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Self-organized lamellar structured tantalum–nitride by UHV unbalanced-magnetron sputtering

G.R. Lee[a,*], J.J. Lee[a], C.S. Shin[b], I. Petrov[b], J.E. Greene[b]

[a]Materials Science and Engineering Department, Seoul National University, San 56-1 Shilim-dong, Kwanak-gu, Seoul 151-742, South Korea
[b]Materials Science and Engineering Department, University of Illinois at Urbana-Champaign, 104 S Goodwin, Urbana, IL 61801, USA

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Abstract

The effect of crystal orientation and microstructure on the mechanical properties of TaN_x was investigated. TaN_x films were grown on a SiO_2 substrate by ultrahigh vacuum unbalanced magnetron sputter deposition in mixed Ar/N_2 discharges at 20 mTorr (2.67 Pa) and at 350 °C.

Unlike the Ti–N system, in which TiN is the terminal phase, a large number of N-rich phases in the Ta–N system could lead to layers which had self-organized nanosized lamella structure of coherent cubic and hexagonal phases, with a correct choice of nitrogen fraction in the sputtering mixture and ion irradiation energy during growth. The preferred orientations and the microstructure of TaN_x layers were controlled by varying incident ion energy E_i (=30–50 eV) and nitrogen fractions f_{N_2} (=0.1–0.15). TaN_x layers were grown on (0002)-Ti underlayer as a crystallographic template in order to relieve the stress on the films.

The structure of the TaN_x film transformed from B1-NaCl δ–TaN_x to lamellar structured B1-NaCl δ-TaN_x+ hexagonal ε-TaN_x or B1-NaCl δ-TaN_x+hexagonal γ-TaN_x with increasing ion energy at the same nitrogen fraction f_{N_2}. The hardness of the films also increased by the structural change. At the nitrogen fraction of 0.1–0.125, the structure of the TaN_x films was changed from δ-TaN_x+ε-TaN_x to δ-TaN_x+γ-TaN_x with increasing ion energy. However, at the nitrogen fraction of 0.15, the film structure did not change from δ-TaN_x+ε-TaN_x over the whole range of the applied ion energy. The hardness increased significantly from 21.1 to 45.5 GPa with increasing ion energy.
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Keywords: TaN; Sputtering; Lamellar; Stress; Hardness

1. Introduction

TaN_x is well known for hard coatings on tools, wear resistant layers, thin film resistors, diffusion barriers in integrated circuits, and mask layers for X-ray lithography. Unlike the more common IVB-VA hard coating material such as TiN, cubic TaN, a VB-VA compound, is metastable. While the Ti–N equilibrium phase diagram is relatively simple with the only compounds being tetragonal Ti_2N and NaCl-structure TiN [1], the Ta–N system is extremely rich [2–4] and relatively unexplored. In addition to the equilibrium phases of bcc α-Ta, solid-solution α-Ta(N), hexagonal γ-Ta_2N, and hexagonal ε-TaN, a variety of metastable phases have been reported. These include tetragonal β-Ta, bcc β-Ta(N), hexagonal Ta_2N, hexagonal WC structure θ-TaN, cubic B1 NaCl-structure δ-TaN, hexagonal Ta_5N_6, tetragonal Ta_4N_5, and orthorhombic Ta_3N_5 [2,3].

Due to the complexity of the Ta–N system, the chemical and phase compositions of as-deposited TaN_x layers have been found to be critically dependent upon growth conditions for all deposition techniques which have been applied: reactive dc- and rf-magnetron sputter deposition [4–9], ion-beam-assisted deposition [10], reactive-electron-beam evaporation with simultaneous nitrogen irradiation [11], and chemical vapor deposition (CVD) [12,13]. Among these techniques, reactive magnetron sputtering is currently the primary candidate for microelectronic device applications, due to its compatibility with conventional metallization schemes in integrated circuit processing, as well as for hard-coating applications.

In this article, we report the results of an investigation of high-energy ion irradiation and N_2 fraction effects on

* Corresponding author. Tel.: +82 2 880 551; fax: +82 2 871 5540.
E-mail address: dklee@psel.snu.ac.kr (G.R. Lee).

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microstructure and texture evolution during deposition of TaN layers and also dependency of film texture and stress on mechanical properties. The films were grown by ultra-high vacuum (UHV) reactive magnetron sputter deposition on amorphous SiO_2 in mixed Ar/N_2 discharges at a total pressure of 20 mTorr and 350 °C. The preferred orientations and film microstructures of TaN_x layers were varied by changing the incident ion energy E_i (=30–50 eV) and nitrogen fraction f_{N_2} (=0.1–0.15) during sputter deposition.

2. Experimental procedure

All TaN_x layers were grown in a load-locked multi-chamber ultrahigh vacuum (UHV) stainless-steel dc magnetron sputter deposition system described in detail in Ref. [14]. The pressure in the sample introduction chamber was reduced to less than 5×10^{-8} Torr (7×10^{-6} Pa), using a 50 l s^{-1} turbomolecular pump (TMP), prior to initiating substrate exchange into the deposition chamber which has a base pressure of 5×10^{-10} Torr (7×10^{-8} Pa), achieved using a 500 l s^{-1} TMP. A water-cooled 6.35-cm-diameter Ta target with a purity of 99.97% was mounted 10 cm from the substrate holder. Sputter deposition was carried at a constant power of 150 W and a total pressure of 20 mTorr (2.67 Pa) in mixed atmospheres consisting of Ar (99.999% pure) and N_2 (99.999%). The nitrogen fraction was varied from 10 to 15. During deposition, the pressure was monitored by a capacitance manometer and maintained constant with automatic mass-flow controllers.

The substrates were $10\times10\times0.5$ mm^3, 400-nm-thick amorphous SiO_2 produced by thermally oxidizing Si(001) wafers. All TaN layers were grown at T_s=350 °C, including the contribution due to plasma heating. The substrate temperature (T_s) was measured with a pyrometer calibrated by a thermocouple bonded to a TaN-coated Si substrate. All TaN films were grown to a thickness of 0.4 μm.

The microstructure and microchemistry of as-deposited samples were characterized by X-ray diffraction (XRD), SEM, transmission electron microscopy (TEM) analyses. ω-2θ XRD scans were carried out in the powder diffraction mode using incident slit divergence of 1°, resulting in a resolution of 0.1° 2θ with Cu K_α radiation (λ=0.15418 nm). Film thicknesses were determined by cross-sectional SEM analyses using field-emission gun Hitachi S4700 microscope. TEM analyses were carried out in a Philips CM12 microscope operated at 120 kV. Nanoindentation responses of TaN films were determined using a Hysitron Tribo Scope instrument attached to the AFM. Epitaxial TiN(001) layers, also grown on MgO(001) and having the same thickness as the TaN samples, served as references for calibration purposes. The triangular Berkovich diamond tip was calibrated. At least three indents were made in each sample using a multiple loading cycle with peak loads of 1, 2, 3, 5, 7, and 9 mN and unloading to 10% of the peak value between. In the final unloading segment, a hold of 100 s was included at 10% of the peak load in order to allow system drift to be measured and corrected for. Hardness H and elastic modulus E values were calculated from each unloading segment using the Oliver and Pharr method [15]. Thus, H and E values were obtained as a function of load (contact depth). Typically, a plateau value was obtained at depths between 20 and 80 nm, which was taken as the film properties. For deeper imprints, the softer SiO_2 substrate dominates the indentation response, while for shallower depths the surface roughness and uncertainties in the exact contact area influence the results. Stresses were determined by the XRD $\sin^2\psi$ technique [16,17] and the Stoney formula.

3. Results and discussion

In order to investigate the independent effects of both film texture and microstructure on mechanical properties,

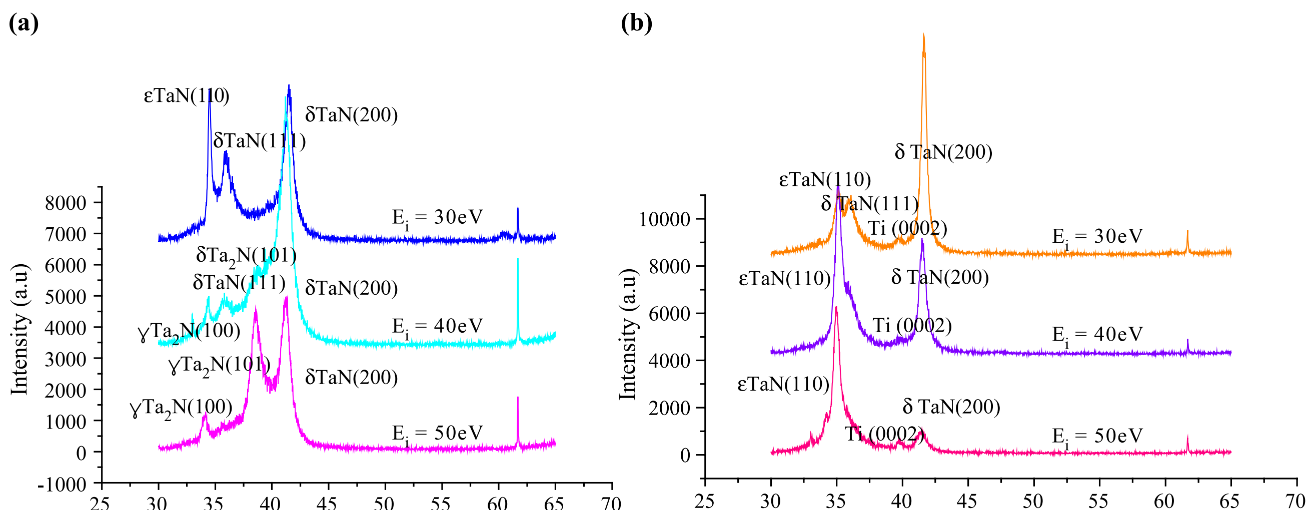

Fig. 1. X-ray diffraction ω-2θ scans from 400-nm-thick TaN layers grown on SiO_2 with E_i=30–50 eV at (a) f_{N_2}=0.1–0.125 and (b) f_{N_2}=0.15.

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