Available online at www.sciencedirect.com
SCIENCE DIRECT®
Journal of Non-Crystalline Solids 352 (2006) 84–91
JOURNAL OF NON-CRYSTALLINE SOLIDS
www.elsevier.com/locate/jnoncrysol

Effect of oxygen content on barrier properties of silicon oxide thin film deposited by dual ion-beam sputtering

Jin-Wook Seong [a,*], Dai Won Choi [b], K.H. Yoon [c]

[a] R&D 1 Team, AT Group Tech., Samsung SDI Co. Ltd., 428-5, Gongse-Dong, Kiheung-Gu, Yongin-City, Gyeonggi-do 449-547, Republic of Korea
[b] Department of Materials Science and Engineering, Carnegie Mellon University, Pittsburgh, PA 15213, USA
[c] Department of Ceramic Engineering, Yonsei University, Sudaemun-Gu, Shinchon-Dong 134, Seoul 120-749, Republic of Korea

Received 13 April 2005; received in revised form 23 September 2005
Available online 7 December 2005

Abstract

A silicon oxide thin film barrier was prepared with various oxygen contents and its chemical composition, surface morphology and optical and barrier properties were related to the deposition conditions used. Our study showed that under Ar and O_2 assisted process conditions, a stoichiometric silicon oxide thin film formed at a critical oxygen content during deposition of 40–50%. The thin films deposited at the critical condition showed the lowest surface roughness giving similar or higher optical transmittance than that of the bare polycarbonate (PC) substrate. The boiling and tensile strength test performed on the thin film deposited with assisted ions before the deposition process showed improvement in the adhesion between the oxide layer and the polymer substrate. In addition, interface modification to improve for improving the barrier layer properties of the silicon oxide thin film was achieved through the introduction of dual ion beam sputtering without pre-treatment.

PACS: 60

Keywords: Films and coatings; Sputtering; SEM; Atomic force and scanning tunneling microscopy; Absorption; FTIR; Silica; Surfaces and interfaces

1. Introduction

Recently, new deposition techniques are being developed which can deposit oxide barrier thin films with lower oxygen permeability as measured by oxygen transmission rate (OTR) analysis [1]. The thin film properties required to obtain such a barrier layer can be classified into three different parts. First, the surface properties, including surface roughness, porosity, scratch, erosion, and corrosion resistance, need to be enhanced. Secondly, the barrier layer needs to have a stoichiometric composition with an optical refractive index similar to that of bulk substrate. Thirdly, interfacial properties between the substrate and the barrier layer including interface bonding, residual stress, and adhesion need to be improved. In addition, the polymer substrate properties needs to be well-matched with that of the oxide barrier layer [2].

With dual ion beam sputtering, the requirements listed above can be achieved through effective control of deposition parameters [3,4]. Dual ion beam sputtering under vacuum can enhance chemical bonding through energy supplied by the ion bombardment that increases the collisions between ions or atoms. In addition, the RF plasma effect can be separated from the sample and will confine ion beams between the target and the sample [5,6]. Therefore, dual ion beam sputtering utilizing ion beam assisted deposition conditions can improve the adhesion, density, stoichiometry, and low optical absorption (at short wavelengths) in thin films.

The formation of columnar and micro-porous structure, poor stoichiometry, and adhesion decreases the quality of thin film [7–9]. Therefore, ion bombardment was applied

* Corresponding author. Tel.: +82 031 288 4499; fax: +82 031 288 4481.
E-mail address: jinwook.seong@samsung.com (J.-W. Seong).

0022-3093/$ - see front matter © 2005 Elsevier B.V. All rights reserved.
doi:10.1016/j.jnoncrysol.2005.10.024

during deposition to surmount such adverse effects [10,11]. Here, ion bombardment during deposition increases the density [12] and prevents the formation of columnar and micro-porous morphology during thin film growth [1]. In addition, ion bombardment using a reactive gas like O^+/O^{2+} can improve the stoichiometry of the as-deposited oxide thin film and can enables the control of the properties dependent upon it. Low-energy reactive ion beam (inert, reactive or mixed ion) bombardment can increase the rate of compound formation, control the stoichiometry and improve adhesion of the deposited thin film [13–15]. Ion bombardment enhances adhesion by producing adhesion sites on the surface which control both stoichiometry and optical property of the oxide thin film deposited [16,17]. Therefore, our study is mainly concentrated on observing the surface, bulk, and the interfacial properties related to the assisted ion content as a deposition parameter for improving the barrier layer properties. In addition, improvements in the surface properties of the oxide thin films deposited by an ion beam assisted process, similar to that employed as a pre-treatment step prior to deposition, have been investigated.

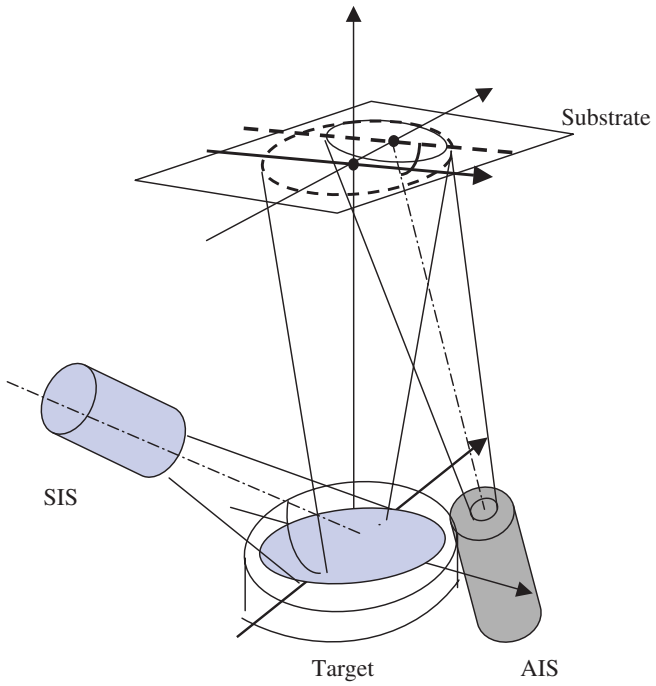

Fig. 1. The schematic view of dual ion beam deposition (DIBD).

2. Experimental procedure

2.1. Chamber equipment and deposition

Two cold hollow cathode type ion guns were attached to the deposition chamber where one of the ion guns generates particle flux from the solid sputtering target and the other assisted the thin film growth by generating various contents of inert or reactive ions. The incident angle of the ion beam used for sputtering the solid target was 50° and the assisted ion-beam source (AIS) was generated by a cold hollow cathode ion source attached to the target holder inside the vacuum chamber. The assisted ion gun is placed at the axis of the thickest point of the deposited thin film with incident angle of 40°. Fig. 1 shows the schematic diagram of a duel ion beam deposition (DIBD) setup. Due to the AIS ion beam current distribution, the homogeneous thin film deposition area is reduced to a circular region of approximately 30 mm in diameter. A movable Faraday cup was used to control the ion beam current at the center of the target. Rotary and turbo molecular pumps were used for vacuum systems where a Pirana and ion gauge were used to measure low vacuum level ($\sim 10^{-2}$ Torr) and high vacuum level ($\sim 10^{-6}$ Torr), respectively. The in situ deposition rate control was possible by placing a quartz crystal oscillator near the substrate.

An ion-assisted process including Ar–O_2 assisted process was applied to obtain silicon oxide thin film using a Si (diameter: 150 mm, thickness: 6 mm, purity: 99.999% (5N): high purity chemical, Japan) target with 1 keV Ar ion bombardment. To control and maintain the deposition condition, the total current and density of SIS was measured using a Faraday cup whereas the deposition rate and thickness was controlled by using a quartz oscillator. For ion assisted bombardment, oxygen (O^+ and O^{2+}) and argon (Ar^+) ion were used. The base pressure of the deposition chamber was 5×10^{-6} Torr but when SIS and AIS was applied, the pressure was 2×10^{-4} Torr and 4×10^{-4} Torr, respectively. The surface oxide layer on the target was removed by Ar ion bombardment at 400 eV for 20 min. Both PC (polycarbonate) and silicon wafers were used as substrates for different analyses. The silicon oxide thin film deposited on the PC substrate was used for characterizing chemical composition (XPS), surface roughness, structure, optical property, and oxygen permeability whereas a silicon substrate was used for refractive index, deposition rate and thickness measurement. The 185 μm thick PC substrate was first cleaned with alcohol after removing the cover film to prevent electrostatic charge built up and then blown dried using N_2 gas to prevent scratching of the surface. The p-type silicon wafer substrate was cleaned by immersion in HF solution to remove the native surface oxide layer followed by sonicating in acetone and de-ionized water for 15 min each. SIS total ion beam current was 35 mA with current density of 1 mA/cm^2. The experimental parameters for the Ar–O_2 assisted processes are summarized in Table 1.

2.2. Analyses and measurements

In order to control the deposition rate, the thickness of the thin film deposited by Ar–O_2 assisted process for equal time period was measured on the cross-sectional image using scanning electron microscopy (SEM: S-4200FE, Hitachi Co.). For chemical composition analysis for silicon oxide thin film, Fourier transformed infrared (FT-IR)