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MECHANICAL PROPERTIES, ADHESION, AND FRACTURE TOUGHNESS OF LOW-K DIELECTRIC THIN FILMS FOR MICROELECTRONIC APPLICATIONS

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ABSTRACT

Due to the radical compromise in thermal and/or mechanical properties that the migration from silicon dioxide to novel low-k dielectric films necessarily incurs, the IC industry is motivated to better understand the failure modes of low-k dielectric films. These failure modes include thermal instability, poor mechanical strength, and chemical-mechanical polishing (CMP) failure due to low cohesive and adhesive fracture toughness. By developing a methodology to predict failure modes, we are able to screen multiple candidate low-K materials. The following study is a discussion of some of the experimental approaches that Motorola has taken to understand and mitigate some of these failure mechanisms.

KEYWORDS

Low-K dielectrics, thin films, adhesion, fracture toughness, mechanical properties

INTRODUCTION

The semiconductor industry is gradually moving from well-established SiO_2/Al interconnects to Cu/low-K to meet the next generation device requirements. Increased device speed, reduced power and heat dissipation requirements and reduced interline cross talk are many benefits driving device manufacturers to move to low-K materials. However the transition is challenging since these new material systems will introduce a number of integration and reliability issues. During the last few years industry has been looking into two major categories of low-K materials namely organic spin-on dielectric (SOD) film and hybrid organic/inorganic silicate films made by a CVD or spin-on process. Each has their advantages and disadvantages. Silicate films are extremely

thermally stable and reasonably hard, but they are fragile. On the other hand organic materials have reasonable thermal stability and are tough, but are soft.

A viable low-K material must be compatible with dual-damascene lithography, etching, stripping and cleaning processes, and especially CMP and device packaging methods. The reliability of devices containing these multi-layer dual-damascene stacks depends on several factors including device mechanical stability, adhesion of the barrier metal to the low-K film, adhesion of etch stop, hard mask and capping layers to the low-K film and barriers and the ability to polish the Cu and package device without failure. Since the dual-damascene stack is exposed to high shear stresses during the CMP process, any flaws at the interfaces or in the low-K film itself can lead to long term reliability problems. Thus detailed reliability and compatibility tests are required to integrate new low-K dielectric materials and Cu interconnects.

For the above-mentioned mechanical device stability four material properties of the device components are important: elastic modulus, hardness, interfacial adhesion, and film fracture toughness.

Mechanical properties of thin films often differ from those of the bulk materials. This can be partially explained by the nanostructure of thin films and the fact that these films are attached to a substrate. Due to typically high yield strengths, thin films can support very high residual stresses. This residual stress can be relieved later during processing or in the actual device operation through plastic deformation, thin film fracture, or interfacial delamination.

Both elastic and plastic properties are important for thin film characterization. Thin film mechanical properties can be measured by tensile testing of freestanding films [1] and by the microbeam cantilever deflection technique [2-4], but the easiest way is by means of nanoindentation, since no special sample preparation is required and tests can be performed quickly and inexpensively.

Nanoindentation is a versatile technique for measuring films mechanical properties (elastic modulus, hardness, interfacial adhesion, and film fracture toughness). It works using the same principals as conventional hardness tests, but is performed on a smaller scale using sensitive load and displacement sensing equipment. During the measurement a sharp diamond indenter is forced into the tested material while continuously recording both the force and the indentation depth. Mechanical properties are measured by either analyzing the continuous load-displacement profile or by measuring the material response to a frequency modulated force oscillation that is imposed on the indentation tip during indentation. Both elastic modulus and hardness can be readily extracted directly from the nanoindentation curve [4-7]. Since the depth resolution is on the order of nanometers, it is possible to indent even very thin (100 nm) films. Indentation has been also used to measure thin film adhesion [8-13], where the mechanical energy release rate, or practical work of adhesion is calculated based on the delamination size. Similar fracture properties such as fracture toughness or adhesion strength are derived from the continuous load-displacement profile and an independently measured geometrical scale parameter, which results from indentation, such as crack length or delamination radius.

Indentation techniques could also be used for measuring fracture toughness. When a sharp tip such as Vickers, Berkovich or a cube corner diamond is indented into bulk brittle materials, radial cracking usually occurs after a critical load has been reached,

which allows ones to calculate fracture toughness based on the maximum indentation load and the crack length [14-16]. This method of analysis in complicated in the case of thin film radial fracture because of the half penny crack shape perturbation by the substrate, film densification, and residual stresses in the film. Current studies have yielded promising developments in this area however.

The objective of this study is to identify proper techniques to characterize low-K materials, and, if feasible, to propose a correlation between the mechanical properties of low-k dielectric thin films and their reliability.

RESULTS AND DISCUSSION

One challenge facing the IC industry lies in generating a low-k film that can withstand chemical mechanical polishing without fracturing or delaminating. Researchers have been putting considerable emphasis on determining hardness or Young's modulus threshold that corresponds with a materials ability to endure CMP, wire bonding or bump processes. According to recent studies at Sematech there appeared to be a correlation between elastic modulus and the films ability to withstand chemical-mechanical polishing. However because these films' mechanical properties are intimately linked with their porosity, trends in hardness often correspond to trends in modulus. Correlation with CMP failure can be just as easily made with the film hardness. Figure 1 shows the relationship of hardness and modulus that stems from the variation in porosity of a group of silicate films. Furthermore, one must be careful not to make the conceptual leap from correlation to causation. Studies at Motorola have shown that it is not just modulus, hardness, adhesion or toughness, but more likely a combination of all of these mechanical properties that cause CMP related failure.

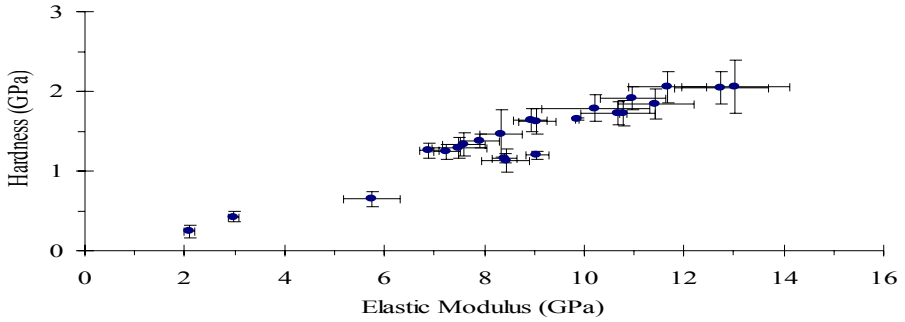

Figure 1. Linear plot of representative silicate low-k dielectric film hardness versus modulus, demonstrating the interrelated mechanical properties of these films that stem largely from their porosity.

Elastic modulus and hardness of different low-K materials from different vendors were measured using nanoindentation. These studies were carried out using a NanoIndenter XP dynamic contact module. Frequency and tip displacement modulated continuous stiffness measurements were made at a frequency of 75 Hz and an oscillation amplitude of 1 nm. Mechanical properties were extracted from hardness and modulus versus displacement profile minima or approximately at a depth of 10% of the film thickness. All films were deposited on thin metallic adhesion/barrier layer to a thickness

of approximately 1 μm. Schematic of a typical low-K test structure is shown in Figure 2. Typical hardness and modulus profiles as a function of indentation depth are shown in Figure 3. Presently it is not well understood whether the increase in modulus and hardness at low depth is an effect of adhesion or an intrinsic indentation size effect [17]. It is important to note that many low-k films exhibit viscoelastic and viscoplastic (creep) behavior, which significantly complicates the measurement of their mechanical properties. These properties from indentation measurements are strain rate and tip oscillation frequency dependent. Progress has been made in techniques utilizing spherical indentation to measure these time dependent properties. However the use of blunt indentation tips often precludes the use of very thin films because of the inability to localize the plastic zone beneath the tip. Furthermore techniques of tip frequency sweep to determine storage and loss modulus in thin films requires that these films adhere to linear viscoelastic theory.

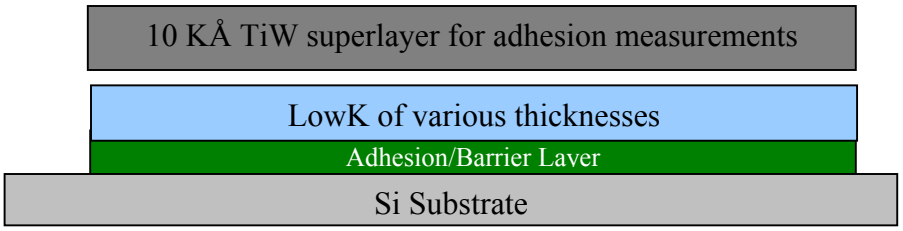

Figure 2. Schematic of a typical Low-K test structure.

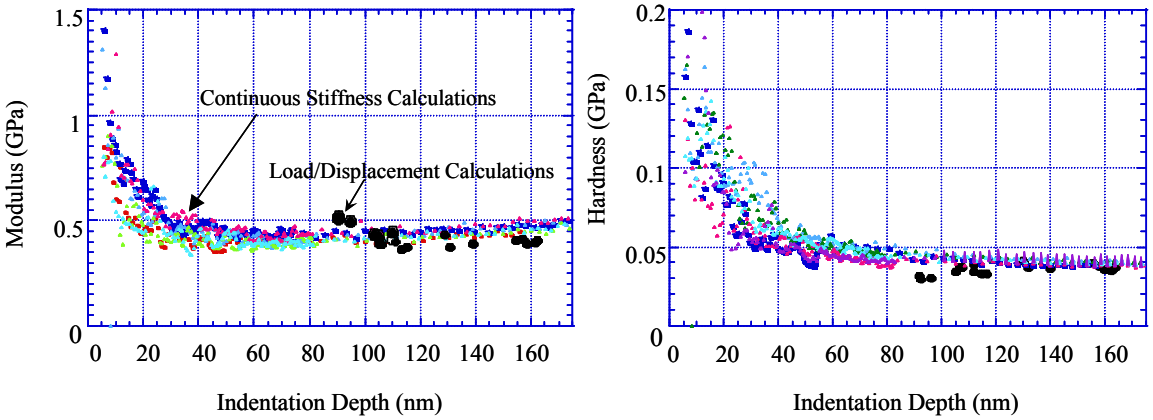

Figure 3. Elastic modulus and hardness profiles as a function of indentation depth.

Beyond measuring the mechanical properties significant advances have been made recently in measuring the adhesion strength of thin films using nanoindentaiton. With the high hardness to modulus ratios one may expect low fracture toughness and adhesion of these materials. Adhesion of low-K dielectric films was measured by means of a superlayer indentation technique [10-13]. Most well-adhered or low modulus thin films can not be delaminated by means of regular indentation and would rather deform plastically around the indenter by forming pileup, or they would not be able to carry the indentation stress to the crack tip. To prevent these problems a high modulus hard superlayer, capable of supporting and storing large amounts of elastic energy is deposited

on top of the film of interest. Upon indentation a delamination blister forms around the indent, and its area is used to calculate the strain energy release rate (practical work of adhesion). Several delamination blisters have been cross-sectioned using Focused Ion Beam (FIB), and it showed that in most cases low-K fracture is cohesive, so what is really measured is the fracture toughness of the low-K film, not the interfacial adhesion.

Knowledge of the elastic modulus and hardness are also required to calculate fracture toughness. Fracture toughness of a bulk brittle material can be calculated based on the maximum indentation depth, P_{max} and the crack length, c [15,16]:

$$K_C = \alpha\left(\frac{E}{H}\right)^{1/2}\left(\frac{P_{\max}}{c^{3/2}}\right) \tag{1}$$

where α is an empirical constant which depends on the geometry of the indenter, E is the elastic modulus, and H is the mean hardness. This expression can not be directly applied in the case of a thin film, since typically the crack shape is no longer halfpenny shape anymore. It was also noted that the maximum indentation load scales linearly with the crack length to the 3/2 power, so as a first order approximation equation 1 can be used to estimate low-K films fracture toughness.

An additional method utilized for measuring toughness involves a lateral scratch, which causes a tangential stress at the trailing edge of the scribe. This has been utilized by both Ostartage, et al [18] and Hoehn, et al [19], noting that

$$K_C = 2\sigma_{\theta\theta}\sqrt{\frac{c}{\pi}\sin^{-1}\left(\frac{a}{c}\right)} \tag{2},$$

where a is the contact radius and c is the half crack. Since a/c is almost always less than ½, $\sin^{-1}(x) \sim x$ and with $\sigma_{\theta\theta} = P_{\max}/\pi a$, one finds that equation (2) reduces to:

$$K_c \approx \frac{2P_{\max}}{\pi^{3/2}}\cdot\frac{1}{ac^{1/2}} \approx const\cdot\left(\frac{P_{\max}}{c^{3/2}}\right) \tag{3}$$

with the latter approximation coming if $c/a \sim$ constant. This then is the same as equation (1), since $(E/H)^{1/2}$ is nearly constant in Figure 1. However, both equation (1) and (3) have inherent composite yield strength, modulus and strain energy release rate built into a laminate system needing detailed analysis.

It is important to understand how low-K films fracture toughness changes with the film thickness. For several low-K materials there a is a critical thickness of approximately 3 μm at which the film fractures due to the residual stress relief. For the low-K brittle films fracture toughness is most likely to decrease with the film thickness due to the higher residual stress and larger flaw size/porosity. Similar or opposite trends have been described in other thin film/substrate systems. For example, thicker W films on steel are tougher, but W(C) film toughness decreases with the film thickness due to the limited crack tip plasticity [20]. We have made initial attempts to employ ellipsometry to measure low-K film porosity [21], but at this point more thorough theoretical analysis is

required to accurately assess thin film fracture toughness using the nanoindentation technique.

CONCLUSIONS

This report describes a methodology to use nanoindentation to measure modulus, hardness, and adhesion and fracture toughness of various classes of low-K materials. These properties are used to predict mechanical reliability of advanced IC devices. By correlating mechanical properties to mechanical reliability we have an effective approach to screening new dielectric materials.

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