AIP Conference Proceedings

Mechanical characterization of low-K dielectric materials

Cite as: AIP Conference Proceedings **550**, 431 (2001); https://doi.org/10.1063/1.1354437
Published Online: 28 March 2001

Thomas M. Moore, Cheryl D. Hartfield, J. Mark Anthony, et al.

View Online

Export Citation

ARTICLES YOU MAY BE INTERESTED IN

Progress in the development and understanding of advanced low k and ultralow k dielectrics for very large-scale integrated interconnects—State of the art
Applied Physics Reviews **1**, 011306 (2014); https://doi.org/10.1063/1.4861876

Review Article: Stress in thin films and coatings: Current status, challenges, and prospects
Journal of Vacuum Science & Technology A **36**, 020801 (2018); https://doi.org/10.1116/1.5011790

Plasma processing of low-k dielectrics
Journal of Applied Physics **113**, 041101 (2013); https://doi.org/10.1063/1.4765297

Lock-in Amplifiers up to 600 MHz

Zurich Instruments

Watch

© 2001 American Institute of Physics.

HTML ABSTRACT

Mechanical Characterization of Low-K Dielectric Materials

Thomas M. Moore, Cheryl D. Hartfield, J. Mark Anthony#, Byron T. Ahlburn, Paul S. Ho*, and Mikel R. Miller*

Texas Instruments, Silicon Technology Development, Dallas, TX
#University of South Florida, Center for Microelectronics Research, Tampa, FL
** University of Texas at Austin, Laboratory for Interconnect and Packaging, Austin, TX*

Abstract. The implementation of materials in device interconnect structure is being driven by shrinking device geometries. In order to meet customer demands for increasing electrical performance, the industry is adopting a solution that provides both lower resistance and lower capacitance. Lower resistance is accomplished by switching from Al(Cu) to Cu interconnect and the capacitance is reduced by replacing SiO_2 in the inter-level and inter-metal dielectric layers with lower dielectric constant materials (low-K materials)[1,2]. A change in materials in a process as complex as IC manufacturing is inherently accompanied by an increase in reliability risk. A thorough understanding of the low-K dielectric candidates is necessary for selection of the best candidate that has sufficient mechanical integrity to survive thermal stresses, CMP, packaging, and test, as well as allows for maximum extendibility to next generation devices. Towards this end, the industry has adopted methods and tools to measure mechanical properties and adhesion energies associated with low-K films. It is expected that porosity will significantly deteriorate the mechanical strength of ILD films compared to non-porous films and the effect on mechanical strength may be markedly different if the pores percolate together to form channels rather than remain isolated. Understanding the mechanical properties of these thin films and choice of appropriate mechanical performance metrics is necessary for successful full-scale integration into a reliable packaged product.

CHARACTERISTICS OF LOW-K MATERIALS

The mechanical characterization of low-K material candidates has shown that lower K typically also means lower elastic modulus and hardness. Mechanical reinforcement of the low-K dielectric stack through the use of metal grids has been reported effective in preventing damage during wire bonding [3]. To ensure a low-cost packaging solution for a low-K device, mechanical strength issues must be understood and considered in parallel with electrical performance targets for the device. Issues such as electrical performance, dielectric material selection, packaging reliability, stack design and reinforcement strategies have become shared responsibilities for device designers, process integration experts and packaging experts who work together in cross-functional teams. The implementation of Cu/low-K has led to an unprecedented synergy between Si process development and packaging readiness.

Although dielectric permittivity is often referred to as the dielectric "constant" K, it is not necessarily constant and may show strong variations with frequency, electric field or temperature due to a variety of possible dielectric mechanisms. Many of these mechanisms are proportional to the electron density of the material. Electron density of conventional dielectrics such as SiO_2 can be reduced in various ways, including reducing the atomic number of various components (i.e. replacing Si or O in the material with materials such as H, F or C), choosing a structure with lower mass density than SiO_2, or reducing the density of an existing material by adding porosity to the film. All these methods have been investigated and will lead to various classes of low-K films. Unfortunately most methods that reduce electron density also reduce the mechanical strength of the material.

A typical plasma oxide has K~4.1 and will be in compressive stress. Addition of SiF_4 to SiH_4 and N_2O in a standard plasma oxide deposition process results in an oxide-like material with K values down to ~3.6 possible. This fluorosilicate glass (FSG) is mechanically similar to the standard plasma oxide. Most FSG materials absorb moisture readily and release H_2O and fluorine, possibly in the form of HF which can cause poor adhesion or corrosion of metal leads. Spin-on dielectrics based on a silsesquioxane group, to which either a hydrogen atom or a simple organic group such

CP550, *Characterization and Metrology for ULSI Technology: 2000 International Conference,*
edited by D. G. Seiler, A. C. Diebold, T. J. Shaffner, R. McDonald, W. M. Bullis, P. J. Smith, and E. M. Secula
© 2001 American Institute of Physics 1-56396-967-X/01/$18.00

as a methyl or phenyl has been attached, have been used in production for several years. Hydrogen silsesquioxane, or HSQ, has a dielectric constant as low as 2.8 when cured without oxidizing any of the Si-H bonds. The early methylsilsesquioxane materials had K values in the same range, while more recent versions have K values as low as 1.8. The silsesquioxane-based films are applied by spin coating, baking the solvents out of the films on a series of hotplates, and finally curing in N_2 at ~400°C. Film stress is usually ~100GPa tensile. These materials have lower K mainly because they have lower density than the standard plasma oxides and FSG. Films similar to the methylsilsesquioxane based films can also be deposited by plasma processes using precursors such as methyl silane or tetramethyl silane. These generic films are now called organo-silicate glasses (OSG). Purely organic films now available also offer K values as low as 2.5. Both spin-on and CVD techniques are possible, but spin-on materials have been far more widely investigated. Hot plate bakes and furnace anneals, or "cures", are fairly similar to those done for the inorganic or OSG type films, but many of the organic materials require adhesion promoters. Low polarizability is probably more responsible for the low K of these films than is density.

K values less than or equal to 2.0 will be required for devices with quarter micron metal pitch. Films with K this low have been prepared and are highly porous. It is likely that any film with such low K will be porous whether it is inorganic, wholly organic, or a combination. The semiconductor industry is working hard to enhance the strength of highly porous films. Attempts include creating a sturdy long range frame by templating silica growth in sol gel processing, controlling pore formation by limiting pore size and connectivity of the pores, creating interpenetrating media, and developing new materials such as porous silicon oxynitride.

CHARACTERIZATION OF LOW-K MATERIALS

Because low-K dielectric films may have reduced modulus and hardness, increased moisture absorption, and reduced adhesion performance relative to traditional dielectrics, there are many potential challenges associated with integrating and packaging devices having low-K materials. These include the possibility of mechanical damage resulting from probe and assembly, as well as in-package stresses. These challenges apply to both wire bond and flip chip products. A thorough understanding of the mechanical behavior of all the materials in the stack as an integrated structure is required to arrive at solutions that maximize net revenue per wafer.

Understanding the mechanical performance of low-K stacks requires an alliance of mechanical characterization with mechanical modeling. Mechanical modeling is extremely valuable for extrapolating the results from blanket films or simple structures to the anticipated performance of integrated structures. However, accurate models of the performance of integrated structures require a substantial amount of physical characterization of key materials and interfaces. A requirement for typical modeling studies is that perfect adhesion must be assumed at all interfaces.

A variety of techniques is available throughout industry and academia for determining mechanical properties of films [4] including nano-indentation, MEMS techniques [5,6], peel tests and blister tests [7,8], as well as double cantilever beam (DCB) testing, 4-point bend testing, and modified Edge Lift-Off Testing (m-ELT) [9,10,11]. For rapid implementation of new processing technologies, it is essential that the capability exists to quickly and accurately test many different types of samples. Specific methods that readily meet these criteria include nanoindentation for determination of Young's modulus and hardness, and m-ELT for determination of interfacial fracture toughness and adhesion energy. These methods often allow quick discrimination between "good" and "bad" samples or processes. The 4-point bend test is also an excellent method to evaluate strain energy release rates and has also been applied to characterization of low-K films, although the through-put is 50-70% slower than the m-ELT test since it is not possible to test multiple samples at a time on a single instrument. For the quantitative measurements from all these tests to be properly interpreted and applied to process development, a thorough understanding of the many factors that affect the measured value are required, especially in the case of porous films.

In addition to mechanical property concerns of low-K materials are thermal concerns. Reduced thermal conductivity through the dielectric layers may be an issue for high power devices. Any thermal decomposition of low-K materials would only adversely affect the material properties and could also cause outgassing, resulting in delamination and via poisoning. Thermal stability can be improved by adding cross-links and introducing rings so it is harder to break the bonds that will release molecular fragments. This may improve mechanical strength, but

adversely affect the K. Thermal stability can be assessed with isothermal thermogravimetric anaysis (TGA), which measures weight loss as a function of time at constant temperature. Thermal decomposition tends to be less of a problem for inorganic materials than for organics, which typically are not thermally stable at maximum BEOL processing temperatures (400-450C for subtractive Al processes). Cu processing can have less demanding thermal requirements, allowing a greater number of material alternatives.

The coefficient of thermal expansion (CTE) of many polymer low-K films is fairly high in relation to surrounding materials. For example, the CTE of SiO_2 is around 4ppm/°C and Cu is 16.5ppm/°C, while materials such as BCB, SiLK, and AF4 all have a CTE of around 60ppm/°C. This alone might make an inorganic low-K film seem a more attractive choice (CTE of HSQ = 14ppm/°C). However, in general the amount of stress induced in films upon temperature excursions is a function of both CTE and modulus, so higher CTE for lower modulus films may not be a problem. All else being equal, however, it is best to minimize CTE mismatch. Dual substrate bending beam is one method to measure CTE of thin films [25]. However, it may not be possible to apply this technique to very low density films such as porous aerogels. Specular X-ray Reflectivity (SXR) is a very precise and direct measurement of thin film density [19, 22]. It has only recently been applied to porous low-K films and is capable of measuring CTE as well as film thickness and density [23, 24].

This paper is focussed on the mechanical characterization of low-K films by nanoindentation, m-ELT and 4-point bend testing.. Results for a variety of low-K films are presented, along with some challenges associated with applying these techniques to low-K materials.

Nanoindentation Testing

Nanoindentation is frequently used on sub-micron films as a method to measure Young's modulus and hardness. These properties are important for mechanical modeling studies, such as comparing different interconnect structure strategies or predicting failure under an applied load. Nanoindentation measurements are made by applying a constant strain rate to a Berkovitch indenter tip, then unloading the indenter, while monitoring the load vs. indenter displacement. Recent improvements in nanoindentation technology facilitate the measurements of thin films and polymers. Commercially available today are systems that enable true dynamic measurements (referred to as continuous stiffness measurements) throughout the loading cycle by applying a small a/c oscillation to the d/c load. The advantages of this dynamic technique include improved surface detection and quantitative measurements as a function of depth. This allows accurate measurements on very thin films and on materials with depth-dependent variations in elastic properties. Also, the influence of the substrate on the mechanical behavior of thin films can be readily measured [5,12,13].

The elastic modulus is defined as the ratio of stress to strain and describes the elastic response of the sample to an applied load. The measured stiffness from the load vs. displacement data are converted to elastic modulus by the formula

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A}} \quad (1)$$

where E_r is the reduced modulus, S is the measured stiffness, and A is the contact area between the indenter tip and the material. The material parameters required for stress analysis are Young's modulus, Poisson's ratio, and the coefficient of thermal expansion (CTE). For modeling studies of stress analysis from a packaging perspective, isotropic and linear elastic material behavior is typically assumed since for most cases, the elastic analysis is the most conservative analysis. Thus, typically more focus is placed on modulus than hardness.

Hardness is important when assessing the trend to permanent deformation, such as plasticity or wear. The hardness is determined by

$$H = \frac{P_{\max}}{A} \quad (2)$$

where P_{max} is the peak load and A is the projected area of indenter contact at peak load. The smaller the indentation at fixed load, the less the area of contact and the harder the material. Hardness is a function of modulus and strength. In ductile samples, the hardness is inversely related to the volume of plastically deformed material under the indenter, and is therefore a measure of the resistance of the sample to plastic deformation [14]. A challenge in direct comparisons of hardness between different classes of low-K materials is that the classical definition of hardness as it applies to a homogeneous solid may not apply uniformly. For brittle porous materials, the apparent hardness may be influenced by collapse of the porous structure and by whether the pore structure is open or closed cell. Also, traditionally hardness has been a good indication of fracture resistance for ceramics and other hard, brittle

materials. However, it may not be a good indication of fracture resistance for polymer materials which can be tougher than ceramic materials, despite having lower hardness [20]. One should bear in mind that these mechanical tests are based on linear elastic theory. Although all materials tested are assumed to have a linear elastic response, this may not be the case and may be a source of error when comparing measurements between material classes.

Nanoindentation studies were performed on several dielectric materials covering a range of elastic moduli. The samples include traditional dielectrics (dense oxides PE-TEOS, HDP and FSG), as well as low-K materials such as H-doped oxide-like materials (HSQ), organo-silicate glasses (OSG), organic polymers, and porous oxides. For testing, bare dielectric films between 500nm and 1000nm thick were deposited on Si (with an intermediate 100nm PE-TEOS layer to facilitate adhesion), and were measured at a depth of less than 10% of the dielectric film thickness. Figure 1 shows SEM images of indents from representative members of different classes of dielectrics (dense oxide, silsesquioxane, OSG, and organic polymer). It is evident that cracks emanate from the indents for all materials except for the Poly2 sample, which may indicate stronger fracture toughness for this material compared to the non-polymer materials. The elastic moduli of all tested materials span a wide range from 0.58GPa up to 72GPa (Table 1). Not surprisingly, the highest moduli were found in the dense oxide group,

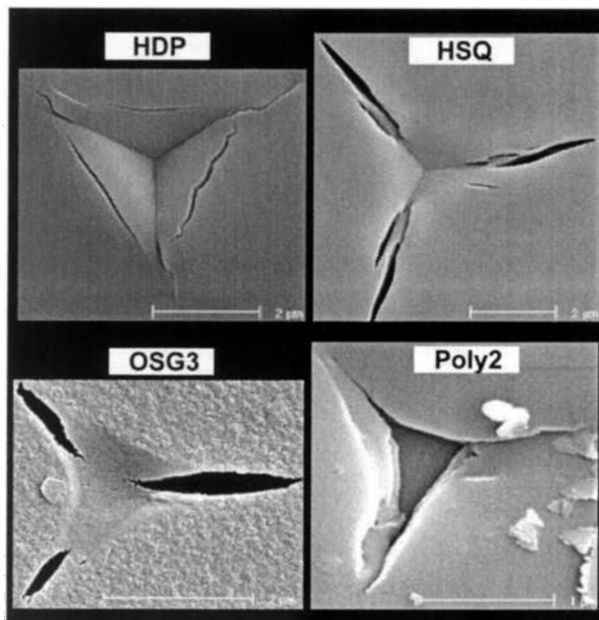

FIGURE 1. SEM images of nanoindentation marks on dielectric group representatives from Table 1. Note: micron bar = 2um on all except Poly2, where the bar = 1um.

followed by the OSG and organic polymer materials.

The porous oxide had the lowest modulus. Lower dielectric constant implies lower elastic modulus, as expected (Figure 2). Other materials that will be in contact with the low-K dielectrics in the stack have much higher moduli (SiN ~140GPa). During probe and wire bonding, a thin, brittle, high modulus layer (such as a nitride or oxide) over a thicker and lower modulus dielectric may be deflected locally until it fractures as the dielectric compresses beneath it. Hard materials may also crack when placed over softer materials that shift during temperature cycling. For example, package shear stresses at die corners can cause Al bond pads and wide metal traces to shift, inducing cracks in the overlaying SiN [21]. Thus it is important to consider modulus in terms of the entire stack structure.

The Modified Edge Lift-Off Test

The modified Edge Lift-Off Test (m-ELT) is a fracture mechanics based approach that measures the applied stress required to induce interfacial or cohesive failure in thin films (typically <1um thick). The test takes advantage of CTE mismatches created during sample cooling from +120°C to –165°C. The presence of an epoxy backing layer with a known stress-temperature profile ensures sufficient stress is present to cause failure and allows measurements of applied fracture toughness values without knowledge of any material properties of the test film(s). Applied fracture toughness values (K_{app}) are calculated using the formula

$$K_{app} = \sigma\sqrt{h/2} \quad (3)$$

where σ is the residual stress and h is the thickness of the epoxy. If the modulus of the test material is known, then adhesion energies can also be measured by this test from the formula

$$G_{app} = [\sigma_0^{\,2} h(1-\nu)]/2E \quad (4)$$

where σ_o is the residual stress, ν is Poisson's ratio, and E is Young's modulus. The accuracy of the technique is generally within 10%. After testing, the failed surfaces can be examined to determine the locus of failure and establish whether the measured values represent cohesive fracture toughness or interfacial (adhesive) fracture toughness [15]. Examination methods include SEM/EDS, XPS, TEM, AFM, SIMS and optical microscopy. The test assumes that the residual stresses in the film are negligible compared to the stresses applied by the thick epoxy backing layer.

TABLE 1. Dielectric Film Nanoindentation Measurements of Young's Modulus and Hardness

	Dense Oxides			HSQ	OSG Materials			Organic Polymers			Porous Oxides
	TEOS	HDP	FSG	HSQ	OSG1	OSG2	OSG3	Poly1	Poly2	Poly3	Nanoglass
K	4.1	4.1	3.3-3.7	3.1	2.6	2.8	2.9-3.0	2.7	2.6	2.5	2.2
CTE (ppm/C)	4	~4	~4	14	17	?	?	62	66	60	~4
Modulus (GPa)	72	72	50	4.9	2.5	3.6	12.2	4.7	4.3	6.5	0.58
Hardness (GPa)	9.5	9.7	6.8	0.85	0.13	0.36	1.7	0.33	0.31	0.41	0.041
Cu modulus and hardness = 137GPa and 0.97GPa, respectively						Al modulus and hardness = ~70GPa and 0.68GPa, respectively					

However, in general it is important to understand the residual stresses in the test films.

The results for m-ELT tests of dielectric candidate materials deposited on PE-TEOS are shown in Table 2. In general, a target m-ELT measurement above 0.3Mpa-$m^{1/2}$ is desired to ensure a process robust enough to survive CMP, wire-bonding, and other assembly processes. Whereas HSQ and OSG3 appeared similar based on nanoindentation results alone, they can now be differentiated based on their adhesion to oxide. As shown in Figure 3, the combination of m-ELT and nanoindentation differentiates candidates to a much greater degree (for this particular interface) than does either test alone. It is clearly evident that based on m-ELT values (dielectric/oxide interface), HSQ can be grouped with the dense oxides, while OSG3 can be grouped with the organic polymers. Since HSQ and OSG3 have similar moduli, the higher fracture toughness of HSQ on oxide suggests that HSQ may be a better choice, based on these tests. However, in order to accurately understand the mechanical performance of the interconnect stack, every critical interface must be measured. Those samples showing reduced interfacial performance have the potential to be improved through process optimization, addition of adhesion promoters, or clean-up steps.

There has been some criticism directed at the use of m-ELT for determination of fracture strength, since the mode-mixity may vary for different samples and the process of generating CTE stresses cannot be de-coupled from temperature-related changes in the layers, which typically will become more brittle with cooling. A 2D FEM plane stress analysis shows that both the mode mixity and the strain energy release rate will change for larger initial crack lengths. This effect disappears when the crack length to backing layer height ratio becomes greater than two, which implies that for the range of epoxy thicknesses generally used, an ideal pre-crack length is around 0.5um. However, typically the length of the initial crack, assumed created

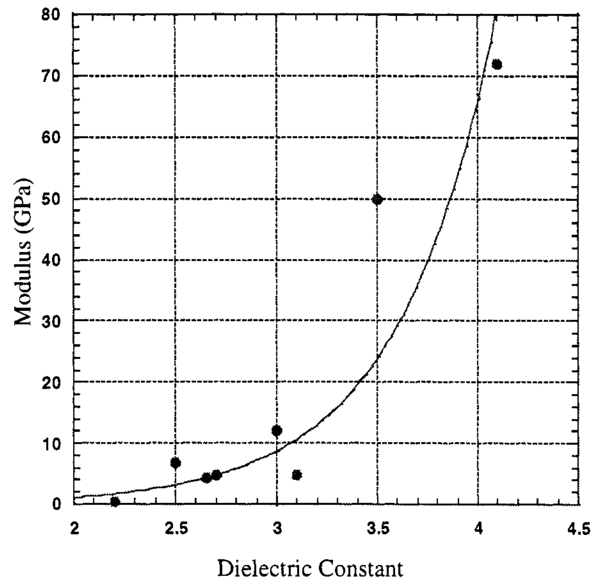

FIGURE 2. Relationship between modulus and dielectric constant (from Table 1).

spontaneously upon sample dicing, is not measured and in many samples, if cracks are present they are too small to be confirmed optically. Thus even with uniform adhesion, a large spread could occur for the temperature at which cracks grow, due to variations in initial delamination length of the m-ELT samples. Therefore one needs take into consideration the initial delamination size. It is believed that calculations of G by 2D plane stress analysis will provide a conservative prediction of delamination growth (lowest values of G). Currently work is ongoing to calculate G from 2D axi-symmetric analysis, extend the effort to 3D geometries, and to validate the modeling predictions using m-ELT data on specimens built with known crack dimensions to ensure correct data reduction methods and develop design guidelines for m-ELT specimen geometries.

Other test factors can also significantly change the measured fracture toughness and must be carefully

TABLE 2. M-ELT Measurements of Applied Fracture Toughness (Dielectric on PE-TEOS)

	Dense Oxides			HSQ	OSG Materials	Organic Polymers		Porous Oxides
	TEOS	HDP	FSG	HSQ	OSG3	Poly2	Poly3	Xerogel
K	4.1	4.1	3.3-3.7	3.1	2.9-3.0	2.6	2.5	2.2
CTE (ppm/C)	4	~4	~4	14	?	66	60	~4
K_{app} (MPa-m$^{1/2}$)	0.46	0.42	0.49	0.44	0.19	0.41	0.13	<0.1
Type of failure	cohesive	cohesive	cohesive	cohesive	adhesive	adhesive	cohesive	cohesive
Location	Si	Si	Si	Si	OSG3-Teos	Poly2-Teos	Poly3	Xerogel

monitored. For example, if the epoxy backing layer is too thick, an artificially low value will result. The epoxy thickness at which this occurs is sample dependent, but typically can be detected by looking for a plateau in a plot of temperature at failure vs. epoxy height. In addition, the method of dicing samples (sawing vs. cleaving) can drastically impact the measured values. Measured fracture toughness of FSG/SiN samples was found to vary as much as 40% depending on the dicing method. This effect has also been noted for polymer materials such as SiLK, where the measured value dropped from 0.4MPa-m$^{1/2}$ to 0.25MPa-m$^{1/2}$ when the sample was sawed rather than cleaved. For this reason cleaving is the recommended dicing method, although samples containing blanket Cu or Al must be sawed due to the increased ductility of these samples after adding the epoxy backing layer, which prevents cleaving.

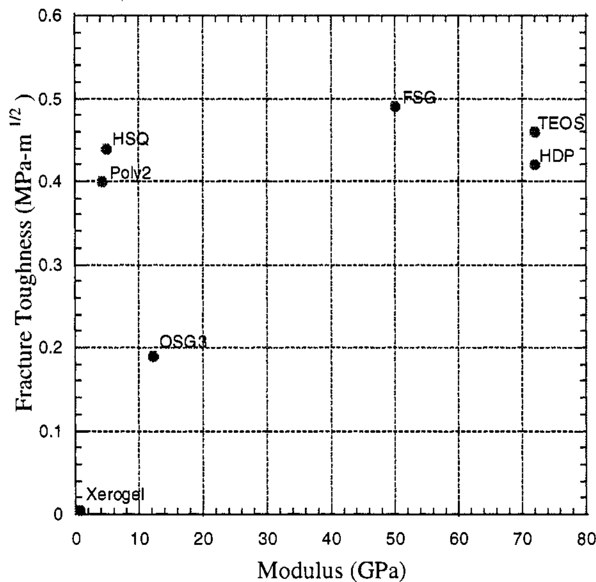

FIGURE 3. Relationship between fracture toughness (on oxide) and elastic modulus.

With proper application of the technique, it has been shown that m-ELT measurements compare well with measurements obtained by the 4-point bend test [10]. Data (not shown) generated from m-ELT tests of BCB on Si wafers correlate well with reported measurements obtained by other techniques [16]. Furthermore, the correlation of low values (~0.25MPa-m$^{1/2}$) for a blistered blanket PE-TEOS/Al metal stack and high values of 0.4MPa-m$^{1/2}$ for an improved non-blistering metal stack validate the m-ELT test for materials other than polymer or low-k samples. In addition, this supports the target 0.3MPa-m$^{1/2}$ value as an indication of whether a sample is strong enough to survive all processing and packaging stresses.

Porous films such as aerogels are challenging to measure using m-ELT. The open pore structure of the films requires that such materials are capped. In addition, the stresses from the epoxy backing layer typically cause the entire sample to delaminate during the epoxy cure, preventing any sort of measurement. For this and other reasons mentioned above, an alternative method to m-ELT is required for measuring fracture energies.

Geometry-Based Fracture Mechanics Tests

Double cantilever beam (DCB) and 4-point bend tests measure the strain energy release rate or adhesion energy G under different stress loadings. DCB measures mode I adhesion energy, while 4-point measures mixed mode adhesion energy with a phase angle typically of 43 degrees in common configurations. Since adhesion varies as a function of mode mixity, typically, measured values from 4-point are higher than those from DCB. Furthermore, the weakest link in a film stack may be different for mode I loading compared to mixed mode loading. An advantage of both tests is the ability to measure sub-critical crack growth as a function of temperature and/or humidity [17, 18].

Typically, for both tests the films of interest are sandwiched between 2 pieces of Si. For DCB, a gap of a predetermined length is built into one end of the sandwich to initiate the crack at a specific interface. This can be challenging to accomplish for interconnect films. In 4-point bend, the upper Si beam is sawed across the width to a depth within 20um or so of the interconnect films. As force is applied during 4-point testing, this will cause propagation of a crack through the Si and then laterally into the thin films at the weakest interface. The load and displacement are recorded during both 4-point and DCB tests, although for DCB the crack length must also be measured throughout the test. For 4-point bend, the adhesion energy G is independent of the debond length when the debond is more than 2x the total beam thickness. Using beam theory, G can then be calculated solely on geometrical parameters by the formula

$$G = \frac{21(1-\nu)M^2}{4Eb^2h^3} \tag{5}$$

where b is the beam width, h is the half thickness, E and ν are Young's modulus and Poisson's ratio of the bulk substrate, and M is the bending moment (defined as M = PL/2, with P being the load and L the spacing between the inner and outer loading lines). Typical samples are sawed into 3mm x 30mm strips, although it is also possible to cleave samples with a width anywhere between 5mm and 10mm. Since the diced edges are perpendicular to the crack front, and the sample width is large compared to the damage zone size along the diced edge, it is expected that the dicing method sensitivity demonstrated in m-ELT is minimized for 4-point testing. Still, polishing of sawed edges helps prevent Si fractures during testing.

An advantage of 4-point bend testing over other techniques includes retention of residual stresses in the film during testing, since stress relaxation of the film is constrained between two large elastic substrates. The test is capable of measuring stronger as well as weaker samples and the mode mixity is better defined compared to the m-ELT test. It is easier to perform on thin films than double cantilever beam testing (DCB), since it is not necessary to fabricate a pre-crack into the test film, nor to measure the crack size throughout the test. The 4-point bend test, with its well-defined mode mixity and environmental flexibility, and m-ELT with its more limited sample applicability but faster sample throughput, represent a complementary approach to characterizing interfacial adhesion.

Representative critical adhesion data for DCB and 4-point bend samples are shown for BCB to common Cu liners in Figure 4. Error bars are shown as 1 standard deviation and are generally less than 10% of the mean. As expected, G values obtained under mode I loading are low compared to mixed mode loading test values. Subcritical crack growth data for the same interfaces under 4-point bend loading are shown in figure 5. This type of data provides insight into the effects of temperature and humidity on interfacial integrity. Since the geometry is accurately known and the low-K layer is extremely thin, the interfacial crack velocity can be calculated from the linear elastic relationship between load and compliance. This allows for a relatively straightforward analysis of this failure mode.

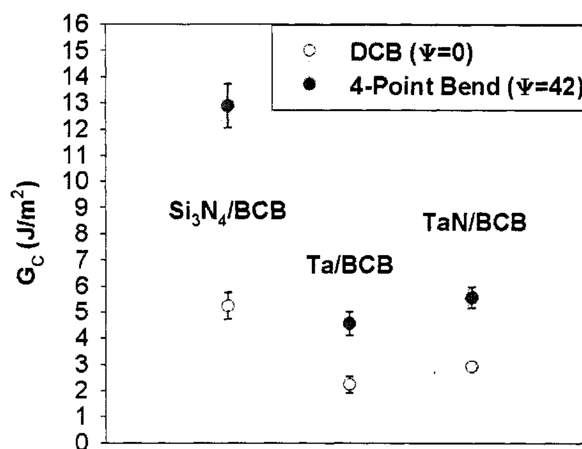

FIGURE 4. Critical energy release rates for BCB interfaces without adhesion promoter.

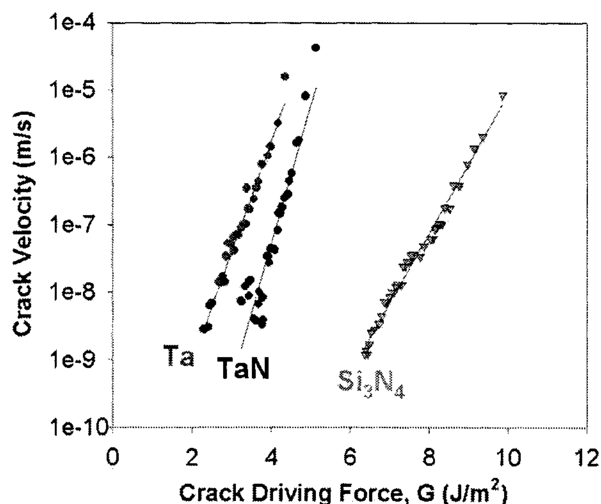

FIGURE 5. Mixed mode subcritical crack growth along BCB interfaces without adhesion promoter.

SUMMARY

The selection of an advanced dielectric material with low dielectric constant, good mechanical properties and adhesion performance, and good thermal conductivity is a requirement for low-K integration. The strong chemical bonds that improve mechanical strength typically are polar so they cannot be added without increasing the dielectric constant. Porous materials with K<2.5 will have degraded thermal and mechanical properties compared to the non-porous parent material. Thus, starting materials that begin with a low K should be chosen for incorporation of porosity to meet the lower target K. Many techniques exist for measuring the material properties of low-K dielectric materials to help answer questions about the impact of reduced dielectric modulus on product reliability, the relative performance of homogeneous and embedded dielectric strategies, the benefit of reinforcement, and time-dependent changes in the dielectric layers. Although figure of merit goals for mechanical properties, thermal behavior, and adhesion strengths can be identified, a mechanical performance metric for the integrated dielectric material ultimately must be based on the reliability performance of the assembled device. Integration of a soft dielectric which deforms rather than cracks (such as a polymer) may seem a good choice for the low-K stack, but it may increase the risk of cracking for brittle layers in the structure (barriers, etch stops, hard masks, etc.) used in the Cu dual damascene process.

Accurate measurements and the choice of an appropriate mechanical metric are necessary for predictive modeling and the selection of a material with scalability to finer geometries. The wish list of attributes for the ideal mechanical test for low-K materials includes: mechanical, adhesion and thermal characterization of patterned sub-micron structures in varied environments; low cost measurement (tool + samples + time); minimal sample preparation; detection of sub-critical crack growth (moisture, cyclic loading); and applicability to porous films. It will most likely require several techniques to satisfy all these requirements.

In parallel with mechanical characterization and modeling, innovative strategies for structural reinforcement are being developed and tested. Successful implementation of a new low-K material requires robustness of the entire process, from bare film deposition through low-cost packaging and reliability tests. The complexity of the technical challenge, the short time lines, and the expense of development combine to require real time cooperation between design, fab processing, package processing, and reliability organizations in order to minimize risk.

ACKNOWLEDGEMENTS

The authors wish to thank Ed Shaffer of Dow Chemical, as well as Barry Lucas and Angela Gizelar of Nano Instruments, for expert consultation on m-ELT and nanoindentation. We thank Vish Sundararaman for his modeling inputs and we wish to thank the many members of the TI low-K team that have assisted us with obtaining samples for characterization including Andrew McKerrow, Andy Ralston and Tae Kim. We especially thank Brian McAdams, our coop student from Lehigh University, who spent countless hours testing m-ELT samples and perfecting the technique.

REFERENCES

1. Lee, W.W. and Ho, P.S., *MRS Bulletin*, **22** (10), 19-23 (1997).
2. Havemann, R.H., Nagaraj, NS, and Steiss, D.E. *TI Technical Journal*, **16** (1), 1-7 (1999).
3. Saran, M. et al., *Proceedings IRPS*,, 225-231 (1998).
4. Mittal, K.L., *J. Adh. Sci. and Tech.*, **1** (3), 247-259 (1987).
5. Lucas, B.N., Rosenmayer, C.T., Oliver, W.C.,"Mechanical Characterization of Sub-Micron Polytetrafluoroethylene (PTFE) Thin Films", in *Thin Films: Stresses and Mechanical Properties VII*, '97 Fall MRS.
6. Saif, M.T.A. and MacDonald, N.C, *Journal of Materials Research*, **13** (12), 3353-3356 (1998).
7. Goldber, H.D., Cha, G.S., and Brown, R.B., *J. App. Poly Sci*, **42**, 1287-1292 (1991).
8. Allen, M.G. and Senturia, S.D., *J. Adhesion*, **29**, 219-231 (1989).
9. K.T. O'-Brien, "Interlaminar Fracture Toughness: The Long and Winding Road to Standardization", in *Composites-Part-B:Engineering*. **29** (1), 57-62 (1998).
10. Dauskardt, R. H., Lane, M., Ma, Q., and Krishna, N., *Engineering Fracture Mechanics*, **61**, 141-162 (1998).
11. Shaffer II, E.O., Townsend, P.H., and Im, J.-H. "A Method for Assessing the Mechanical Reliability of Low-K Polymeric Dielectric Materials", *Conference Proceedings ULSI XII*, 1997, pp. 429-435.
12. Oliver, W.C. and Pharr, G.M., *Journal of Materials Research*, **7** (1992).
13. Lucas, B.N., Oliver, W.C., Pharr, G.M., Loubet, J.-L. "Time Dependent Deformation During Indentation Testing", in *MRS Symposium Proceedings* 436 edited by W.W. Gerberich, H. Gao, J.-E. Sundgren, and S.P. Baker, 1997, pp. 233-238.

14. Mencik, J. et al., *J. Mater. Res.* **12** (9), 2475-2484 (1997).
15. Shaffer II, E.O., Meyers, G.F., Potter, G., and Hayes, F. "Interface Studies of Cyclotene 5021 and Copper", in *Conference Proceedings ULSI XIII*, 1998, pp. 337-341.
16. Shaffer II, E.O., Townsend, P.H., and Im, J.-H. "A Method for Assessing the Mechanical Reliability of Low-K Polymeric Dielectric Materials", in *Conference Proceedings ULSI XII*, 1997, p. 429-435.
17. Snodgrass, J.M., Hotchkiss, G., and Dauskardt, R.H., "Adhesion and Subcritical Debonding of Polymer Interfaces for Microelectronic Packaging", *in Electronic Packaging Materials Science X*, MRS Symposium Proceedings 515, Warrendale, PA, USA, 1998, pp 37-43.
18. Wylde, J.W. and Spelt, J.K., *International Journal of Adhesion and Adhesives*, **18** (4), 237-246 (1998).
19. Chason, E. and Mayer, T. M., *Crit. Rev. Solid State Mat. Sci.* **22** (1) 1997.
20. Shaffer, E. O., Howard, K.E., Mills, M.E., and Townsend, P.H., "Mechanical Integrity of Ultra-low k Dielectric Constant Materials in ULSI BEOL Structures", *MRS Symposium*, San Francisco, CA April 2000.
21. Suo, Z. et al., "Passivation Film Cracking in the Interconnect Structures Caused by Temperature Cycling: A New Mechanism", *MRS Symposium*, San Francisco, CA April 2000.
22. Dietrich, S .and Haase, A. *Phys. Rep.* **260** (1), 1995.
23. Wallace, W. E. and Wu, W. L.. *Appl. Phys. Lett.* **67** 1203 (1995).
24. Wu W.-L., Wallace W. E., Lin E. K., Lynn G. W.,Glinka C. J., Ryan E.T., and Ho H.-M., *J. Appl. Phys.*, submitted.
25. Zhao, J. H., Malik, I., Ryan, T., Ogawa, E.T. and Ho, P.S., *Appl. Phys. Lett.* **74** 944 (1999).