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Effects of magnetic field strength and deposition pressure on the properties of TiN films produced by high power pulsed magnetron sputtering (HPPMS)

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ABSTRACT

The drawbacks of titanium nitride (TiN) films deposited by high power pulsed magnetron sputtering (HPPMS) are low deposition rate (compared with conventional magnetron sputtering) and high residual stress. Many methods have been used to deal with these issues. In this study, TiN films were deposited by HPPMS at different magnetic field strength $\boldsymbol{B_s}$ (40 and 115 mT) and deposition pressure $\boldsymbol{P_d}$ (0.2, 1.0, and 2.0 Pa). The effects of $\boldsymbol{B_s}$ and $\boldsymbol{P_d}$ on film deposition rate, residual stress, and mechanical properties were also investigated. The plasma in the HPPMS discharge was diagnosed by optical emission spectroscopy. Due to the stronger magnetic confinement and back-attraction around the target at higher $\boldsymbol{B_s}$ (115 mT), lower $\boldsymbol{B_s}$ (40 mT) led to a significant increase in the Ti^+/Ti ratio of the substrate, by a factor of ~3, and a large increase in the deposition rate, especially by a factor of ~5.5 when the $\boldsymbol{P_d}$ was 2.0 Pa. The high Ti^+/Ti ratio in front of the substrate at the lower $\boldsymbol{B_s}$ (40 mT) increased the degree of ion bombardment and resulted in a preferred and stronger (111) orientation, greater film hardness, and better adhesion strength of the TiN films. Furthermore, the increase in $\boldsymbol{P_d}$ caused more collisions and stronger scattering for metal atoms and ions, which resulted in a sharp decrease in residual stress (from 8.5 to 0.8 GPa at 40 mT $\boldsymbol{B_s}$) and a significant increase in the adhesion strength of TiN films.

1. Introduction

Owing to its extreme hardness, high thermal stability, and golden yellow color, titanium nitride (TiN) has been widely applied in various fields, such as in hard coatings on mechanical tools, decorative coatings, and diffusion barriers in the microelectronics industry [1–3]. TiN thin films can be deposited by a variety of methods including physical vapor deposition (PVD) techniques, such as magnetron sputtering [4], ion beam assisted deposition [5], and e-beam evaporation [6], and chemical vapor deposition (CVD) [7]. Among the various PVD techniques, high power pulsed magnetron sputtering (HPPMS) is a recent development magnetron sputtering, which utilizes extremely high-power short pulses to ionize the sputtered metal atoms. It is characterized by high plasma densities in the order of ~10^{17}–10^{19} m^{-3}, high power densities ranged from 0.05 to 10 kW/cm^2, and low duty cycles of <10% [8–10]. The quality of TiN films deposited by HPPMS, especially the adhesion, density, and surface roughness, is better than that by conventional magnetron sputtering [11–13]. However, in most cases, the deposition rate of HPPMS is lower than that of conventional magnetron sputtering [14,15]. This is caused by the back-attraction of the positively charged metal ions to the cathode [16,17], magnetic confinement of the sputtered flux [18], and target poisoning.

Many methods have been taken by researchers to improve deposition rate, such as changing electrical parameter [19,20], adjusting magnetic field characteristic, applying assisted deposition technique [21–23], and so on. Altering magnetic field strength is considered an effective way to improve the deposition rate of HPPMS. Bohlmark et al. [18] and Jones Alami et al. [24] have showed that the magnetic configuration has an important impact on discharge properties and ion transport in HPPMS plasma. Bohlmark et al. have demonstrated that use of an additional, adjustable magnetic field can lead to the deposition of ionized metal flux being redirected to the substrate, thus improving the deposition rate. Ehiasarian [25] have recommended the use of lower magnetic field strength (lower than 40 mT for example), which can weaken the magnetic confinement of the plasma and allow more positive ions reaching the substrate. Ehiasarian and Vetushka [26] have demonstrated that a decrease in magnetic field strength from 50 mT to 25 mT can increase the deposition rate of Cr films by >30%. In addition, Mishra et al. [27] and Capek et al. [28] also have found that the deposition rate of Ti and Nb films can be increased by weakening magnetic field.

In this paper, TiN films were fabricated by HPPMS at different magnetic field strength ($\boldsymbol{B_s}$: 40 and 115 mT) and deposition pressure ($\boldsymbol{P_d}$:

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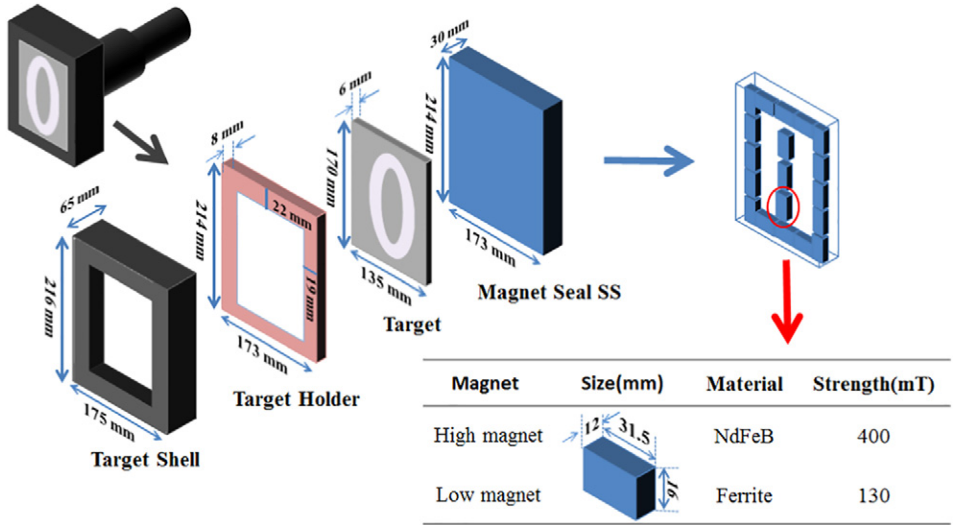

Fig. 1. The schematic diagram of sputtering target system and the arrangement of magnet.

0.2, 1.0, and 2.0 Pa). The effects of $\boldsymbol{B_s}$ and $\boldsymbol{P_d}$ on film properties (deposition rate, residual stress, surface morphology, and mechanical properties) were discussed.

2. Experimental methods

All the experiments were carried out in a vacuum deposition system using a grounded stainless steel chamber, which is cylindrical shaped with a 500 mm diameter and 500 mm height [29]. Four unbalanced magnetron plasma sources with a rectangular planar titanium target (135×170 mm^2) were placed inside the vacuum chamber. The cathode of the HPPMS device was powered by a pulsed power supply fabricated by Chengdu Pulsetech Electrical (HPS-450D, China). A 3 Ω resistor R_1 in the HPPMS power supply was provided to protect the power source from arcing and limit the plasma current.

In this study, the magnetron's magnetic field strength at the target surface, $\boldsymbol{B_s}$, was modified by replacing the magnets behind the target. The structure and size of the sputtering target system, and the arrangement and characteristics of the magnet, are shown in Fig. 1. The sputtering target system consisted of a target shell, target holder, target, magnet seal stainless steel and magnets. Nineteen magnets were arranged regularly in magnet seal stainless steel. The magnetic field strength of a magnet made by NdFeB was ~400 mT, while another magnet made by ferrite was ~130 mT. The Ti target surface was exposed to a $\boldsymbol{B_s}$ of either 40 mT or 115 mT during HPPMS. TiN films were deposited on Si wafers (10×30 mm^2) and 316 L stainless steel (SS) samples by HPPMS at different $\boldsymbol{B_s}$ and $\boldsymbol{P_d}$. Before the deposition of all films, the Si wafers and 316 L SS samples were ultrasonically clean in acetone (5 min), alcohol (5 min) and distilled water (5 min), and then all of them were installed on a substrate holder with a target to substrate distance of 80 mm. After the vacuum chamber was pumped to a base pressure of 2.0×10^{-3} Pa, the target in vacuum chamber was cleaned by a DC magnetron sputtering (3 A) for 10 min and the substrate was cleaned by a glow discharge with Ar ions (3.0 Pa, applied DC bias voltage −1700) for 15 min. A Ti interlayer was prepared on all substrate by DC magnetron sputtering (3 A, 1 min) with pulse substrate bias (1 kV, 50% duty cycle). During TiN films deposition, all the samples were grown at a floating potential without additional heating. A pulse length ($\boldsymbol{\tau}$) of 200 μs, a frequency ($\boldsymbol{f}$) of 150 Hz, and an average target power ($\boldsymbol{P_{ave}}$) of 1.2 kW were maintained for all experiments. The highest peak current density was 0.78 A/cm^2 and the highest peak power density was 0.61 kW/cm^2. The amount of nitrogen and argon gas injected into the vacuum chamber was kept constant at a ratio of 3:20, while the $\boldsymbol{P_d}$ was varied between 0.2 Pa, 1.0 Pa, and 2.0 Pa by controlling the pumping rate. The substrate temperature monitored by a thermocouple during deposition was about 200 °C. Sample numbers H-0.2, H-1, and H-2 correspond to films fabricated using a higher magnetic field strength (115 mT) at 0.2 Pa, 1.0 Pa, and 2.0 Pa, respectively. Sample numbers L-0.2, L-1, and L-2 correspond to films fabricated using a lower magnetic field strength (40 mT) at 0.2 Pa, 1.0 Pa, and 2.0 Pa, respectively.

Waveforms of the discharge voltage and the target current during HPPMS discharge were measured by a Tektronix TDS-200 oscilloscope with a Tektronix P-5100 voltage probe and a Pearson 411 current monitor, respectively. The discharge plasma of the target surface and the substrate were diagnosed by optical emission spectroscopy (Avantes, AveSpec-2048-USB2-RM). To observe the plasma at the target surface, the probe of the optical fiber was oriented towards the cathode. For the plasma at the substrate, the optical fiber probe overlooked the discharge at a distance of 1 cm from the substrate and oriented parallel with the substrate surface. The observed spectra ranged in wavelength from 200 to 1100 nm with a resolution of 0.1 nm. The slit width was 10 μm. The acquisition time for spectra at the target surface and the substrate was 20 ms and 250 ms, respectively. Obtained spectra were analyzed by a spectrum analyzer (Avantes, AvaLIBSSpecline-AMS).

A stylus profiler (Ambios XP-2, USA) was used to measure the thickness of the TiN films. Residual stress in the TiN films was measured by the wafer curvature method [30]. The crystal structure of the TiN films

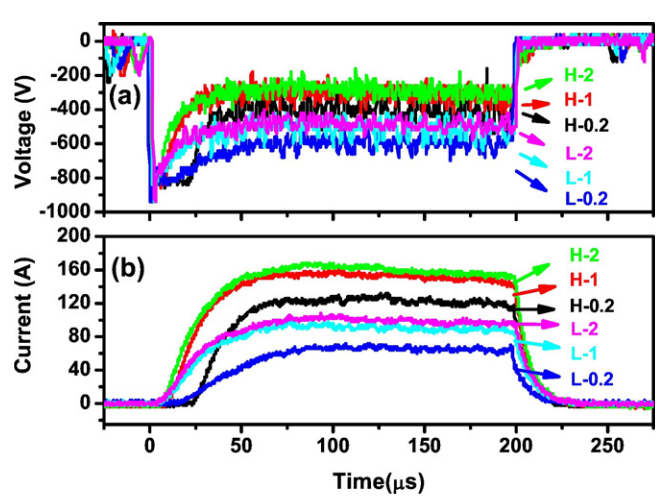

Fig. 2. Waveforms of the discharge characteristics of (a) target voltage and (b) target current at different $\boldsymbol{B_s}$ and $\boldsymbol{P_d}$.