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Electrical properties of TiN films deposited by filtered cathodic vacuum arc

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High quality TiN films were deposited by an off-plane double bend filtered cathodic vacuum arc technique. The influence of deposition pressure, substrate bias, and deposition temperature on the structure and electrical resistivity of TiN films were systematically studied. As the deposition pressure is increased, the film structure evolves from hexagonal $\alpha$-TiN$_{0.30}$ to cubic TiN, and the electrical resistivity decreases drastically at the pressure below $2 \times 10^{-4}$ Torr, then increases slightly with the further increase of deposition pressure. With the increase of substrate bias, the electrical resistivity decreases drastically, reaching the minimum of 45 $\mu$Ω cm at a substrate bias of $-100$ V, then increases greatly, which results from the variation of N content in TiN films with increasing substrate bias. The increase in the deposition temperature results in a significant decrease in the defect density and a slight increase in the grain size, which accounts for a linear decrease in the electrical resistivity. Our results indicate that the main factors that affect the electrical resistivity of TiN films are the N content, phase structure, and defect density in the films. The grain size plays only a minor role in the electrical resistivity of TiN films.

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I. INTRODUCTION

TiN is an important diffusion barrier material for the design of very large scale integrated and ultralarge-scale integrated (ULSI) device due to its excellent diffusion barrier characteristics, such as very good electrical conductivity, and excellent adhesion/glue layer performance. Many chemical vapor deposition, plasma enhanced chemical vapor deposition, and physical vapor deposition techniques have been developed to deposit TiN films. Due to the simpler nature, high deposition rate, lower cost, as well as the high quality (high density, low resistivity, and low impurity content) of the deposited TiN films, magnetron sputtering is almost exclusively used in manufacturing applications. However, with the shrinking of the circuit dimensions and the increase in the aspect ratio of semiconductor device features, conventional sputtering has become less useful for deposition diffusion barriers, adhesion layers, and seed layers because of the cosine angular distribution of species sputtered from the magnetron source as well as a scattering with working gas in the target-substrate volume result in a mostly isotropic deposition profile. Effective deposition into deep vias or contact holes is almost impossible if a standard magnetron sputtering technique is used due to the edge overhang and subsequent void formation.

Filtered cathodic vacuum arc (FCVA) is a promising technique for producing TiN films in the features with high aspect ratio. It employs electromagnetic and mechanical filtering techniques to remove unwanted macroparticles and neutral atoms. Only ions within a defined energy range reach the substrate, thus producing films with good controllability and reproducibility. The high ions fraction in the plasma makes the depositing trajectories of the metal ions more controlled and potentially more conformal in high aspect ratio structures. In addition, the long distance between the target and substrate as well as the low deposition pressure (10$^{-4}$ Torr) will also benefit the efficient directionality.

Electrical resistivity is one of the most important properties for the application of TiN films as barrier layers in the ULSI. Many factors affect the electrical resistivity of the deposited TiN films. The resistivity of the deposited films has been reported to depend largely on the deposition technology and the deposition processes. Patsalas reported that the electrical resistivity of TiN films deposited by dc reactive magnetron sputtering decreases linearly with increasing deposition temperature to 400 °C and drastically with increasing substrate bias from $-40$ to $-100$ V. Tsai observed that a $-115$ V bias is required at the deposition temperature of 40 °C, whereas a $-100$ V bias is necessary at 500 °C to produce the lowest resistivity in sputtered TiN films. Thorpe also found that the TiN films deposited at 500 °C possessed the lowest resistivity.

In this article, TiN films were deposited by an off-plane double bend filtered cathodic vacuum arc technique. The phase of the films is identified by x-ray diffraction (XRD). The electrical resistivity is measured by four-point probe. The influence of deposition parameters, i.e., deposition pressure, substrate bias, and deposition temperature, on the structure and electrical resistivity were systematically studied.
The emphasis is placed on the correlation between the film structure and the electrical resistivity of the deposited films.

II. EXPERIMENTAL DETAILS

TiN films were deposited by an off-plane double bend filtered cathodic vacuum arc (FCVA) technique. The detailed deposition apparatus used in this work was published elsewhere. A 99.999% pure Ti target with a diameter of 80 mm was used as cathodic. High purity (99.99%) nitrogen gas was used as reactive gas. The base pressure was $2.0 \times 10^{-6}$ Torr. A curvilinear axial magnetic field is introduced in the system via an off-plane, double bend curved toroidal duct. The arc current was set at 145 A. The substrates used were (100) p-type silicon wafers with thickness of 400 ± 25 µm. The substrates were heated using a quartz halogen lamp whose power was controlled by varying the input voltage. The substrate temperature during deposition was monitored by a thermocouple attached to the substrate surface.

During the deposition, the deposition pressure, substrate bias, and deposition temperature can be varied in the range $1 \times 10^{-5}$–$6 \times 10^{-4}$ Torr, 0 to −500 V, and 50–500°C, respectively. The film thickness is kept at 100–150 nm.

The phase and crystal structure of deposited films were identified by using an X-ray diffractometry (Rigaku, Japan) with a thin film attachment. A Cu Ka X-ray source (wavelength of 1.54 Å) was used at 50 kV and 20 mA. The incident beam angle was fixed at 0.5°. The sheet resistance of the samples was measured by four-point probe (ResMap). The electrical resistivity of the deposited films is defined as the sheet resistance divided by the film thickness. The thickness of the deposited films was determined by measuring the step height between masked and unmasked regions on the substrate using a surface profilometer (Tencor P10).

III. RESULTS

A. Effect of deposition pressure

The XRD patterns in the $2\theta$ range of 30°–70° for TiN films deposited at the pressure of $1 \times 10^{-5}$, $5 \times 10^{-5}$, $2 \times 10^{-4}$, and $6 \times 10^{-4}$ Torr are shown in Fig. 1. For this series of experiments, the deposition temperature and substrate bias were kept at 450°C and −100 V, respectively. The XRD pattern of TiN films deposited at a pressure of $1 \times 10^{-5}$ Torr is mainly composed of three sharp and strong peaks at the $2\theta$ of 34.8°, 39.8°, and 62.5°, which fit quite well with the (110), (101), and (110) plane of hexagonal α-TiN$_{0.30}$, respectively, as shown in Joint Committee on Powder Diffraction Standards (JCPDS) 40–1276. For the films deposited at a pressure of $5 \times 10^{-5}$ Torr, three very weak peaks at the $2\theta$ of 36.9°, 42.3°, and 62.0°, corresponding to (111), (200), and (220) plane of cubic TiN$_{0.90}$, respectively, can be observed in the XRD pattern. After closer inspection, three subpeaks at 34.8°, 39.8°, and 62.5° corresponding to (100), (101), and (110) plane of α-TiN$_{0.30}$, respectively, can also be distinguished from the background of the XRD pattern. This indicates the coexistence of hexagonal α-TiN$_{0.30}$ phase and cubic substoichiometric TiN$_{0.9}$ phase in the deposited films. It is interesting to note that the intensity of all peaks is the lowest and the full width at half maximum (FWHM) is the broadest as compared with the XRD patterns of the TiN films deposited at higher or lower pressure. A similar phenomenon has been observed in the deposition of other transition metal nitride films. In the deposition of WN films by sputtering, Shen$^{20}$ reported that an amorphous phase was observed in the range of about 12%–28% N in WN films. Min$^{21}$ also found that an amorphous phase was formed at about 33 at. % nitrogen content in TaN films deposited by dc magnetron sputtering. The mismatch of the lattices of hexagonal α-TiN$_{0.30}$ phase and cubic TiN$_{0.90}$ phase prevents the growth of the grains and induces defects in the deposited films, which may contribute to the low intensity and broadening of the XRD peaks. For the films deposited at a pressure of $2 \times 10^{-4}$ Torr, the XRD pattern is composed of three sharp peaks at 36.5°, 42.6°, and 61.8°, which fit well with the (111), (200), and (220) plane of stoichiometric TiN, respectively. This is consistent with our X-ray photoelectron spectroscopy results, not shown, which show that stoichiometric TiN films can be deposited at a pressure of $2 \times 10^{-4}$ Torr. Except for a slight increase in the peak width, no significant change in XRD patterns can be observed for the films deposited at higher deposition pressure.

The full width at half maximum (FWHM) of the XRD peaks is correlated with the grain size. The average grain size can be calculated using the Scherrer equation$^{22}$:

$$D = \frac{0.9 \lambda}{B \cos \theta},$$

where $\lambda$ is the X-ray wavelength, $B$ is the breadth in 2θ units, and $\theta$ is the Bragg diffraction angle. Figure 2 shows the influence of deposition pressure on the FWHM of the (111) peak.
peaks in XRD patterns and the calculated average grain size of the deposited films. At the pressure below $2 \times 10^{-3}$ Torr, the increase in the deposition pressure results in a significant decrease and increase in the FWHM and grain size, respectively. The further increase in the deposition pressure leads to a great increase and decrease in the FWHM and grain size, respectively.

Thin film growth is a dynamic process whereby the absorbates migrate over the surface until they reach a potential minimum such as surface steps or nucleation sites. As N$_2$ is induced into the chamber, the nitrogen atoms will be adsorbed on the growing film surface, which can act as a roadblock to the diffusing Ti atoms or can trap diffusing Ti atoms by forming chemical bonds, resulting in a decrease in the mobility of the adsorbed Ti atoms, and therefore a decrease in grain size of the deposited films. However, increasing N$_2$ pressure may also lead to an increase in the ratio of $J^N / J^Me$, where $J^N$ and $J^Me$ are the incoming nitrogen and metal ion flux, respectively. At higher nitrogen pressure each deposited metal atom is struck by more energetic ions before it is incorporated into the growing film. Adibi proposed that due to the low impact energies most of the momentum transfer to the lattice occurs at the surface, and the primary effect of the increasing incident ion flux at these low energies is to collisionally enhance adsorbed cation surface mobility. This, in turn, leads to the increase of the grain size with increasing nitrogen pressure.

Figure 3 illustrates the influence of the deposition pressure on the electrical resistivity of the deposited films. The electrical resistivity for the films deposited at a pressure of $1 \times 10^{-5}$ Torr is fairly high (180 $\mu$Ω cm). As the deposition pressure is increased, the electrical resistivity of the deposited films decreases drastically, and reaches the minimum of 44 $\mu$Ω cm at a pressure of $2 \times 10^{-4}$ Torr. The further increase in the deposition pressure results in a slight but continuous increase in the electrical resistivity. The variation of the electrical resistivity with deposition pressure results from the change in the composition and crystal structure of the deposited films, which will be discussed below.

**B. Effect of substrate bias**

The XRD patterns for TiN films deposited at the substrate bias of 0, –100, and –300 V are shown in Fig. 4. For this series of experiments, the deposition pressure and temperature remained constant at $2 \times 10^{-4}$ Torr and 450 °C, respectively. All the XRD patterns are composed of three strong peaks corresponding to the (111), (200), and (220) plane of cubic TiN. No significant change in the line shape and position were observed as substrate bias is increased. However, the quantity analysis of the XRD patterns indicates that the FWHM of (111) peak is greatly affected by the substrate bias.

Figure 5 shows the dependence of the full width at half maximum of (111) peak in XRD patterns and the calculated average grain size in TiN films on the substrate bias. As the substrate bias is increased in the bias range of 0 to –150 V, the FWHM of (111) peak increases rapidly. The further increase in the substrate bias results in a great decrease in the FWHM. The variation trend of the grain size with substrate bias is contrary to that of the FWHM. It is also observed that the color of the deposited films is greatly affected by the substrate bias, which indicates the variation of N content in the deposited films with substrate bias. Golden color TiN films, which is the characterization of the stoichiometric TiN films, can only be obtained in the substrate bias range –50 to
The color of the film deposited at lower or higher bias range is light yellow, which indicates the lower N content in the deposited films. This is also consistent with our x-ray photoelectron spectroscopy measurements. Wulff and Eggs also reported that increasing substrate bias in the range 0 to 120 V resulted in a linear increase of N content in the TiN films deposited by magnetic sputtering. As discussed above, the increase in the N content in TiN films with increasing substrate bias to −150 V leads to a decrease in the adatom mobility, which, in turn, results in a decrease in the grain size. In addition, the increase in the substrate bias is reported to improve the nuclear of TiN grain in the lower bias range, which may also contribute to the initial decrease of grain size with increasing substrate bias. However, the further increase in the substrate bias results in self-sputtering of the deposited films due to the bombardment of energetic ions. The sputtering rate of N atoms is higher as compared to Ti atoms, leading to the preferred sputtering of N atoms in the deposited films, and therefore the decrease of N content in the TiN films deposited at higher substrate bias, which accounts for the increase in the grain size. In addition, the increase in the substrate bias may transfer more energy to the growing film surface, leading to the increase in the adatom mobility, which may also account for the increase in the grain size.

The dependence of substrate bias on the electrical resistivity of TiN films is shown in Fig. 6. The electrical resistivity of TiN films is greatly affected by the substrate bias. As substrate bias is increased from 0 to −100 V, the electrical resistivity decreases drastically from 124 μΩ cm to 45 μΩ cm. However, the further increase in the substrate bias to −300 V results in a great increase in the electrical resistivity to 125 μΩ cm.

C. Effect of deposition temperature

XRD patterns for the films deposited at the deposition temperatures of 50, 200, and 500 °C are plotted in Fig. 7. For this series of experiments, the deposition pressure and substrate bias is set to be 2×10⁻⁴ Torr and −100 V. The XRD patterns of TiN films are strongly dependent on the deposition temperature. For the films deposited at a temperature of 50 °C, two weak broad peaks at the 2θ of 36.4° and 61.6°, corresponding to the (111) and (220) plane of TiN, respectively, can be observed in the XRD pattern. The weak feature indicates the high defect density in the deposited films. As the deposition temperature is increased, the intensity of the XRD peaks increases significantly and the sharpening of the peaks is also observed, which indicates the decrease of the defect density and increase of the grain size in the deposited films. In addition, one addition peak at the 2θ of 42.6°, which corresponds to the (200) plane of TiN, respectively, can be observed in the XRD pattern. The weak feature indicates the high defect density in the deposited films. As the deposition temperature is increased, the intensity of the XRD peaks increases significantly and the sharpening of the peaks is also observed, which indicates the decrease of the defect density and increase of the grain size in the deposited films.
total energy of the deposited films. Then, the adatom would be arranged into the (200) planes to lower the surface energy as well as the total energy of the deposited films, and therefore the evolution of the preferred orientation of the deposited films.

The influence of deposition temperature on the grain size is shown in Fig. 8. As the deposition temperature is increased from 50 to 500 °C, the average grain size increases linearly from 13 to 19 nm. The variation trend of the grain size with deposition temperature is the very reverse of that of the electrical resistivity as shown below. It is well known that the increase in the deposition temperature results in the enhancement of the adatom surface mobility as well as the atomic diffusion, thereby enhancing grain growth, which leads to the linear increase in the grain size with deposition temperature.

Figure 9 illustrates the dependence of the electrical resistivity of TiN films on the deposition temperature. The electrical resistivity of the deposited films decreases linearly from 86 μΩ cm to 43 μΩ cm with increasing substrate temperature from 50 to 500 °C. This is consistent with the previous results, which showed a linear decrease in the electrical resistivity of TiN films deposited by sputtering with increasing deposition temperature.

IV. DISCUSSIONS

Single-crystal TiN is a metallic conductor with an electrical resistivity of 23 μΩ cm at room temperature. However, TiN films rarely achieve the resistivity of single-crystal TiN. In the present study, TiN films were deposited on substrates held at temperatures (50–500 °C) of much less than half the melting temperature of TiN (2950 °C); growth occurs under highly nonequilibrium conditions, which results in the formation of amorphous adsorbates and metastable phases containing lattice vacancies and pores. According to the band structure, strongly hybridized N 2p and Ti 3d orbitals contribute nine valence electrons to the electrical conductivity. In this scheme, electron scattering can reduce the effective number of valence electrons, thereby increasing the room-temperature resistivity. The resistivity of polycrystalline TiN films may be depending largely on composition, morphology, film thickness, grain size, and impurities. Due to the fact that deposited films are atomically smooth, of high purity, and with comparable thickness, the influence of morphology, film thickness, and impurities on the variation of electrical resistivity of TiN films is negligible.

It is demonstrated that the electrical resistivity of the deposited films is affected by the grain size. Within the boundary region, there is some atomic mismatch in a transition from the crystalline orientation of one grain to that of an adjacent one, which leads to the disordered arrangement of atoms and lack of complete bonding along grain boundaries, which in turn, results in the existence of high density defect in the grain boundary. These defects act as the scattering center to the electron, which results in the increase in the electrical resistivity of TiN films at the room temperature. According to the electron scattering theory of Wissman and Muller, the resistivity increments induced by grain boundaries are given by

$$\Delta \rho = \rho_0 Z A l_0 / d,$$

where $\rho_0$ is the resistivity inside the crystal grains of the film due to isotropic background scattering of electrons, $d$ is the average grain diameter, $Z$ is the scattering center densities at grain boundaries, $A$ is mean scattering cross sections at grain boundaries, $l_0$ is the mean-free path of conduction electrons in the metal. Another important factor that determines the electrical resistivity of the deposited films is the films’ composition. In the substoichiometric range high density of nitride vacancies is known to be formed in the crystal lattice, leading to carrier scatter and the increase of electrical resistivity as nitrogen content decreases.

As shown in Fig. 3, the electrical resistivity of the deposited films is strongly dependent on the deposition pressure. This results from the variation in the phase structure, composition, and grain size with deposition pressure. For the films deposited at a pressure of 1 × 10^{-5} Torr, XRD indicates that the deposited films are composed of hexagonal $\alpha$-TiN$_{0.30}$ phase, which contributes to the highest electrical resistivity. The increase in the deposition pressure results in a increase in the N content in the films, which may reduce the nitride vacancies, and therefore the decrease in the electrical...
resistivity. In addition, the increase in the grain size with increasing deposition pressure to $2 \times 10^{-4}$ Torr, as shown in Fig. 1, will also contribute to the decrease in the electrical resistivity of TiN films. At the deposition pressure above $2 \times 10^{-4}$ Torr, the grain size decreases with increasing deposition pressure, which leads to a slight increase in the electrical resistivity of TiN films. It is interesting to note that increasing deposition pressure from $1 \times 10^{-5}$ to $2 \times 10^{-4}$ Torr results in an increase in the grain size by 8 nm and a decrease in the electrical resistivity by 136 $\mu \Omega$cm, but increasing deposition pressure from $2 \times 10^{-4}$ to $6 \times 10^{-4}$ Torr results in a decrease in grain size by 6 nm and an increase in electrical resistivity by 20 $\mu \Omega$cm. In these two regions of the deposition pressure, the variation in the grain size is almost the same, but the change in the electrical resistivity is much different. This indicates that the variation of grain size with deposition pressure plays a minor role to the electrical resistivity of TiN films. The main factor that affects the electrical resistivity should be the crystal structure and the N content in the deposited films.

The electrical resistivity of TiN films is greatly affected by substrate bias as shown in Fig. 6. As discussed above, the increase in the substrate bias to $-100$ V results in an increase in the N content in TiN films, which accounts for the great decrease in the electrical resistivity. It is well known that the ion energy is determined by the substrate bias in the deposition of thin films by the filtered cathodic arc. The increase in the substrate bias results in a linear increase in the ion energy. The bombardment of the energetic ion on the growing TiN film surface leads to the densification of the deposited films due to the atomic peening effect. It has been demonstrated recently that the electrical resistivity of sputtered TiN films decreases with increasing film density. The densification of the deposited films may also contribute to the decreases in the electrical resistivity. The further increase in the substrate bias causes the decrease in the N content in the deposited films due to the preferred sputtering of N atoms by the bombardment of energetic ions, resulting in the increase of nitride vacancies in the deposited films, and therefore the increase in the electrical resistivity. It is interesting to note from Fig. 5 that the variation trend of the grain size with substrate bias, which decreases at the bias of below $-200$ V, then increases with the further increase of substrate bias. This is in contrast to that of the electrical resistivity. This further supports the argument that the grain size plays a minor role in the electrical resistivity of TiN films.

The electrical resistivity of TiN films is also strongly dependent on the deposition temperature (Fig. 9). The increase in grain size with increasing deposition temperature may result in the decrease in the electrical resistivity. However, Fig. 8 shows that increasing deposition temperature to 500 °C results only in the increase of grain size by 7 nm. As discussed above, the small increase in the grain size results in a slight decrease in the electrical resistivity. This indicates that there exist other factors, which cause the great decrease in the electrical resistivity with increasing deposition temperature. As shown in Fig. 7, for the films deposited at 50 °C, the XRD peaks are very weak, which indicates the existence of high-density defect in the deposited films. These defects cause the electrical scattering, contributing to the high electrical resistivity for the films deposited at low temperature. The increase in the deposition temperature results in a drastic increase in the intensity of all XRD peaks, indicating the significant decrease in the defect density, which will be the main reason for the drastic decrease in the electrical resistivity of TiN films with increasing deposition temperature.

As mentioned above, high quality dense stoichiometric TiN films with a resistivity of 44 $\mu \Omega$cm can be deposited by using FCVA at a relatively low deposition temperature (450 °C) owing to the high ion energy. The very low deposition pressure ($2 \times 10^{-4}$ Torr) is very important for the conformal deposition of TiN films in high aspect ratio structures. This indicates that FCVA is a more promising technique for the deposition of TiN films over the techniques currently used in industry. Conformal TiN films and Cu films have been deposited in our laboratory into the trench of 500 nm in depth and 300 nm in width (not shown here).

V. CONCLUSIONS

TiN films were deposited by an off-plane double bend filtered cathodic vacuum arc technique. XRD and four-point probe were used to characterize the crystal structure and electrical resistivity of the films. The influence of deposition pressure, substrate bias, and deposition temperature on the structure and electrical resistivity were systematically studied. As deposition pressure is increased to $2 \times 10^{-4}$ Torr, the structure of the films develops from hexagonal $\alpha$-TiN$_{0.30}$ to cubic TiN, leading to a drastic decrease in the electrical resistivity. The further increase of deposition pressure results in a great decrease in the grain size, which contributes to the slight increase in the electrical resistivity of TiN films. As the substrate bias is increased, the electrical resistivity of TiN films decreases significantly, reaching the minimum at a substrate bias of $-100$ V, then increases greatly with the further increase of substrate bias, which results from the variation of N content in TiN films with increasing substrate bias. The increase in the deposition temperature results in a drastic decrease in the defect density and an increase in the grain size of TiN films, which accounts for a linear decrease in the electrical resistivity of TiN films. It indicates that the main factors that affect the electrical resistivity of TiN films are the N content, phase structure, and defect density. The grain size plays only a minor role in the electrical resistivity of TiN films.

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