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Mechanical properties of PECVD thin ceramic films

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Abstract

Silicon dioxide (thickness 350 nm and 969 nm) and silicon nitride (thickness 218 nm) films deposited on silicon substrate using plasma enhanced chemical vapor deposition process were investigated using a Berkovich nanoindenter. The load-depth measurements revealed that the oxide films have lower modulus and hardness compared to the silicon substrate, where as the nitride film has a higher hardness and slightly lower modulus than the substrate. To delineate the substrate effect, a phenomenological model, that captures most of the 'continuous stiffness measurement' data, was proposed and then extended on both sides to determine the film and substrate properties. The modulus and hardness of the oxide film were around 53 GPa and 4–8 GPa where as those of the nitride film were around 150 GPa and 19 GPa, respectively. These values compare well with the measurements reported elsewhere in the literature.
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Keywords: Ceramic films; PECVD; Nanoindentation; CSM; Modulus and hardness

1. Introduction

Ceramic oxide and nitride films are used extensively as structural members such as flexible membranes, tunable inductors, tunable optical filters, etc., in MEMS devices.[1,2] Nitride and oxide stacked layers are also used as gate dielectrics in microelectronics, in thin film transistors and in metal–oxide semiconductor integrated circuits.[3,4] In semiconductor industry, silicon dioxide (SiO_2) and silicon nitride (Si_3N_4) films are used as masking materials to isolate active circuits from each other and to provide mechanical and chemical protection during fabrication of devices using chemical vapor deposition (CVD) techniques. Further applications of nanofilm materials are envisioned in ultra small and lightweight bio- and chemo-sensors. Rare-earth oxide doped thin film coatings are also used for field emission display devices in electronic industries.[5] Coatings that alter tribological properties and extend lifetime of biological implants and computer hard disks, and patterned dichoric coatings for optical filters are some of the prime examples[6] of this emerging concept. Many of these applications require nano-thickness materials with highest quality, reproducible characteristics and reliability.

Although the principal function of thin film components are often non-structural,[7] these membranes frequently carry mirrors and operate under electrostatic forces to deflect them in response to applied bias voltage.[1] Thus, the mechanical characterization of thin films is essential for determination of structural integrity and performance. Nonetheless, the processing method associated with the manufacture of these materials often introduces residual stresses sufficient to induce mechanical deformations that can lead to performance degradation and/or malfunction in electrical, magnetic and optical properties of this class of materials.[8] Thus, characterization of mechanical deformation and understanding of failure behavior of nanofilm materials is an important first step to insure mechanical integrity, reliability under operating and adverse conditions, and proper performance of various micro- and nano-devices.

Unfortunately, the determination of mechanical properties of thin films is non-trivial because of their small dimension along the thickness direction. For many applications, the film thickness is of the order of a few tens or few hundred nanometers while the lateral dimensions can be several hundred times greater. Owing to this small thickness, the properties of these materials are significantly different than their bulk counterparts, and standard methods of testing bulk materials cannot be employed successfully at this length scale. Moreover, thin film property determination using indentation techniques uti-

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lizes very small sample volume which is also prone to influences from the substrate properties even at depths smaller than one-tenth of film thickness.[9] Also, it is now well recognized that the properties of these thin films vary with processing scheme. For example, Young's modulus of silicon nitride (Si_3N_4) thin films varies between 97 GPa and 210 GPa depending on the processing scheme.[1,4] Therefore, determination of film properties is essential whenever a new process is adopted to produce thin films. At small thicknesses, the influence of surface roughness, substrate behavior and mismatch between the film and substrate properties further complicate the mechanical characterization processes. As a result, the experimentally obtained material properties exhibit considerable scatter depending on the techniques used,[7] thus rendering the usefulness of measured parameters somewhat limited.

Numerous mechanical testing techniques, such as micro-tension tests[10,11] and micro-cantilever bend tests[12] have been used to determine the mechanical properties of such thin films. The advantage of these tests is their similarity to conventional macro-scale tests and ease of property determination. The disadvantage is the high level of cost in developing such micro-test devices and the design of test specimens. However, in recent years, instrumented nanoindentation technique has emerged as a powerful experimental alternative to the above techniques due to its simplicity and its requirement of small material volume during the test.[13–17] This technique allows precise control of either load or displacement during the test and allows for experimental measurements on small samples by subjecting them to loads as low as a few micro-Newtons and at depths in the nanometer range. Mechanical properties such as elastic modulus (E) and hardness (H) of the film can be extracted from a record of load–displacement (p–h) curves.[18–20] Characterization of a single layer deposited on a substrate has been the focus of numerous investigation.[7,15,21–28] However, the methods adopted are still under scrutiny and are not universally applicable. Most of the above investigations are also focused on metallic films and similar results on a range of ceramic films are not widely available yet. Ceramic thin films, being brittle in nature, behave in a considerably different manner than their metallic counterpart. In this article, we explore the nanoindentation technique to determine properties of thin ceramic films of various thicknesses. We also suggest a new empirical relationship relating materials properties and indentation depth that allows us to evaluate stiffness and hardness of ceramic thin films. Effect of substrate properties can be clearly delineated from that of the film using this relationship. By choosing suitable thicknesses of films and conducting systematic nanoindentation experiments at various depths, we have been able embark on a procedure to isolate the influence of the substrate and effectively determine the properties of thin films.

2. Materials

Two silicon dioxide (SiO_2) films of thickness 348 nm and 969 nm, and one silicon nitride (Si_3N_4) film of thickness 218 nm, all deposited separately on single crystal silicon (Si) wafers were obtained from Army Research Laboratories, Adelphi, MD. Each of these dielectric films was deposited using plasma enhanced chemical vapor deposition (PECVD) process in a Plasma-Therma 790 deposition chamber operating at a pressure of 900 mT and a temperature of 250 °C using appropriate precursor gases. The precursor gases for the Si_3N_4 film were silane (SiH_4), nitrogen (N_2), and ammonia (NH_3) with helium (He) as a carrier gas, and for the SiO_2 film the precursor gasses were SiH_4 and nitrous oxide (N_2O) with He as the carrier gas. Following the deposition process, the substrates were annealed at 700 °C for 60 s in flowing N_2 so as to remove trapped hydrogen from the films and to stabilize the residual stress state within the films. The film thickness was measured at 33 locations across the wafer using a multi-angle multi-wavelength ellipsometer. The wafers were then diced into quarters with one quarter diced into 4 mm × 4 mm samples and another quarter diced into 6 mm × 6 mm samples following application of an AZ5200 series photoresist.

3. Experimental approach

Nanoindentation experiments were conducted using an MTS Nanoindenter® RXP equipped with a Berkovich diamond indenter (tip radius of 100 nm). The indenter was calibrated using fused silica as the standard. On average, ten indentations were performed at each depth for six depths ranging from 20 nm to approximately twice the film thickness. The continuous stiffness measurement (CSM) option was used at each depth. This option superposes an oscillation on the load signal to continuously monitor the surface stiffness and provides data on film stiffness (E) and hardness (H) values as a function of depth (h) of indentation. The CSM signal was set to an amplitude of 2 nm at a frequency of 45 Hz. The indenter tip was set to approach the film surface at a velocity of 5 nm/s from a distance of approximately 1000 nm. Surface contact was determined by a measured increase in contact stiffness by 40–50% of the presumed surface stiffness of 80 N/m. The indenter was allowed to continue penetration on the surface at a target strain rate of 0.050/s. Once the indenter reached the prescribed indentation depth, it was held at that load for 10 s. The indenter was then withdrawn at the maximum loading rate until the load on the sample was 10% of the maximum load achieved. Around 10 indentations were performed at each depth for each film thickness. Some additional indentations were also performed at selected depths on Si substrate for comparison to the film properties. All the indentations were later interrogated using both a field emission scanning electron microscope (FE-SEM) and an atomic force microscope (AFM). The FE-SEM was used primarily for characterization of the indentations, including crack pattern determination. The AFM was used for profiling the indentations for determining the residual depths, and whether pile-up occurred around the indents.

3.1. Experimental results

3.1.1. p–h curves

Load–displacement (p–h) curves at various depths on Si_3N_4 and SiO_2 films along with those on Si substrate are shown in Fig. 1(a) and (b), respectively. For clarity, only one curve at

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