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SCIENCE DIRECT®
Microelectronic Engineering 82 (2005) 639–644
MICROELECTRONIC ENGINEERING
www.elsevier.com/locate/mee

Characterization of atomic layer deposited nanoscale structure on dense dielectric substrates by X-ray reflectivity

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Available online 18 August 2005

Abstract

Interfaces play a crucial role in determining the ultimate properties of nanoscale structures. However, the characterization of such structures is difficult, as the interface can no longer be defined as the separation between two materials. The high sensitivity of electron density to surface and interface reactions has attracted increasing interest in application of X-ray reflectivity (XRR) as probing technique. In this study, the nanolaminate structures were formed by atomic layer deposition (ALD) of tungsten nitride carbide (WNC) and tantalum nitride (TaN) thin films on silicon carbide (SiC), silicon oxide (SiO_2) and silicon oxynitride (SiON) substrates and subsequently characterized by XRR. The goal is to establish a relationship between surface chemistry, interface properties and density of the final structure. To achieve this objective, the density variations at the interfaces between ALD TaN and ALD WNC with SiC, SiO_2 and SiON films will be quantified by modeling analysis of XRR reflection spectra. From these modeled electron density profiles, specific mechanisms leading to surface dependent structural behavior are proposed.

Keywords: XRR; Density; ALD; Dielectrics

1. Introduction

Specular X-ray reflectivity (XRR) has already proven to be a fruitful method for studying surface and interface phenomena that result in variations of electron density with depth, which relate to the forces controlling the interface formation. The fundamental importance of interfacial characterization in thin film applications and the

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0167-9317/$ - see front matter
doi:10.1016/j.mee.2005.07.068

usefulness of XRR in achieving this are illustrated in the literature for a variety of multilayer systems [1–5] in combination with measurements of structural forces between two solids in a non-polar liquid [6–8]. Atomic layer deposition (ALD), on the other hand, is an attractive method for the fabrication of nanoscale structures for both interconnects (IC) and metal gate applications [9]. It relies on chemisorption and surface reaction by a sequential delivery of source gases from two or more vapor precursors. The process is sensitive to the chemical nature of the substrate (e.g. chemical functionality, bond architecture, etc.) of the film on which it is deposited [10]. However, on nanoscale laminate structures, the interface cannot be identified any longer as the separation between two films of bulk materials, while it defines the final composition and structure of the laminate structure. As such, the characterization of the interface becomes an important challenge.

In this work, the nanolaminate structures were formed by ALD of tungsten nitride carbide (WNC) and tantalum nitride (TaN) thin films on dense dielectrics (silicon carbide (SiC), silicon oxide (SiO_2), silicon oxynitride (SiON)) and subsequently studied using XRR. Our choice of substrate (dense instead of porous dielectric) is convenient for various reasons. For IC applications, besides surface chemistry, substrate porosity plays an important role in the growth of ALD films. Surface processes are indeed more complex when the substrate is porous. In that case, precursor molecules can penetrate into the bulk and degrade film properties. To prevent this from happening, one has to consider appropriate surface engineering approaches. Such treatments often result in the formation of a denser (as compared to bulk) surface layer, which prevents in-diffusion of precursors or gaseous species in the porous network [11]. However, this crust layer is usually SiO-like or SiC-like depending on the treatment used. Therefore, this work offers on one hand side the opportunity to study the effect of surface chemistry on the density of ALD films and on the other hand, enables a fundamental understanding of the reaction mechanisms of ALD layers with model surface systems for treated porous dielectric surfaces.

2. Experimental

2.1. Dielectric-metal film stack preparation

The starting substrates for all tests were 200 mm p-type silicon (Si) substrates. SiC films were deposited by plasma enhanced chemical vapor deposition (PECVD) directly on Si substrates without removal of native oxide. Typical film thickness was 50 nm. Thermal SiO_2 films were grown by wet oxidation at 900 °C for 20 min on hydrogen terminated silicon, Si–H, wafers. The film thickness was targeted to 20 nm. For SiON, a nitridizing plasma treatment was applied following the thermal SiO_2 film formation. The film thickness was 20 nm.

2.2. Atomic layer deposition

WNC layers were deposited in an ASM ALCVD™ Pulsar® 2000 reactor integrated with a Polygon® 8200 automated wafer handling platform. The films were deposited using a 3-precursor process with 2 s nitrogen (N_2) purges between each precursor pulse. The precursors, in process sequence, are triethylborane (TEB, $(C_2H_5)_3B$), tungsten hexafluoride (WF_6), and ammonia (NH_3). The WF_6 and NH_3 gases are diluted with nitrogen gas (N_2) and introduced in the chamber, while the liquid precursor $(C_2H_5)_3B$ is evaporated and transported by N_2 carrier gas at 175 hPa and 18 °C over the liquid. The temperature during deposition was 300 °C and the maximum pressure was 1.6 hPa. The targeted film thickness was 10 nm corresponding to 120 ALD cycles.

ALD TaN layers were deposited by alternating pulse purge steps of a 2-precursor system using pentakis(dimethylamido)tantalum (PDMAT, $Ta(N(CH_3)_2)_5$) and ammonia (NH_3) in an applied materials ALD chamber integrated on an ENDURA wafer handling platform. Purging was done after each precursor pulse with a flow of 1250 sccm Argon gas. The deposition temperature was 275 °C and the pressure during deposition was 3.3 hPa. The targeted film thickness was 5 nm corresponding to 100 ALD cycles.