Effect of annealing temperature on the structural and electrical properties of titanium nitride film resistors

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Abstract: Titanium oxy-nitride (TiN₂O₂) thin films were deposited on SiO₂/Si substrates using reactive dc magnetron sputtering, and were then annealed at various temperatures in air ambient to incorporate oxygen into the films. The effect of annealing temperature on the structural and electrical properties of the films was investigated. The grain size of the films decreases with increasing annealing temperature. On the other hand, crystallinity of the films is independent of annealing temperature in air ambient. Resistivity of the films increases remarkably as an annealing temperature increases and temperature coefficient of resistance (TCR) of the films varies from a positive value to a negative value. The films annealed at 350 °C for 30 min exhibited a near-zero TCR value of approximately -5 ppm/K. The decrease of the grain size with increasing annealing temperature was attributed to an increase of oxygen concentration incorporated into the films during annealing treatment.

1. Introduction

Thin film resistors of copper-nickel (CuNi),1 nichrome (NiCr),2 and tantalum nitride (Ta₂N),1 were commonly used for microelectronics, portable terminal Π-type attenuators, and telecommunication devices. However, the materials such as CuNi, NiCr or Ta₂N have a drawback, i.e., their rather low resistivity. Hence, these materials are not suitable for applications in thin film resistor field where the high resistivity and low temperature coefficient of resistance (TCR) of thin film resistors are required. In this work, we will develop high resistivity TiN₂O₂ material with low TCR. The structural and electrical properties of the films are carefully investigated as a function of annealing temperature.

2. Experimental procedure

Titanium oxy-nitride thin films about 300 nm thicknesses were deposited at room temperature (without intentional heating the substrate) and a nitrogen-argon flow ratio of 5% by reactive dc magnetron sputtering with a titanium target. After deposition, the samples are annealed at various temperatures in air ambient for 30 minutes in order to incorporate oxygen atom into the films for purpose of increasing resistivity. The crystal structure and preferred orientation of the films were determined by X-ray diffraction measurement. The oxygen concentration incorporated into the TiN₂O₂ films was analyzed by Rutherford backscattering spectroscopy (RBS). The sheet resistance of the films was measured at room temperature with an electrometer (CMT-5R 1000) using the standard four-probe. The TCR of the sample was measured in the temperature range of 25 ~ 120 °C in a thermostatically controlled oven using a digital multimeter (HP3458A).

Table 1: Deposition conditions of tantalum nitride

<table>
<thead>
<tr>
<th>Substrates</th>
<th>SiO₂(600nm)/ Si (100)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target material</td>
<td>Titanium</td>
</tr>
<tr>
<td>Base pressure</td>
<td>8x10⁻⁶ torr</td>
</tr>
<tr>
<td>Working pressure</td>
<td>4 x 10⁻³ torr</td>
</tr>
<tr>
<td>D.C power of Ta target</td>
<td>100 w</td>
</tr>
<tr>
<td>Ar + N₂ gas flow rate</td>
<td>10 sccm</td>
</tr>
<tr>
<td>N₂/(Ar+N₂) flow ratios</td>
<td>5%</td>
</tr>
<tr>
<td>Annealing temperature</td>
<td>Room temperature ~ 500 °C</td>
</tr>
</tbody>
</table>

3. Results and discussion

Fig. 1 shows the XRD patterns of the films annealed at temperatures from room temperature to 500 °C. As-deposited films as well as the films annealed at various temperatures exhibited a single TiN₂O₂ phase with a (111) preferred orientation. The intensity of (111) diffraction peak became weaker and a small shift of (111) peak towards the high angle was observed as annealing temperature increases. The grain size decreased with increasing annealing temperature, as shown in the inset of Fig.1.

Figure 2 shows the RBS spectrum for the samples as-deposited at room temperature and annealed at 350 °C and 500 °C in an air ambient. Corresponding values of the ratios of (TiN, TiO), (x, y), determined with the backscattering yields are (x = 1.12, y = 0.3) for as-deposited samples, (x = 1.08, y = 0.41) for samples annealed at 350 °C, and (x = 0.85, y = 0.77) in case of 500 °C.
Fig. 1. XRD patterns of the films annealing at various temperatures.

Fig. 2. Variation of the resistivity as a function of annealing temperature.

The dependence of resistivity of the films on annealing temperature is shown in Fig. 3. The resistivity of the films drastically increased with increasing annealing temperature. The increase of the resistivity is due to the increase of nitrogen concentration in the films during annealing treatment.

The variation of TCR value of the films is shown in Fig. 4. The results indicated that the TCR strongly depends on an annealing temperature. The TCR of the films as-deposited at room temperature is approximately 55 ppm/K. However, as annealing temperature increases from room temperature to 500 °C, the TCR changes from a positive value (55 ppm/K) to a negative value (-251 ppm/K). Typically, a near-zero TCR value of about -5 ppm/K can be obtained from the samples annealed at 350 °C for 30 min. from RBS data, the composition of the films is TiN$_{1.06}$O$_{0.41}$.

Fig. 3. TCR of tantalum film as a function of nitrogen/argon ratio.

Fig. 4. AES depth profiles of Ta$_2$N thin film deposited at 200°C.

4. Conclusion

The structural and electrical properties of TiN$_x$O$_y$ thin film resistors were investigated as a function of annealing temperature in air ambient. The film annealed at 350 °C shows a near-zero TCR value of about -5 ppm/K and a quite high resistivity of approximately 5000 $\mu$-ohm-cm. The results suggest that titanium oxy-nitride annealed at 350 °C can be well apply in fabricating thin film resistors.

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Reference