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Stable temperature coefficient of resistance in TiSiON thin film resistors deposited by magnetron co-sputtering

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ABSTRACT

Thin films of titanium/silicon (Ti:Si) are widely employed in the electronics industry because of their metal-like characteristics. The use of films as thin film resistor (TFR) based on Ti:Si has not yet been reported. In this paper, the TFR characteristics of TiSiON with different compositions and deposited in Argon atmosphere with either 1% Oxygen or 1% Oxygen/3% Nitrogen are studied. The film composition was varied through a co-sputtering approach from titanium and silicon targets and the sheet resistance (Rsh) and temperature coefficient of resistance (TCR) were evaluated. The film composition was evaluated by X-ray photoelectron spectroscopy (XPS); carrier mobility, type and concentration by Hall effect methods and film microstructure by X-Ray diffraction. Nitrogen addition during the deposition reduces oxidized species in the TFR and increases film stability. The addition of nitrogen results in TFR films with partially oxidized titanium and silicon and TFR values closer to zero TCR without impacting film resistance. Films deposited without nitrogen result in more unstable films and larger TCR. A near-zero TCR film was found for a Ti:Si ratio of 1.6, exhibiting a Rsh at 25 °C of 0.7 kOhms with a TCR value of -171 ppm/°C after annealing at 450 °C in forming gas.

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1. Introduction

Thin film resistors (TFR) are commonly used in integrated circuits and, to ensure stable circuits, their electrical resistance is expected to be essentially independent of temperature. Currently, the most commonly used TFR films are based on complex metal alloys including NiCr and NiCrSi. Single metal systems such as Ta or TaN and cermets containing Cr, Si, B, C and O have also been investigated. TFRs based on NiCr alloys can achieve temperature coefficient of resistance (TCR) values in the range of 250–50 ppm/ °C with sheet resistance <100 Ohmq/sq when varying the Ni composition from 80 to 40 wt% in ~50 nm thick films [1]. Further addition of Si has shown a TCR value of $-153 \text{ ppm}/^{\circ}\text{C}$ with Rsh of 350 Ohms/sq in 30 nm thick films [2]. Closer to zero TCR values have been achieved (<5 ppm/°C) by adding Al and Cu to the NiCr alloy at the expense of the considerably decreasing Rsh to ~10 Ohms/sq [3]. TaN films deposited with 13% N₂ partial pressure have been reported to show a TCR of -200 ppm/°C with Rsh of 4 Ohms/sq in ~675 nm film [4]. More recent work on Ta₂N improved the TCR value of this single metal system to -47 ppm/°C with a similar Rsh at a film thickness of 350 nm [5]. TCR and Rsh in TaN are closely related to the crystallinity of the films; hence, factors such as environment, pressure and temperature during processing need to be closely controlled. Multicomponent TFR materials have been able to achieve TCR values of -325 ppm/°C with a Rsh of 25 kOhm/ sq at a film thickness of 30 nm in materials containing Cr, B, Si and O and even closer to zero TCR value of -6 ppm/°C with Rsh of 1 kOhm/sq at a film thickness of 90 nm when introducing C [6,7].

Electrical resistance and temperature exhibit an opposite correlation in metals and semiconductors. In metals, increasing temperature results in increased phonon scattering of charge carriers, causing a drop in effective mobility and, as a result, the resistivity increases yielding an overall positive temperature coefficient of resistance (TCR). In semiconductors, increasing temperature results in increased carrier concentration and as a result the resistivity decreases yielding an overall negative TCR. Such behavior can be used to develop a TFR film by properly mixing metal and semiconductor materials in the TFR; where the opposing resistance vs temperature behavior can potentially yield a zero TCR with acceptable resistance. The film resistance can then be adjusted by adding a dielectric material such as SiC that can also improve thermal stability [8]. Given these conditions as well as the need of semiconducting, metallic and dielectric materials in the TFR film for proper resistance and TCR a large amount of research is focused on TFR systems comprised of five or more elements/compounds [6,9,10]. This large number of components can prove to be very







complicated when controlling film composition. An alternative approach is based on materials that can be arranged in series or parallel to compensate opposing TCR behavior [11,12]. However, this method imposes the undesirable need of patterning more layers during the fabrication process.

Titanium and silicon are elements commonly used in the microelectronics industry. In particular, titanium silicide has been commonly used as source and drain contact and as interconnect metal in complementary metal-oxide semiconductor (CMOS) technology. Other uses include Schottky barrier contacts and diffusion barriers [13–15]. Traditionally, TiSi films are formed by depositing titanium on silicon followed by an annealing to further react the films and form the corresponding silicide [16]. One advantage of using co-sputtering approaches for silicide deposition is that the silicide stoichiometry can be easily tuned [17]. Moreover, this deposition method allows the use of reactive gasses to introduce additional elements in the resulting Ti:Si films, such as nitrogen and oxygen, resulting in films with variable concentrations of oxygen and nitrogen in the bulk of the films. The use of postdeposition thermal treatments is then used to stabilize the films and achieve uniform and homogeneous films. Titanium silicide is mostly known for its thermal and chemical stability as well as low resistivity [18,19]. However, the low resistivity represents an important challenge when evaluating the feasibility of Ti:Si as TFR where high resistance is desired. Nevertheless, the introduction of nitrogen and oxygen during deposition can be used to further increase film resistivity. An advantage of using Ti and Si is that both of these elements are easily oxidized when deposited in a reactive oxygen atmosphere resulting in films with higher electrical resistance. The present work explores the introduction of oxygen and nitrogen during the deposition of Ti:Si films with different Ti:Si ratios to improve the TFR characteristics of the resulting films. For the film deposition, a pressure of 10 mTorr is used while varying the percentage of oxygen and nitrogen in the plasma. Given the high reactivity of Ti and Si towards oxygen, oxygen was kept at 1% and nitrogen varied from 1.5 to 6%. Argon was used as the base gas for a total flow of 20 sccm; therefore, $%Ar + %O_2 + %N_2 = 100\%$. Silicon and titanium targets with 99.995% purity were used for the depositions. After deposition, the films were capped with a silicon nitride layer to prevent further oxidation from exposure to the ambient. The films resistance and TCR stability were tested by annealing the films up to 450 °C in a forming gas flow.

2. Materials and methods

2.1. Thin film resistor deposition

To evaluate the chemical composition of the TiSiON films the ratio of titanium to silicon in the films was varied by co-sputtering from two different targets: titanium (DC source) and silicon (RF source). The power supplied to the Ti gun was kept constant at 200 W while the power supplied to the Si gun was varied in the range from 30 to 90 W. The targets were 2 inch in diameter and acquired from Kurt J Lesker with a purity of 99.995%. Films were deposited in a commercial sputtering system from AJA International equipped with magnetron guns at the bottom of the chamber at a substrate holder separation of 13.4 cm. The deposition chamber was initially evacuated to a base pressure ~5 $\times~10^{-8}$ Torr and monitored with an ion gauge. The chamber was then back filled with ultra-high purity argon, oxygen and nitrogen to a working pressure of 10 mTorr for film deposition, as measured with a capacitance manometer gauge. All depositions were carried out at room temperature. Intrinsic silicon (100) wafers with 300 nm of thermally grown SiO₂ were used as substrate. Before deposition, the substrates were cleaned with acetone followed by isopropyl alcohol in sonication, rinsed in water and dried with nitrogen gas. To vary the composition of the films, the power for each target was adjusted to achieve films with Ti:Si ratios ranging from 0.5 to 5. After deposition the films were brought to atmospheric pressure and promptly transferred to a plasma enhanced chemical vapor deposition (PECVD) chamber where 20 nm of silicon nitride (Si_3N_4) were deposited at a temperature of 250 °C. This capping layer was deposited over all the films before further processing to eliminate spurious oxidation of the TFR films when exposed to ambient. All characterization was performed on as-deposited and annealed samples. Annealing was carried out at 325 and 450 °C in forming gas (5% H₂: 95% N₂) with a flow of 3 L/min for 1hr. XRD was performed on the annealed film at 450 °C in a Rigaku Ultima equipment with an Cu K_{α} source kept a grazing angle of 0.5°. Chemical characterization was performed by XPS (PHI series) equipped with an Al source. During the analyses the samples were neutralized with electron and ion guns. Sputtering with Argon at 500 V over a 3×3 mm square area was used to etch the Si₃N₄ capping layer to reach the bulk of the TFR. For the electrical characterization the samples underwent a fabrication process consisting of three photolithography layers described below.

2.2. Test structure fabrication process

The fabrication process for the TFR test structures consists of three photolithography steps, as shown in the schematic in Fig. 1. The test structures consist of the TFR film (green) with the Si₃N₄ capping layer acting also as a hard mask (blue). The first photolithography step uses a dry etch in CF₄ at 140 mTorr and 50 W of power to define the TFR features. This step etches the top Si₃N₄ and TFR sequentially to reach the SiO_2 used as substrate (Step 1 – Definition of TFR/Hard mask). After step 1, the photoresist used was removed with acetone and the films cleaned using isopropyl alcohol in sonication, rinsed with water and dried with nitrogen gas before the next fabrication step. An over-layer of Si₃N₄ was then deposited under the same PECVD conditions used before. This second deposition of Si₃N₄ is used to cover the sides of the defined TFR features (Step 2 - TFR overcoat), effectively isolating the TFR from contact with the ambient. Next, to open vias through the Si_3N_4 and expose the TFR (Step 3 – Open vias) surface, a dry etch in CF₄ followed by an HF dip used to ensure full opening of the vias. The vias are 40 μ m in width and separated at varying lengths from 10 to 150 µm. After removing the photoresist, metallization is carried out in a sputtering system. The contact consists of a titanium adhesion layer (10 nm) followed by titanium nitride (100 nm) finished with aluminum (200 nm). All the metals are deposited in-situ at 4 mTorr. Titanium and aluminum were deposited in argon while nitrogen was introduced to deposit the titanium nitride layer. When the metal deposition was completed the samples were subjected to a final fabrication step to define the contacts and contact pads shown in brown in Fig. 1 (Step 4 -Contact definition). A plasma enhanced dry etch process at room temperature in a chlorine based chemistry is used for this purpose. The testing structures achieved with this process include probes for transmission line method (TLM) and Van der Pauw geometries for Hall effect measurements.

2.3. Electrical characterization

Current vs. voltage (I-V) characteristics were measured in a probe station with a chuck with heating range up to 150 °C. To extract the TCR the films sheet resistance was evaluated at 25, 50, 100 and 150 °C using the TLM approach in which contacts are placed on top of the TFR at different separation lengths and I-V characteristics are measured for each length. Next, resistance is

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