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A comparative study on the measurement of toughness of stacks containing low-k dielectric films

Z.W. Zheng [a], I. Sridhar [a,*], S. Balakumar [b]

[a] School of Mechanical and Aerospace Engineering, Nanyang Technological University, Singapore 639798, Singapore
[b] Semiconductor Process Technologies Laboratory, Institute of Microelectronics, Singapore 117685, Singapore

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ABSTRACT

Good adhesion strength of thin film stack is desirable for the integrated circuits (IC) manufacturing stage such as chemical mechanical planarisation (CMP). The scratch test and four-point bending test are two most commonly used methods to find the adhesion properties of thin film because of their simplicity and quantifiability. In this research, scratch test is used to find the practical adhesion energy (W_{pa}) of thin film stack and four-point bending test is used to find the critical strain energy release rate (G_c). The comparison of W_{pa} from scratch test and G_c from four-point bend test reveals that, for the same thin film stack, W_{pa} value is comparatively higher than G_c. The scratch test is semi-quantitative, in that the normal load at which a predefined failure event or delamination occurs is defined as a measure of adhesion. However, the four-point bending test is more quantitative in finding the adhesion properties. Thus, for practical application, such as CMP process, the G_c measurements from four-point bending test are recommended as references.

1. Introduction

Microelectronics integrated circuit (IC) chips consist of several layers of material having the functionalities of conductivity, insulation, semi-conduction, capacitance, etc. These layers are manufactured through various process routes, such as physical vapor deposition (PVD), chemical vapor deposition (CVD), ion implantation and metallization. The IC manufacturing process contain several stages including mechanical polishing. Delamination and peeling of film stacks during chemical mechanical planarisation (CMP) process is one of the challenging issues in the fabrication of semi-conductor interconnects [1–4]. With decreasing size of logic devices and increasing speed, the resistance–capacitance (RC) delay time becomes a crucial design parameter as it limits the overall performance of very large scale integration (VLSI) circuits. To improve the RC delay low-k and ultra low-k materials with k value less than 2.5 are proposed and experimented with recently. There is an increasing interest in enhancing the mechanical properties of the low-k films, since they effect the performance of the device during chemical–mechanical polishing (CMP) and other integration processes. The delamination is mainly due to poor adhesion strength, accumulation of residual stress in the film stack due to various process conditions. Good interfacial strength between the layers of film stack is desirable for the reliable performance of multi-layered microelectronic integrated circuits. An understanding of adhesion strength degradation at the interface of barrier layers and interconnect metals or dielectrics is necessary to develop a robust process without the occurrence of peeling and delamination.

A number of thin-film adhesion assessment methods, such as peel, indentation, blister and Brazil-nut tests, have been developed to measure interfacial properties [5,6]. While many require difficult sample preparation methods, as exemplified by the blister and edge-delamination test, scratch test and four-point bending test require limited, if any, preparation to induce and quantify interfacial failure. In this research, microscratch test and four-point bending test are used to quantify the adhesion energy between interfaces in multi-layered thin film structures. Herein, no attempt has been made to study the factors influencing the interfacial adhesion. Only a comparative study on the measurement of work of fracture from microscratch and four-point bending tests is carried out.

Scratch test is one of the commonly employed test for metals, polymers for the evaluation of scratch hardness or resistance, modelling of abrasive wear, etc. and it is currently being employed to provide a measure of coating/substrate adhesion. The recent special issue of *Tribology International* [7] is specially devoted to the developments in 180 years old scratch test technique. The adhesion measurement from a scratch test is influenced by a number of intrinsic and extrinsic factors which are not adhesion related and the results of the test are usually regarded as

* Corresponding author. Tel.: +65 67904784; fax: +65 67911859.
E-mail address: msridhar@ntu.edu.sg (I. Sridhar).

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semi-quantitative [8]. The critical load determined by this method is widely regarded as being representative of coating adhesion behavior [9–13]. It has been used to compare the performance of different coating system on similar substrates and/or of similar coating systems on different substrates. While extracting a pure measure of adhesion from the measured critical load is difficult, as it depends on the testing parameters (e.g. loading rate and stylus tip radius) and sample parameters (e.g. coating thickness and substrate hardness), it is possible to calculate the practical work of adhesion W_{pa}. The practical adhesion signifies the work or force required to remove or detach film from the substrate, which includes the energy required to deform both the the film and the substrate, as well as the energy dissipated as heat or stored in the film, and the component representative of actual fundamental adhesion [5]. Four-point bending test is one type of sandwich specimen tests, in which a macroscopic fracture mechanics sample is made with a thin film incorporated into the test structure [14,15]. This test is a modification of classical fracture mechanics test.

There are a number of different mechanisms of adhesion which can be summarized as mechanical interlocking; diffusion of one component into the other; electrons transfer and adsorption (by van der Waals forces, hydrogen or chemical bonds). At the outset, it is to be clarified that the objective of the study is not to investigate the adhesion mechanisms in the film stacks, but rather to identify an appropriate experimental tool to choose in quantifying the interfacial toughness measurement for thin-film stacks. In this paper, a few multiple film stacks that are commonly used in microelectronic industry are tested for their adhesive strengths using continuous microscratch and four-point bend testing. The values of practical work of adhesion measured from scratch test are compared with the critical strain energy release rate of four-point bend tests. The toughness values set an upper-limit on the pressure that should be applied in the polishing stage of these film stacks.

2. Experimental procedures

2.1. Sample preparation

The samples used in the experiments, as listed in Table 1, consists of different thin films deposited on a 8-inch (100)-oriented p-type Si wafer substrate. Various deposition techniques are employed to produce the multiple film stack. The blanket films of undoped silicate glass (USG) and low-*k* dielectric material black diamond (BD) were deposited onto the silicon wafer using the plasma enhanced chemical vapor deposition (PECVD) technique. The barrier layers of Ta and TaN were deposited using self-ionized metal plasma (SIP) technique. The low-*k* dielectric porous-silk (P-silk) was deposited by spin-on coating technique. Copper seed layer was deposited using SIP technique, and it was followed by the deposition of electrochemical plating (ECP) of thick copper.

The wafers coated with thin film stacks were then diced into testing specimens with the size of 10 mm × 10 mm (type I) for scratch test and 46 mm × 7 mm (type II) for four-point bending test. The disco diamond blade (model NBC-ZH 104F-SE) was used in conjunction with the Disco Automatic Dicing Saw Machine (DAD651) to carry out the dicing process. The type I specimens can be directly used for scratch tests. While type II specimens need further to be glued to bare silicon of the same dimension by an epoxy. Type II specimens were then cured at 100 °C for 1 h and allowed to dry by cooling them in the furnace itself to room temperature. After this procedure there was no warpage of the specimen. The total thickness of each prepared sample, including two substrates, epoxy and thin film stack, is 1.45 ± 0.02 mm. Then the individual four-point bending samples were centrally notched with the same dicing machine to about 100 μm to the thin film interface.

Table 1
Experimental samples used in the scratch test and four-point bending test

Sample	Substrate/thin film stack
A1	Si/8000 Å USG
A2	Si/8000 Å BD
B1	Si/8000 Å BD/500 ÅTa
B2	Si/8000 Å BD/500 ÅTaN
C1	Si/5000 Å p-silk/250 ÅSiC/250 ÅTa/500 ÅCu
C2	Si/5000 Å USG/250 ÅSiC/250 ÅTa/500 ÅCu

2.2. Scratch test

The scratch tests were done using the NanoTest™ manufactured by Micro Materials Ltd., Wrexham, UK, and operated in the increasing normal load mode. In a typical scratch test a diamond stylus is drawn across the film surface with increasing normal load. A normal load is applied to the tip during scratching until the coating detaches from the substrate. However, in the scratch module of NanoTest™, the sample surface is moved perpendicular to the diamond probe axis allowing the abrasive wear resistance of a coating to be investigated by either single or repetitive scratch tests. Accurate repositioning combined with optional software enables complex multi-pass scratch tests to be scheduled with much greater precision than a conventional scratch tester. The diamond stylus used in the scratch test is sharp Rockwell indenter (conical with spherical tip) with a spherical apex of 25 μm. The sample surface is moved tangentially at a speed of 0.5 μm/s against the indenter and at the same time, normal load is applied at a rate of 0.5 mN/s. The normal load, tangential friction load and scratch distance are continuously recorded.

In this method, the indenter both initiates and propagates the delamination. The scanning electron microscopy (SEM) observations illustrate the features of scratch track. Comparison of the SEM micrographs with the load–scratched distance plots reveals the critical load P_c^s at which the film debonds from the substrate. Then the critical load P_c^s is used in the calculation of the practical work of adhesion W_{pa}. The relationship between the critical load and the practical work of adhesion can be expressed as [14]

$$W_{pa} = \frac{2h_f}{E_f}\left(\frac{P_c^s}{\pi r^2}\right)^2, \tag{1}$$

where r is the probe radius, h_f and E_f are the film thickness and Young's modulus, respectively. Young's modulus E_f can be measured by the continuous stiffness measurement (CSM) technique of nanoindentation test. The CSM allows the measurement of contact stiffness at any point along the loading curve and not just at the point of unloading as in the conventional measurement. The measured Young's modulus is a function of the indentation depth, which is theoretically analyzed [16]. In the calculation using Eq. (1), an average value of E_f is selected.

2.3. Four-point bending test

The model of four-point bending test is shown in Fig. 1. The film stack on Si substrate is attached to another Si substrate with an epoxy glue. The upper substrate has a notch in the center. A crack propagates downward from the notch and through the upper substrate, then kinks into the thin film stack. At the weakest interface of adhesion strength, the thin film system will delaminate horizontally. At this point the strain energy release rate (G) reaches steady state, which corresponds to the load plateau (P_c^f) in the load–displacement curve. The critical strain energy release rate (G_c) can be calculated from the critical load P_c^f by [17]