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In-situ electrical characterization of ultrathin TiN films grown by reactive dc magnetron sputtering on SiO_2

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ABSTRACT

Ultrathin TiN films were grown by reactive dc magnetron sputtering on thermally oxidized Si (100) substrates. The electrical resistance of the films was monitored *in-situ* during growth in order to determine the minimum thickness of a continuous film. The coalescence thickness has a minimum of 1 nm at a growth temperature of 400 °C after which it increases with growth temperature. The minimum thickness of a continuous film decreases with increasing growth temperature from 2.9 nm at room temperature to 2.2 nm at 650 °C. In-situ resistivity measurements show that films grown at 500 °C and above are resistant to oxidation indicating high density. X-ray photoelectron spectroscopy and X-ray diffraction measurements show that the TiN grain stoichiometry and grain size increases with increasing growth temperature.

1. Introduction

Ultrathin conducting films are an essential part of modern microelectronics [1]. Titanium nitride (TiN) thin films are widely used as adhesion layers, as diffusion barriers in device interconnects [2] and as a direct-metal-gate material for metal-oxide-semiconductor devices [3,4], due to the thermal stability and low bulk electrical resistivity of TiN. With device dimensions constantly shrinking, the required film thickness is approaching a few nanometers [5]. For such thicknesses the continuity of a metallic film becomes an important issue [6,7].

Size effects also start to limit the electrical conductivity as the dimensions of a conductor approach the mean free path of the conducting electrons. The reduced conductivity can be attributed to surface scattering [8], interface roughness [9], grain boundaries (i.e., reduced crystallinity) [10] and islandization [11]. In addition, the film density affects the conductance [12]. The increased resistivity limits device operating frequency and results in Joule heating of the device, which in turn can cause electromigration [13]. It is therefore important to minimize the resistivity by reducing roughness and increasing density and grain size.

The electrical and structural properties of thin TiN films have been studied extensively. The electrical resistivity is found to be reduced for elevated growth temperatures [12]. TiN has a cubic NaCl-type crystal structure with a lattice constant of 0.424 nm. A change in crystallographic orientation from [200] to [111] occurs with increasing thickness but increased growth temperature or nitrogen process gas ion-to-atom ratio makes the [200] orientation more favored [14]. Sub-nm room temperature grown TiN films have been shown by transmission electron microscopy to be amorphous [15]. However, a systematic study of the thickness at which the film becomes continuous at different growth temperatures has not been carried out previously.

Here we report on the growth and characterization of ultrathin titanium nitride films by reactive dc magnetron sputtering on thermally oxidized Si (100) substrates. The electrical resistance of the films was monitored during growth *in-situ* at several different growth temperatures. The in-situ measurement has the advantage that it gives immediate information about the coalescence thickness and the minimum thickness of a continuous film. In addition, film oxidation can be monitored in-situ which allows the effects of structure and oxidation on the resistivity to be separated. Finally,

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the film texture was examined *ex-situ* by grazing incidence X-ray diffraction (GI-XRD) measurements and film composition was determined by X-ray photoelectron spectroscopy (XPS).

2. Experimental apparatus and method

The TiN thin films were grown in a custom built magnetron sputtering chamber [16] with a base pressure of 4×10^{-6} Pa. The sputtering gas was argon of 99.999% purity mixed with nitrogen gas of 99.999% purity. The argon flow rate was 40 sccm and the nitrogen flow rate 2 sccm to give a growth pressure of 0.4 Pa. The Ti target was 50 mm in diameter and of 99.99% purity. The applied power was set to 100 W.

The substrates used were thermally oxidized Si(100) with an oxide thickness of 500 nm. Au contact pads were defined using a photolithographic lift-off process prior to TiN deposition. The substrate holder is made from a macor ceramic and the four electrical probes are held in contact with the sample contact pads by a ceramic shadow mask, as illustrated in Fig. 1(a). The substrate temperature was controlled during growth with a 1.5 in. circular plate heater, separated from the substrate holder by a 2 mm gap. A *K*-type thermocouple is placed between the heater and sample holder, touching the bottom of the sample holder, to monitor the sample temperature. The substrate holder design is described in more detail by Arnalds et al. [16].

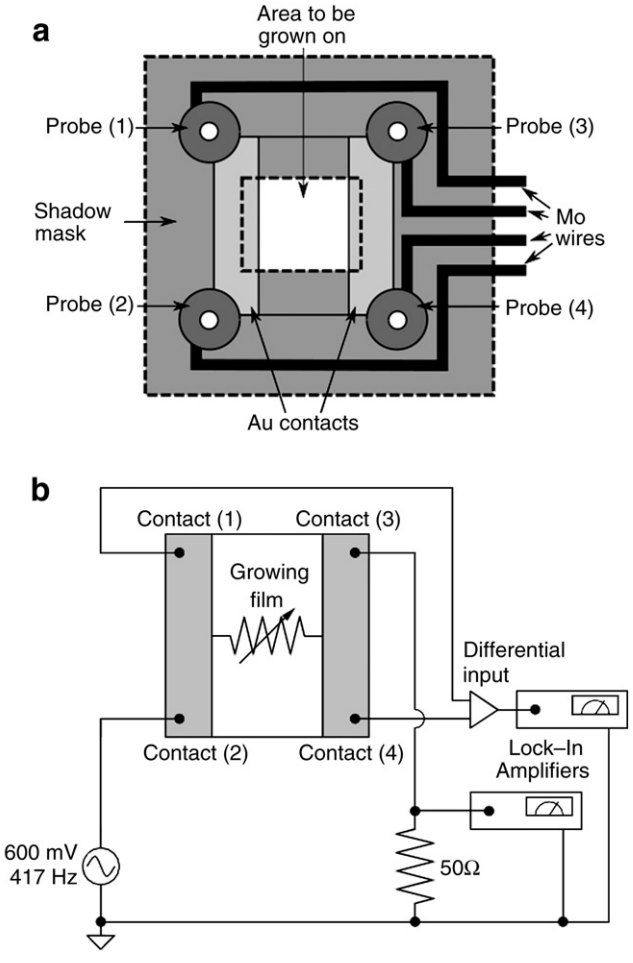

Fig. 1. (a) A schematic of the sample holder. Electrical probes are held in contact with the Au contact pads of the sample by a shadow mask. The shadow mask also protects the probes and leads from the TiN flux. (b) The dual lock-in amplifier setup used to measure the electrical resistance of the growing films. The setup is a standard four-point probe measurement with the current through the film determined from the voltage over the 50 Ω resistor and the voltage across the film measured directly across contacts (1) and (4).

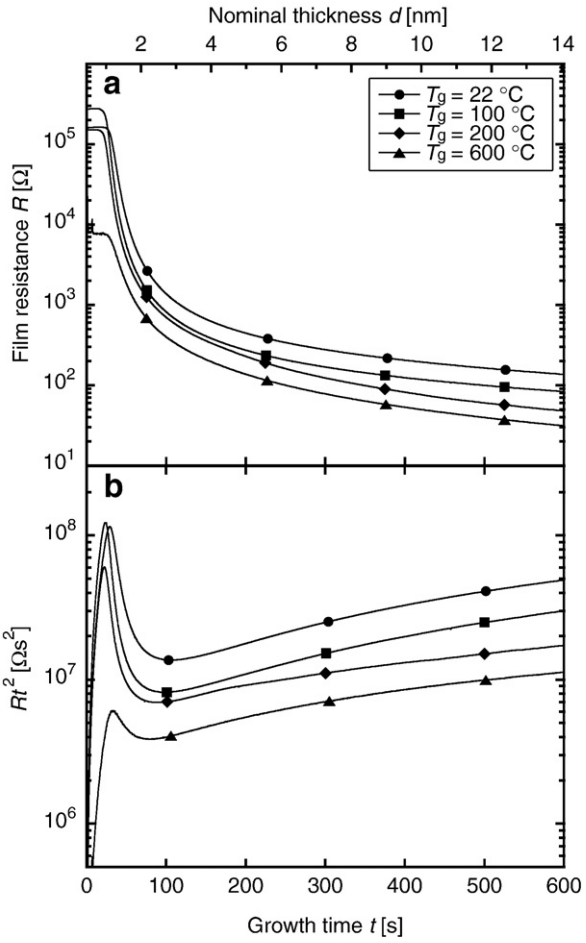

Fig. 2. (a) The film resistance R as a function of TiN deposition time t measured *in-situ* during growth, at four different growth temperatures T_g. The nominal film thickness d based on a constant growth rate is also shown. (b) Rt^2 versus growth time t. The local maximum and minimum give the coalescence and continuity time/thickness, respectively.

The differential resistance of the TiN film was measured in a four-terminal configuration during growth using a simplified version of the dual lock-in amplifier setup described by Barnat et al. [17], as shown in Fig. 1(b). The in-situ measurement system is described in detail elsewhere [16]. The nominal coalescence thickness was determined by finding the maximum of Rd^2 vs. the film nominal thickness d, where R is the in-situ measured film resistance (or equivalently Rt^2 vs. growth time t). The nominal film thickness which completely covers the substrate was determined by the minimum of Rd^2 vs. d [6,18,19].

X-ray measurements were carried out using a Philips X'pert diffractometer (Cu Kα, wavelength 0.15406 nm) mounted with a hybrid monochromator/mirror on the incident side and a 0.27° collimator on the diffracted side. The film thickness was determined by low-angle X-ray reflectivity (XRR) measurements with an angular resolution of 0.005° while structural characterization was performed using grazing incidence (GI) XRD. The GI scans were carried out with the incident beam at $\theta = 1°$. For all samples, a 360° scan of the azimuthal out of plane angle was performed at the TiN (111) peak to investigate film texture.

The growth rate was determined by growing a series of films with thicknesses ranging from approximately 7 to 40 nm. At 600 °C the growth rate was found to be constant at 0.023 nm/s for thicknesses