

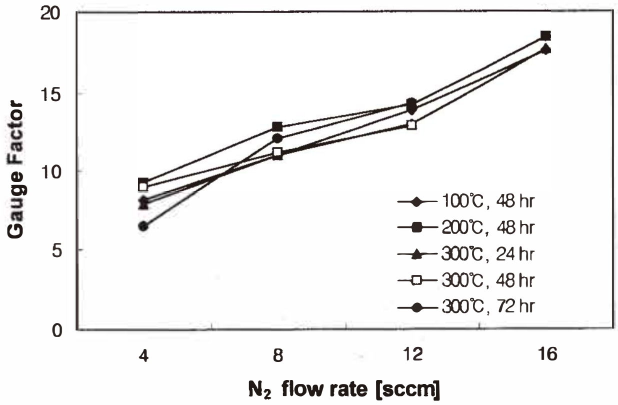


Fig. 9. Variations of gauge factor for various annealing conditions of CrN thin-film strain gauges (temperature: 100~300°C, time: 24~72 h).

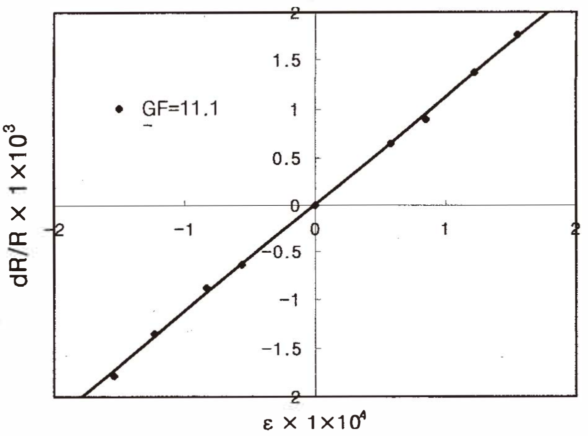


Fig. 10. Response characteristics of longitudinal gauge factor according to stress of CrN thin film strain gauges (N_2 flow rate: 8 sccm, annealing conditions: 300°C, 48 h).

sputtering and annealing conditions have been defined in order to optimize the piezoresistive properties of the sensing element. Optimum conditions for the fabrication of CrN thin-film strain gauges were a thickness of 3500 Å and annealing conditions of 300°C and 48 h, with an N_2 flow rate of 8 sccm. CrN thin films for strain gauges were obtained with an electrical resistivity of 1147.65 $\mu\Omega$cm, TCR = −186 ppm/°C and a longitudinal gauge factor of 11.17 under optimum conditions. A metallic conduction mechanism has been identified in the deposited CrN thin-films. These properties of CrN thin-film strain gauges make them very suitable for application as mechanical sensors.

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