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Magnesium Oxide Films as Temperature Sensor

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Mg oxide thin films, with thermal sensitivity superior to Pt thin films, were fabricated through annealing of Mg films deposited by r.f. magnetron sputtering. The annealing was carried out in the temperature range of 500-700 °C under atmospheric conditions. Resistivity of the resulting Mg oxide films were in the range of $1.7\text{x}10^7$ μΩ-cm/°C to 2.81×10^{12} μΩ-cm/°C, depending on the extent of Mg oxidation. The temperature coefficient of resistance (TCR) of the MgO films also depended on the extent of Mg oxidation. The average TCR of MgO resistors, measured between 0 and 200°C, were $7630\text{x}10^2$ppm/°C for the 500°C and $2988\text{x}10^2$ppm/°C for 650 °C respectively. Because of their high resistivity and linear TCR, MgO thin films are superior to pure Pt thin films for flow and temperature sensor applications.

Key Words: Mg oxide, Thermal sensitivity, Resistivity, TCR, Flow sensors

INTRODUCTION

Inexpensive and reliable temperature measurement are important for various applications, including environmental monitoring and control, indoor air conditioning, weather forecasting and automotive and aerospace systems [1-3]. Effort has been made to develop such sensors using thin film technology [4-5]. In general, temperature sensors are used together with flow sensors in order to obtain more accurate data. There are some sensors based on mechanical elements [6-8], but flow sensors based on a thermal mechanism have been studied and developed over the years [5]. Various designs have been examined to improve the properties of such sensors. Flow sensors using a thermal mechanism and resolution is dependent on the sensing material's TCR and resistivity, while accuracy depends on linearity of resistance variation with temperature. Polysilicon films has been extensively investigated as the thermal element need in these sensors, but its temperature response is very non-linear for doped films, making it difficult to attain the required wide temperature range of operation at desired accuracy [2-3]. Pt thin films resistors have been studied for flow sensing and few cases on thin bridge micromechanical structures formed by silicon micromachining were also investigated[1]. However Pt resistors, generally used in temperature sensors, are superior to Mg resistors because of their linearity of response but not resolution. So for temperature or flow sensing applications, Mg is better than Cu and Pt due to its higher TCR In light of these properties, Mg oxide is expected to be superior to Pt [9-10] and Mg as a material for flow sensors application. In present study, Mg thin films were deposited and then oxidized followed by annealing in atmospheric conditions. Temperature dependent resistivity and TCR of the annealed MgO thin films were investigated as a function of the oxidation extent. Finally, the potential of Mg oxide thin films for flow sensors was examined using resistors made by laser processing [9].

EXPERIMENTAL

Mg films were deposited using a circular (4 in.-diameter and 0.20 in.-thick) Mg target in a r.f. magnetron sputtering system. The deposition conditions for Mg thin films are summarized in table1. A high-purity quartz substrate with surface roughness less than 0.1μm and a thickness of 0.60 mm was used. After the working chamber was evacuated to below 5×10^{-7} Torr, high-purity (99.999%) Ar gas was introduced into the chamber. Mg input power density was kept at 6.5 W/cm^2 and working pressure was kept at 5.5 mTorr. Films were oxidized for 4hr at annealing temperatures in the range of 500-700 °C. Thickness of the deposited films was measured with α-step, and the sheet resistance was measured by a conventional Vander Pauw four-point probe. The oxidation extent of Mg thin films were analyzed by energy dispersive X-ray spectrometer (EDX). X-ray diffraction (XRD) patterns were obtained directly from the Mg oxide thin films in a conventional θ-2θ configuration between 30° and 80° using Cu Kα radiation. In order to measure TCR, Mg oxide resistors with a resistance of 1 kΩ at 0°C were patterned by a laser processing system, with remaining processes for packaging such as wire-bonding and passivation done subsequently[10]. Resistances were measured in the temperature range of 0-200°C using a 4-wire resistance measuring method by applying a 1 mA dc current to the MgO resistors and measuring the voltage with a highly accurate digital multimeter. The TCR was then calculated by following relation

$$TCR = (\Delta R/R_0).\ \Delta T\ (ppm/^0C)$$

Where R_0 is the resistance value at 0 °C, ΔR the resistance change with respect to 0°C resistance and ΔT is the change in temperature.

RESULTS AND DISCUSSION

The resistivity of the Mg thin films was found to be a function of annealing temperatures as seen in Fig 1. It increased from $9.5x10^7$μΩ-cm after annealing at 500°C to 2.81×10^{12} μΩ-cm after annealing at 700°C. However the resistivity of Mg thin films decreased somewhat after annealing at 500°C and 550°C because of the stabilization and improvement of the Mg thin films due to annealing. Mg films were dramatically changed into Mg oxide films at annealing temperatures of 650°C and above. Accordingly, the resistivity of the films at 650°C was 7.008×10^{10} μΩ cm, higher than $1.7\text{x}10^{7}$ μΩ cm at 500°C. The resistivity of the films annealed at temperature above 650°C was too high to be useful as a sensing material. The observed resistivity variations are explained by XRD analysis shown in Fig 2. X-ray diffraction results for different annealing temperatures are shown in Fig 3. Below 600°C, Mg crystallinity increased with annealing temperature, but over 600°C, Mg crystallinity decreased dramatically, while MgO_x crystallinity increased relatively. At higher than 750°C, metallic Mg nearly disappeared and MgO_x crystallinity increased sharply. These results indicate that the oxidation of Mg thin films accelerated over 600 °C and explain nicely the resistivity variations for different annealing temperature. (Fig 1)

Table 1

	Condition
Target	Pure Mg (99.999%) 4 in.
Power density	6.5 W/cm^2
Base pressure	5×10^{-7} Torr
Working pressure	5.5 mTorr
Substrate	SiO_2 (100) (pure), 0.60 mm (*t*)
Substrate temperature	23 °C

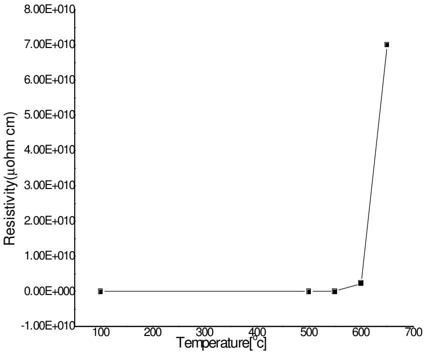

Fig 1. Resistivity variations of Mg thin films with increasing oxidation-annealing temperatures.

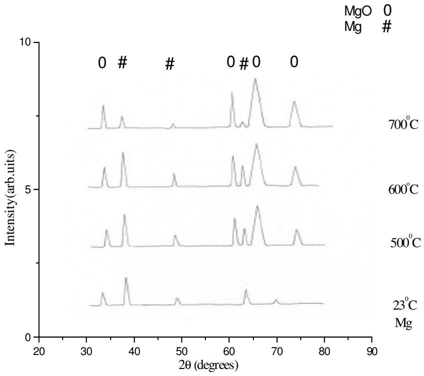

Fig 2. XRD patterns of Mg thin films after different annealing temperatures.

Fig 3. shows the oxidation extent of Mg thin films according to annealing temperature. The wt.% O in Mg thin films was less than 2.55 before annealing, but the values were 26.77 and 43.78 after annealing at 650°C and 700°C (for 5 hr), respectively. This result is explained by the fact that oxidation rate increases with increasing annealing temperature. The oxidation rate may also increase if annealing time is increased more than 5 hr at 550°C or above. Resistors were made from the Mg oxide films. TCR values from 0°C to 200°C were measured and are shown in Fig 4. With increasing wt.% O in the films, TCR decreased but the linearity of resistance variation improved. For500°C and 650°C annealed films, the average of TCR values, measured were 7630 x 10^2 and 2988 x10^2 ppm/°C, respectively for the operating temperature range of 0-200°C. However, the resistance variation was far more linear in the latter case. Considering factors such as resistivity, TCR and linearity, the Mg oxide films from 600°C oxidation annealing had the best properties for application in thermally based micro flow sensors. Using these Mg oxide thin films, resistors of 1 kΩ at 0°C were fabricated by laser process technology; their long-term resistance stability was tested at 150°C in air for a continuous 45-day period, with the results presented in Fig 5. The resistance increased somewhat in the first 10days because the films were exposed to oxidative condition for a long period, even though 150°C is a low temperature for Mg oxidation. Particularly, the resistor sidewalls formed by the laser etching are prone to additional oxidation. Once this effect saturates the rate of increase in resistance decreases after 10 days. The films showed no resistance variation at room temperature in air for almost 45 days.

Conclusions

Mg thin films were deposited for application to micro flow sensors by a magnetron sputtering and oxidized through annealing at 500-700°C under atmospheric conditions. The characteristics of the resulting Mg oxide thin films were investigated. Mg oxide thin films from annealing at 600°C for 5 hr had 12.34 wt.% O. The long-term resistance stability of the resistors, made from Mg oxide thin films, at 200°C for 45 days was found to be good. Because of the high TCR and resistivity characteristics, Mg oxide thin films are superior to Ni and Pt thin films for micro flow sensors operating at temperatures below 250°C. In future, further experiments and analysis of annealing from 600°C to 650°C can be used to optimize the films for micro flow sensors.

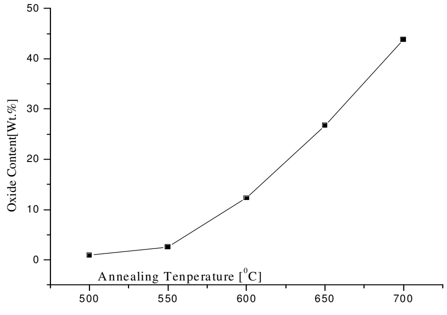

Fig 3.Variations of wt.% O in Mg thin films with increasing oxidation-annealing temperatures.

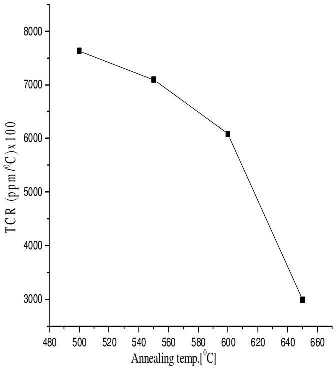

Fig. 4.TCR variations in Mg thin films with increasing oxidation-annealing temperatures.

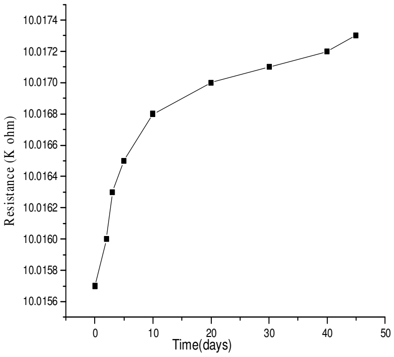

Fig. 5. Long-term resistance stability data at 150 °C for 45 days.

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