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CROSS-SECTIONAL NANOINDENTATION: A NOVEL TECHNIQUE TO MEASURE THIN FILM INTERFACIAL ADHESION

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ABSTRACT

Thin film interfacial adhesion is a critical material property in assessing the thermo-mechanical reliability of microelectronic components. Cross-sectional Nanoindentation (CSN) is a novel technique developed to characterize the adhesion of thin film interfaces. The technique consists of indenting a cross-sectional sample with a Berkovich diamond indenter using a Nanoindenter System. The indentation is made normal to the cross-section at a specific distance from the interface of interest. This produces a controlled bending of the thin film structure. The onset of interfacial delamination is related to sudden steps in the load-displacement curve. From optical and SEM micrographs, the delamination crack paths are directly observable. Based on the crack lengths, a relative determination of interface fracture toughness can be made. CSN results correlate to fracture toughness values obtained with the four-point bending technique(3).

KEYWORDS

Cross-sectional Nanoindentation (CSN), Interfacial adhesion, Microelectronics, Thin film, Berkovich indentation

INTRODUCTION

The microelectronics industry is running on an increasingly complex treadmill that requires novel materials to be integrated to meet electrical performance targets. The path to success requires the integration of new metal and dielectric interconnect materials that may increase the risk of thin film delamination. Lack of chemical affinity and large differences in thermal expansion coefficients of the various thin films, together with the presence of defects or residues at the interface, are some of the causes of interfacial delamination.

It is of critical importance that accurate, quantitative techniques are available to assess the mechanical integrity of thin film interfaces. More sophisticated methods are needed to replace older techniques such as the tape test and stud pull test (1-2) which are not generally applicable. The technique of four-point bending has recently been used to obtain quantitative values of thin film interface fracture toughness (3-5). However, lengthy sample preparation is required and the technique is limited to blanket thin film samples.

Nanoindentation has been widely used to measure materials properties such as hardness and modulus(6-8). It has also been used to study delamination by top-down indentation of thin films(9). However, the interface at which delamination occurs is not clearly distinguishable. CSN represents the first application of the indentation technique to the study of thin film interface adhesion using cross-sectional samples. It allows direct observation of the delaminated interface. Due to the simplicity of sample preparation and the quick turn-around time (approximately four hours), CSN has the potential to be used as a quick-turn monitor for the fabrication engineers. A significant advantage of the technique is its application to patterned as well as blanket thin film samples.

EXPERIMENTAL PROCEDURE

Sample preparation

Blanket thin film samples consisting of 1 μm silicon nitride on 1 μm silicon oxide were studied. The silicon oxide was deposited using a chemical vapor deposition (CVD) process. The silicon nitride thin films were deposited using various processes: plasma-enhanced chemical vapor deposition (PECVD), high density plasma (HDP) and low deposition rate CVD. A test chip was analysed as part of CSN tests on patterned material. This consisted of a two metal layer integrated short loop with silicon nitride passivation, polyimide (PI) and Controlled Collapsible Chip Connectors (C4) bumps.

Sample preparation was a simple cross-sectioning using diamond scribing to initiate a precrack then cleaving with glasscutters' pliers. This produced a clean and flat cross section ready for indentation. In the case of the patterned material, the cleave was made through metal lines near the die's edge to determine if they would arrest cracks due to corner blunting.

CSN test procedure

The CSN test configuration used for the silicon nitride /silicon oxide blanket samples is illustrated in Fig. 1. The orientation of the three-sided Berkovich diamond tip and its positioning with respect to the interface are critical parameters for controlled delamination. The optimum orientation of the diamond tip is that depicted in the figure, where one of the sides of the triangular indentation mark is parallel to the interface. The optimum distance to the interface (d) is 1 to 5

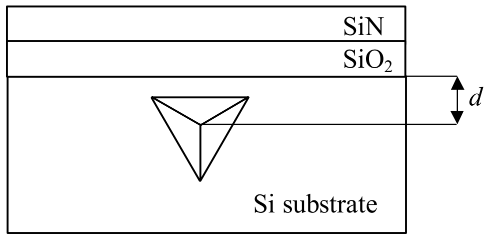

Figure 1: Thin film structure and orientation of Berkovich indenter with respect to thin film interface.

micrometers. The optimum load range for delamination was found to be 30 mN to 200 mN.

Indentations were made using a well-calibrated Berkovich diamond indenter (Nanoindenter II and Nano XP, MTS Nano Instruments, Inc.) with a load resolution of about 50 nN and a z-axis displacement resolution of 0.01 nm. The resolution of the diamond tip positioning system in the x-y directions is 0.5 µm. CSN tests were carried out using strain-rate control with a tip displacement rate of 10 nm/s. Load vs. tip displacement curves were recorded during the tests (data acquisition rate: 45 Hz). After the indentation experiments, SEM micrographs were collected to measure crack lengths and delamination areas.

CSN Fracture Interpretation

A 3D view of the CSN experiment is shown in Figure 2. Figure 3 shows a SEM image of the indentation zone. Cracking begins at the two corners of the indentation that are closer to the interface. These radial cracks, characteristic of brittle materials loaded with pyramidal indenters(10-11), propagate on loading through the silicon substrate and the strong silicon/silicon oxide interface, producing a wedge (shown in Fig 2).

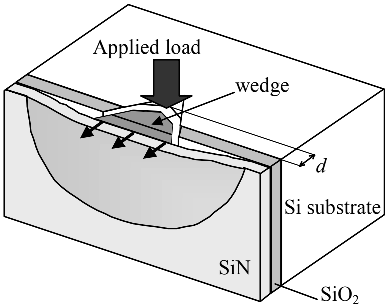

Figure 2: Sample cross-section showing location of applied load and bending of thin film structure producing delamination

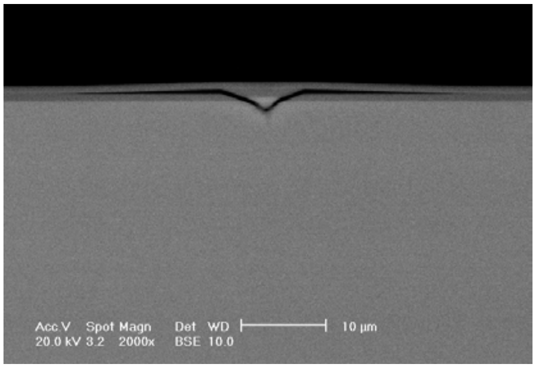

Figure 3: SEM micrograph of silicon nitride/silicon oxide thin film sample after cross-sectional indentation.

However, when the cracks reach the weak silicon oxide/silicon nitride interface, they tilt out of their original planes following the Si_xN_y/SiO_2 interface.

Delamination produces a sudden movement of the diamond tip that is registered as a step in the load vs. tip displacement (Fig. 4). Such a step is not detected when the maximum indentation load is lower than that required for delamination. In this case, the load vs. tip displacement curve is similar to that obtained in a hardness test of silicon.

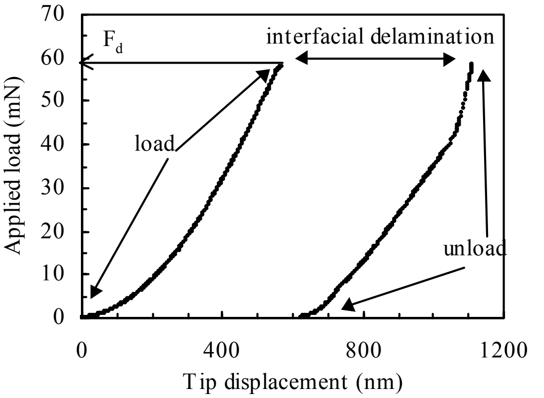

Figure 4: Load-displacement curve showing sudden jump corresponding to thin film delamination.

RESULTS

Blanket Thin Film Analysis

Adhesion of silicon nitride to silicon oxide blanket thin films was characterized for three nitride deposition processes, plasma-enhanced chemical vapor deposition (PECVD), high density plasma (HDP) and low deposition rate CVD. For each process, results with and without a plasma pretreatment are compared. The crack lengths at the interface were measured after indenting to a load of 100 mN. Results are given in Table 1, showing that the PECVD nitride deposition process produces the strongest interface. In this case, the interface could not be debonded. In general, CSN results are well-correlated to adhesion results obtained with the four-point bending technique(3).

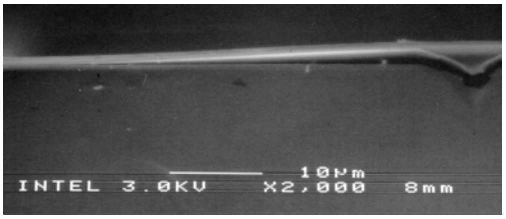

Figure 5a: CVD Silicon Nitride; crack Length = 27 μm

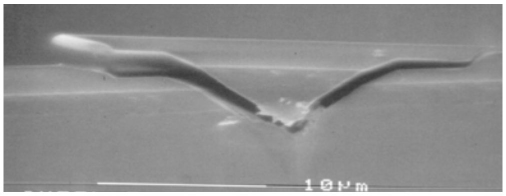

Figure 5b: HDP Slicon Nitride; crack Length = 4.7 μm

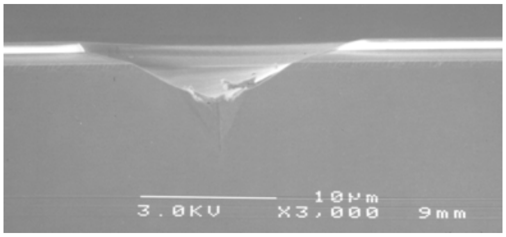

Figure 5c: PECVD Sillicon Nitride; no debonding

Table 1: Comparison of CSN and four-point bending adhesion results for silicon nitride/silicon oxide samples with different processing conditions.

Nitride Dep Process	CSN Crack Length (um)	Four-point bend interface energy (J/m2)
CVD (Fig 5a)	28	2.84 ± 0.93
HDP (Fig 5b)	4.7	7.23 ± 0.97
PECVD (Fig 5c)	No debond	No debond

Patterned Thin Film Analysis

CSN measurments were carried out on a patterned test chip. The purpose of the study was to characterize polyimide thin film to silicon nitride thin film adhesion and the effect of metal lines on delamination at this interface. Indentations were made in the silicon substrate in two locations: in an open area and directly below the edge of the metal lines. The thin film stack in the open area consists of polymer / silicon nitride / silicon oxide / silicon substrate and in the locking structure location consists of polymer / silicon nitride / patterned aluminum /silicon oxide / silicon substrate.

SEM results for an indentation made in an open area of the scribeline are shown in Figure 6. Note the symmetry of debonding. This shows that the crack initially propagates to the silicon nitride / silicon oxide interface before jogging into the weaker polymer / silicon nitride interface. Figure 7 shows SEM results for the indentation made below the patterned metal lines. Note that the cracking is asymmetric. On the left side, the crack is shorter, being arrested due to corner blunting of the patterned metal line. The crack initially propagates to the metal / silicon oxide interface and then to the polymer / silicon nitride interface before stopping at the edge of a metal line.

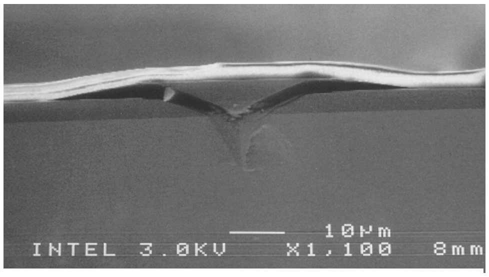

Figure 6a: Low mag image: CSN in open area

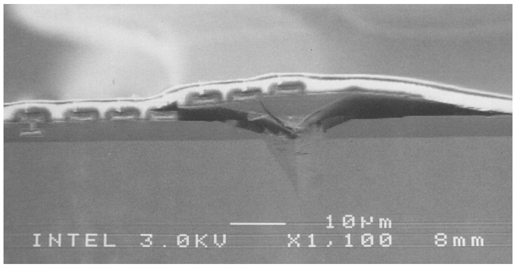

Figure 7: CSN below patterned lines

The repeatabilty of the indentation results was measured by conducting multiple CSN tests on samples from the same wafer. Tests were carried out under identical loading conditions (100 mN) and at the same distance from the Si/ silicon oxide interface (3±1 µm). Wafer-to wafer repeatability was also tested by conducting tests on different wafers from different lots. The results are consistent, showing that the crack length at the polymer / silicon nitride interface is significantly lower in the case of indentation made below patterned metal lines. Table 2 summarizes results.

TABLE 2: CSN REPEATABILITY RESULTS FOR PATTERNED MATERIAL

Sample ID	Crack Length (µm) for indent below patterned lines	Crack Length (µm) for indent in open area
Lot 1, sample 1	1.25	18
Lot 1, sample 2	2.5	16
Lot 1, sample 3	2	8
Lot 2, sample 1	2	12.5
Lot 3, sample 1	2	5

Discussion/Conclusions

Cross-sectional nanoindentation results for blanket thin film samples clearly show that the technique is capable of resolving differences in adhesion strength. Using crack length at the interface as a relative measure of adhesion strength, it has been shown that CSN results correlate well with four-point bend results. In order to obtain quantitative values of interface fracture toughness using the CSN technique, modeling is required. A model based on the elastic plate theory has been developed and applied to ceramic-ceramic systems(12). In order to make the CSN technique fully quantitative, further model refinements are needed.

The application of the CSN technique to patterned thin film samples shows the great value of the technique in distinguishing differences in delamination behavior due to local geometry effects. The direct observation of crack blunting at patterned metal lines provides compelling evidence of the effectiveness to stop crack growth at the polymer to ceramic interface.

The electronic industry's silicon and packaging processes will need to fully comprehend thermal mechanical issues. As devices become faster and hotter and as novel materials are incorporated into an increasing number of interconnect layers, a comprehensive understanding of interfacial adhesion is critical. The cross-sectional nanoindentation technique has been demonstrated to be a reliable test method. Its advantages are ease of sample preparation, quick turn-around time, direct observation of delamination, and application to patterned and blanket thin films.

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