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Application of Nanoindentation to Characterize Fracture in ILD Films Used in the BEOL

Eva E. Simonyi, E. Liniger, M. Lane, Q. Lin,
C. D. Dimitrakopoulos, C. Tyberg
IBM Research Division
Thomas J. Watson Research Center
P.O. Box 218
Yorktown Heights, NY 10598

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Abstract

It is of importance to understand cracking behavior in low dielectric constant, low modulus materials. Nanoindentation method is presented as a tool to estimate the critical film thickness, thickness above which spontaneous cracking could occur, for ILD films used in the BEOL. The critical film thickness was then used to calculate cohesive energies and fracture toughness of the films. Materials were investigated using nanoindentation combined with AFM imaging. The results were compared to data acquired by four point bend methods.

Introduction

The drive in the semiconductor industry for ever decreasing feature sizes in the BEOL drives the requirement for materials with ever decreasing dielectric constants. The materials with the lowest dielectric constants exhibit poor mechanical characteristics: low modulus and significant cracking propensity. Due to the large number of materials that are evaluated for the manufacturing process, there is a need to investigate the mechanical characteristics with a fast turnaround time in order to support the efforts of optimizing these materials to achieve low dielectric constants with robust mechanical characteristics. Traditionally cohesive energy of thin dielectric films is measured with 4 point bend(1) measurement setup, stress with a beam bending rig(2), and modulus and hardness by nanoindentation. The motivation for developing this method was to have a measurement method, which does not require time consuming sample preparation as is required for 4 point bend measurements, and will give data that agree with 4 point bend.

Experimental

Hardness and modulus were measured with a Nanoindenter XP system (Nano Instruments Innovation Center) fitted with the dynamic contact modulus (DCM) head. The DCM head provides the XP system with an overall miniaturization, allowing to perform indentations at maximum indentation forces of .01 to 12mN. The DCM machine uses a Berkovitch indenter. This is a 3 sided pyramid with 65.3° between vertical axis and face. The instrument was operated using the continuous stiffness measurement option (CSM). This method superimposes a small oscillating force on the applied load, allowing a continuous measurement of the hardness and modulus during the indentation process. Tip calibration was based on the Oliver –Pharr (3) method. The indentation was done maintaining a constant strain rate. Surface was detected by a stiffness change of 4. The stress was measured with a beam bending apparatus - applying

Stoney[4] equation- The measurement error is about 10 percent. This method requires the measurement cracks created by indentation at a given load. This load is not arbitrary: the lowest value is about 10 percent higher than the force necessary to initiate a crack, and less than the force, that would cause irregular cracking or delamination from the substrate. For the crack creation CSIRO (Australia) UMIS indentation instrument was used, with a corner cube indenter as the indenter diamond. The cube corner indenter is a three sided pyramid with 35.1° between the face and the vertical axes. The advantage of this sharper diamond vs. the Berkovitch is that it creates bigger cracks at the same indentation force; it displaces more material at the same load. This is important, because it allows minimizing the influence of the substrate and allows the measurement of the crack lengths to be more accurate. Constant loading speed was maintained for the crack creation. Typical indentation load vs. force is shown in Figure 1.

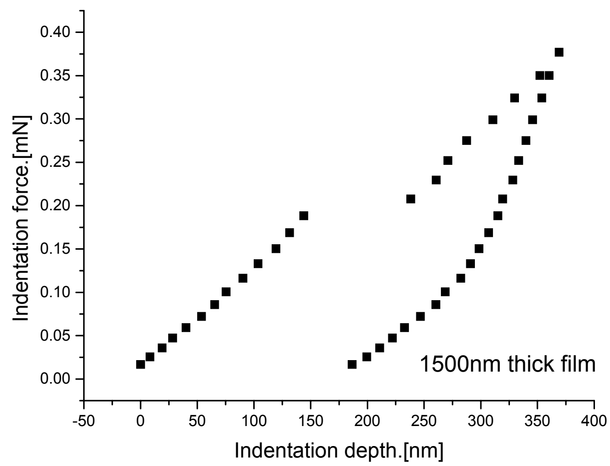

Figure 1 Indentation force vs. indentation depth with typical crack discontinuity.

Crack lengths were measured with a Digital Instruments Atomic Force Microscope. (AFM). Minimum 5 indents were taken for each load.

The crack length is defined here as the distance from the middle of the indent. Only well defined cracks, symmetrical ones around the corner cube indenter were used. [Most indents were these types, and the accompanying load vs. indentation depth graphs were also very similar to each other. The crack initiation values stayed also consistently the same for the same film.] The crack length measurements were performed within 5-10 minutes after the indentation itself was finished, in order to avoid crack extension due to crack corrosion, crack extension due to moisture. A typical AFM image of a crack is shown in Figure 2. Average error of the crack length measurements is ~10% or less.

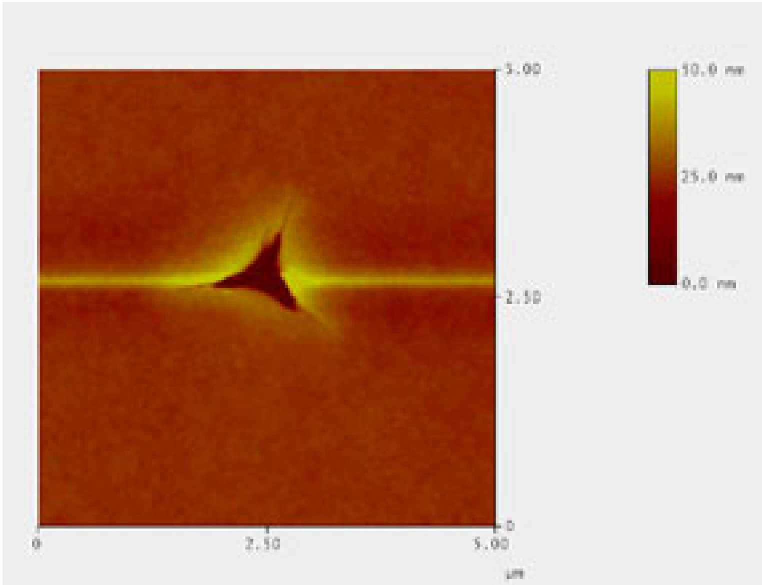

Figure 2 Typical AFM image of a corner cube indent of a 1500nm thick film at .3mN indentation load.

The advantage of using AFM to image the cracks is that it shows very clearly any irregularities, delaminations from the substrate.

Data and discussion

Fracture toughness (K_c) of films can be correlated to indentation induced film cracking by the following equation:

$$Kc = \beta\left(\frac{E}{H}\right)^{.5}\frac{F}{c^{1.5}} \quad ^{(5)} \quad (1)$$

where c =crack length, E=modulus, H=hardness, F=indentation force, and β is a constant related to the indent and to the indenter geometry.
The mechanical robustness of films can be characterized by cohesive strength:

$$J_{cohesive} = \frac{K_c^2}{E} = Z\frac{\sigma^2 h}{E} \quad (2)$$

where Z is a constant depending on the modulus and Poisson ratio of the substrate and the film, here ~1.3, σ is the film stress as measured in the bending rig, and h is the film thickness.
As the film thickness is increased the strain release energy exceeds the cohesive energy and spontaneous cracking occurs. This specific film thickness is defined here as the critical film thickness (h_c). The assumption is that this critical film thickness

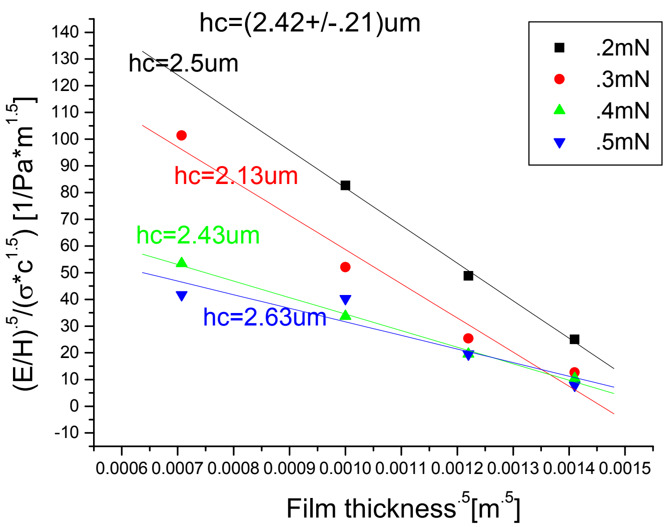

Figure 4. Material A. $(E/H)^{.5}/\sigma\ c^{1.5}$ vs. $h^{.5}$

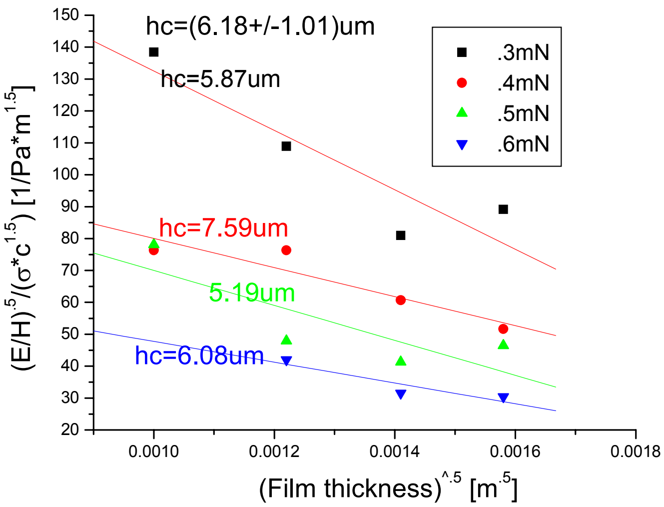

Figure 5. Material B. $(E/H)^{.5}/\sigma\ c^{1.5}$ vs. $h^{.5}$

can be estimated by calculating the film thickness at which the indentation induced crack length approaches infinity. When plotting the square root of the film thickness at a given indentation force verses constant $* \frac{1}{c^{1.5}}$, of the linear (or exponential) fit to the data will give the desired critical film thickness (h_c) as the crack length approaches infinity. Both films investigated were blanket films deposited onto Silicon wafers, the film thickness ranged between 500 and 2500nm. Figure 4 (material A) and Figure 5 (material B) are examples of these fits.

Film mechanical characteristics are compared for films A and B (Table 1). Sample A has a higher stress and a higher modulus as compared with sample B. The critical film thickness (h_c) and cohesive strength (J) calculated from both indentation induced crack length measurements and 4-point bend measurements are compared for the two samples (Table 2).

Sample	σ [MPa]	E [GPa]	H [GPa]
A	44+/-7	3.2+/-0.2	0.47+/-.02
B	32+/-3	2.06+/-0.2	0.35+/-.02

Table 1. Stress, modulus, hardness values.

Sample	hc_{nano} [um]	$hc_{4\text{-point}}$ [um]	J_{nano} [J/m^2]	J_{4point} [J/m^2]
A	2.42+/-0.21	2.94+/-0.3	2.6+/-0.4	3.2+/.03
B	6.18+/-1.01	7.7+/-0.8	3+/-0.3	3.6+/-0.1

Table 2. Comparison result of 4-point bend and nanoindentation results for samples A and B.

Using the load verses indentation curve and the observed crack length one can estimate the work done by the indenter in the process of creating a crack. Depending on the loading force, the film thickness, and the material itself, the range of work values for the work done by the indenter is about 0.1-0.5 J/m^2 These values are smaller than the STD of the measurements so they were not taken into account.

The equation (1) was originally developed for bulk materials, by Lawn [(5)], for half penny shaped cracks, but loading force and film thickness combinations that avoid largely the substrate effects allow the use of this equation for thin films, approximating them as bulk materials.

Even though the indentation depths at the maximum applied indentation forces were kept at less than 30% of the total film thickness, the substrate effect cannot be ruled out; especially if one goes to the higher indentation forces Therefore, it is important to keep the indentation forces somewhat above, but close the crack initiation forces, additionally as thick as possible films should be used in order to create radial cracks with little penetration into the film. Some of the radial cracks originate from under the surface. These radial emanating cracks never propagate toward the substrate, but to the surface of the film. This kind of behavior is typical for materials that have low dielectric constants; that are porous as these films are, and very brittle. Exception to this observation is the case, when the indentation force applied is high enough to create not only cracking, but substrate film delamination. These types of indents were never used.

Despite the slight influence from substrate effect on nanoindentation calculations, the data shows acceptable agreement, within the estimated standard deviation value, between the two methods for both critical film thickness measurements and cohesive energy, verifying the validity of the nanoindentation method. These results also indicate that there is significantly different cracking behavior for the two samples evaluated. The values deduced from the nanoindentation method can be called a practical critical thickness value: during CMP external work creates cracks in films with thicknesses less than the critical film thickness, similar to cracks generated by nanoindentation.

Summary

Corner cube induced crack length measurement with AFM instrumentation proved to be an easily executable way to estimate the fracture propensities of different films, useful to achieve a fast turnaround time for the BEOL integration effort. Critical film thickness and cohesive energy can be compared for different films with minimal sample preparation. These values show good correlation to cohesive energy and critical film thickness calculated by the standard 4 pt. bend method.

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