Advanced Metrology for Rapid characterization of the thermal mechanical properties of low k dielectric and copper thin films

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Abstract

With the proliferation of several types and classes of low k materials, the screening, evaluation and integration of these new materials into copper damascence structures require a new and more effective approach. Evaluation of the thermal mechanical properties of new materials require multiple complementary metrology toolsets. Correlating results from these analysis can be very time consuming and conclusions ambiguous. To overcome these limitations, we report two advanced metrology tools that have a potential to rapidly screen and evaluate thermal mechanical properties of new materials through the integration process. The first is an in situ integrated metrology chamber that simultaneously measures several physical, optical, chemical and electrical changes during a heat cycle, including Stress hysteresis, Outgassing, In situ film shrinkage, in situ reflectivity and sheet resistivity changes . Complementing this tool, we also describe a new image analysis based Quantitative Adhesion Tester based on the MELT (modified edge lift off test) principle, for fast screening and ranking of adhesive or cohesive qualities of new materials through their integration process. The end result is shortening of material screening and characterization time.

Introduction

To overcome the RC time delay with the progressive decrease in device feature size, the industry is currently targeting the incorporation of low dielectric constant (k) materials (k value between 2.2 to 3.7) into higher conductive metals (like copper). Evaluating the thermal-mechanical properties of these new thin film materials, requires the use of several complementary metrology tool sets. For example, initial screening, information on adhesion, stress hysteresis, thermal stability, thermal desorption spectroscopy (TDS), film shrinkage, thermal expansion coefficient, and other material and electrical property changes, after heat cycling, all need to be determined. Following curing, cleaning, etching, stripping and CMP steps, you must remeasure adhesion, outgassing, and electrical properties. [1].

With the increase in several types and classes of low materials in recent months, the screening, evaluation and integration of these new materials into the next generation devices is challenging, and time consuming. According to the International Technology Roadmap for Semiconductors [2], the continued scaling of devices would require evaluation and integration of progressively lower k materials or extra low k materials (XLK) , k < 2.2 to 1.5. In order to shorten design cycles, process integration involving new materials requires more efficient methodology and toolsets than are currently available.

In view of these goals, we describe two newly developed tools that can potentially speed up the characterization of thermal mechanical properties of new thin film materials. The first tool, designed to evaluate the Thermal properties or stability of films is the FSM 900TC-vac, an integrated metrology-annealing chamber. The system simultaneously measures several physical, optical, chemical and electrical property changes in the film during a heat cycle.

Complementing this tool is the FSM Laminar Adhesion Tester, which quantitatively measures the adhesive or cohesive strength of the weakest film in a multilayer stack.

Characterizing Thermal Properties : Integrated Metrology Chamber

This tool comprises a sealed annealing chamber based on a rapid thermal processing (RTP) type chamber design. The system operates either under high vacuum (up to $5x10^{-6}$ Torr range) or in a controlled inert gas environment to closely simulate processing. Multiple metrology probes are placed either inside the chamber or through optical windows. Information such as film stress hysteresis, thermal stability, TDS, film shrinkage, reflectivity, resistivity changes may be simultaneously monitored in situ.

Low k and copper thin films pose a

characterization problem for most metrology tools because these oxidation sensitive materials must be processed or annealed in an atmosphere with very low partial pressure oxygen, in many cases, <10 ppm. This low oxygen criterion is impossible to achieve with traditional high temperature stress tools because of their unsealed heat chuck design. In contrast, the FSM900TC-vac chamber is designed to operate in a high vacuum environment or in a controlled inert gas environment where <10 ppm oxygen can be easily realized for simulating actual thermal processes.

Most outgassing studies on the new low k materials are currently done on small broken pieces of wafers, on a tool like the TDS. The results may raise correlation issues, as they may not reflect whole wafer desorption under actual thermal processes. The new integrated tool works with whole wafers. With a highly reflective, cold walled chamber, radiant heat from the lamps is focused directly and predominantly on the sample. With this design, background desorption from the chamber wall is minimized during outgassing measurements. Full wafers, up to 300 mm, can be characterized.

Film shrinkage and reflectivity changes are customarily measured using a spectrometer or ellipsometer. Measurements are made at room temperature, before and after a thermal process. While this approach is adequate for evaluating total change, it cannot determine the rate of shrinkage at the various temperatures, nor provide information about abrupt changes in thickness or reflectivity at certain temperatures. By incorporating a spectrometer that monitors film thickness and reflectivity changes in situ, for example, through an optical window on top of the chamber, useful information can be extracted. By correlating data obtained from stress hysteresis and TDS, fast verification of distinct thermal load limitations on the materials under study can be made.

Characterizing Mechanical Properties : Quantitative Adhesion Measurement (MELT method)

Unlike conventional dielectric materials like SiO2 and SiNx, which in general, have very good adhesion to most interfaces used in microelectronics, low k dielectrics as a whole, have poorer adhesive properties. Adhesion becomes one of the critical parameters that must be tested during material screening and integration. The principles relating to interface fracture mechanics, debond energy and fracture toughness for multilayer thin films and interfaces have been extensively reported [3] [4] . Several techniques have been developed to measure adhesion in thin films, including the Peel or Stud pull methods [5], the indentation methods [6], the Scratch test, and the blister test. However, these techniques typically lead to qualitative results. Because of large errors [7] caused by the following: i. the plastic bending of thin film during debonding, which is difficult to distinguish from the measured adhesion energy, and ii. the relaxation of residual stresses in the thin film during debonding which contributes to the debond driving forces.

One promising quantitative test for multilayer interface on a rigid substrate that is fairly rapid, is the Modified Edge Lift Off Test (MELT) [8], the principle of which the FSM Laminar is based upon. The MELT method consist of applying a thick backing material (like epoxy) onto a thin test layer (the multilayer film stack) in a rigid substrate, such as a Si wafer. Because the backing layer is much thicker than the film stack, the applied debond energy is approximately equal to the energy stored in the backing layer. The wafer is then diced into square coupons (approximately 1 cm^2) so that 90^0 edges to the substrate are formed . The sample is cooled in a cryostat chamber until debonding occurs . The temperature and initial location of debonding in the test coupons are observed, and recorded using image capture and image analysis via a CCD camera. Up to 30 test coupons can be tested per cycle, lasting 2 to 3 hours. If 5 coupons are sampled from each wafer, the system can test up to 6 wafers with film stacks processed under different conditions, per cycle. If we know the stress–temperature behavior of the backing layer, the applied fracture (stress) intensity , $K_{C,}$, is given by σ, the Stress of the backing layer, times the square root of half the backing layer thickness, h . That is :

$$K_{C,} = \sigma \sqrt{h/2}$$

Once K_C is calculated, it can be converted to G_C , the debond energy of the interface, by ,

$$G_C = \frac{K^2_C}{E'}$$, where E' is the biaxial modulus of the failing layer

After testing, the sample interface is examined to determine the locus of failure.

Results and Discussion

Integrated Metrology Chamber

Some results were already reported on work done on CVD low k and copper samples using the Integrated Metrology tool [9]. For this paper, results on how the FSM 900TC-vac can be used to rapidly evaluate the suitability of materials during the integration process will be shown.

Two 200mm wafer samples were evaluated for thermal stability and outgassing profile. Sample 1 was a SiC CVD low k deposited at 360 degrees C, while Sample 2 was a spin on low k sample. Thermal stability evaluation was based on the Stress Hysteresis and outgassing profiles using the FSM 900TC-vac. Base operating vacuum was 1 x 10 –5 to 5 x 10 –6 Torr. Temperature ramp rate was 10 degrees C/min for both samples.

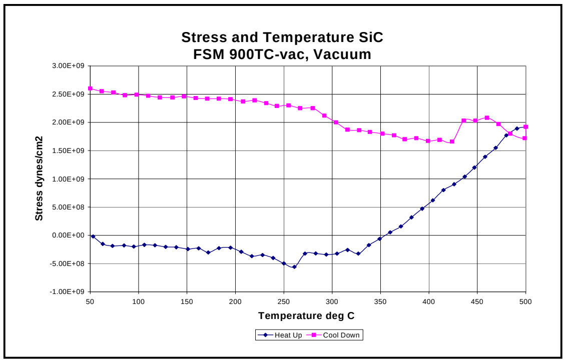


Figure 1 : Stress Temp. of SiC sample

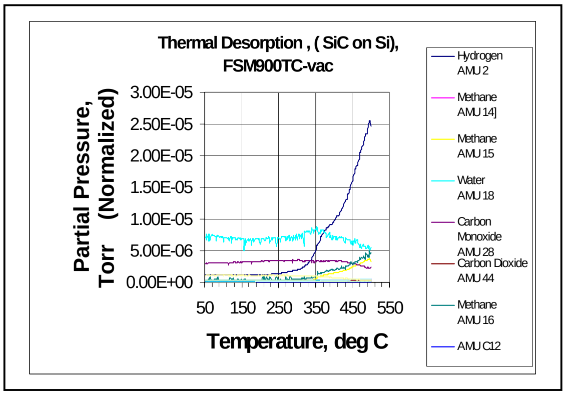


Figure 2 : Thermal desorption of SiC sample

Figure 1 shows the Stress Hysteresis for the SiC sample as elastic to 260 degrees C, thereafter the stress change become very tensile. The stress values reached 200 Mpa, upon heating to 500 degrees C. The sample did not recover its original stress value upon cooling. This information indicates that the material may have changed properties or materials are lost after the heat cycle.

Simultaneous Thermal Desorption Spectroscopy (figure 2) confirmed increased outgassing trends for all carbon and hydrogen based species upon an increase in temperature. Hydrogen (AMU#2) outgassing was significant above 270 degrees C.

This observation correlated to the abrupt change in stress hysteresis profile from compressive to tensile for the SiC wafer from 260 to 500 degrees C.

Figures 3 and 4 showed the Stress Hysteresis and TDS data of sample 2, a spin on low k sample.

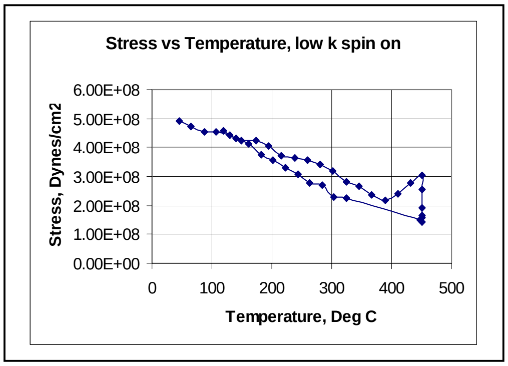


Figure 3 : Stress Temp. of spin on low k sample
Sample annealed at 450 deg C for 30 minutes

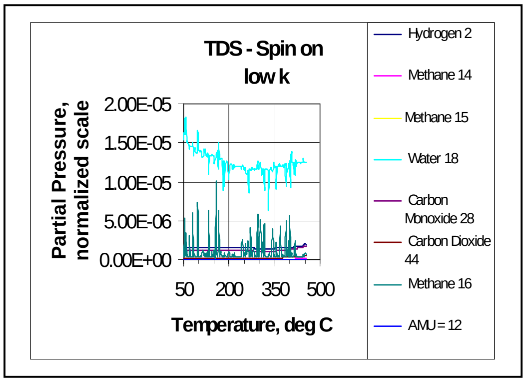


Figure 4 : Thermal desorption of spin on low k

Stress hysteresis was linear up to 450 degrees C for sample 2 (figure 3). Upon annealing and cooling the stress returned close to its starting stress. Outgassing data showed little desorption of materials for the temperature range.

By comparing such results, we can quickly screen, evaluate and rank performances of different materials without the corresponding sample to sample and tool to tool correlation issues typical in new material characterization.

Quantitative Adhesion Tester

Diagrams for the sample preparation, Imaging and Analysis using the FSM Laminar Adhesion Tester are shown in Figures 5 to 8. The system automatically tracks the delamination temperature and the location of debonding of the various test coupons. A report is generated ranking the adhesion strengths of the coupons according to their fracture toughness, K_c.

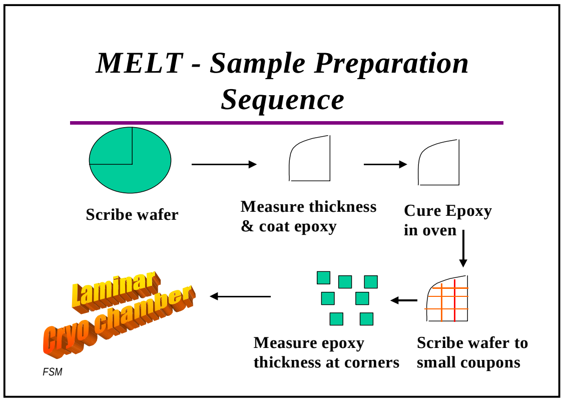


Figure 5 : Diagram of sample preparation

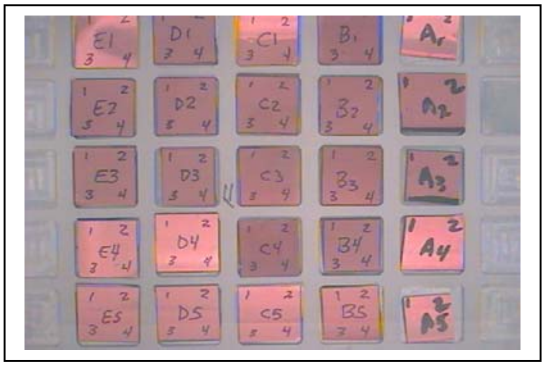


Figure 6 :Image Capture (before debonding)

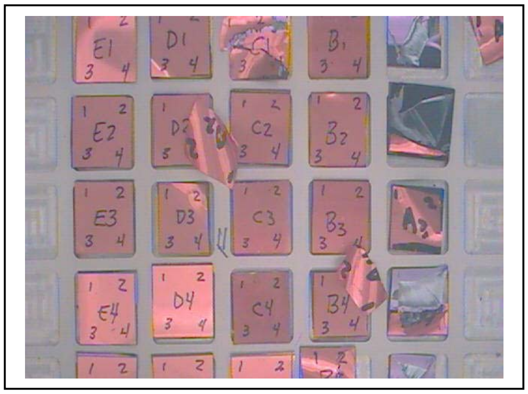
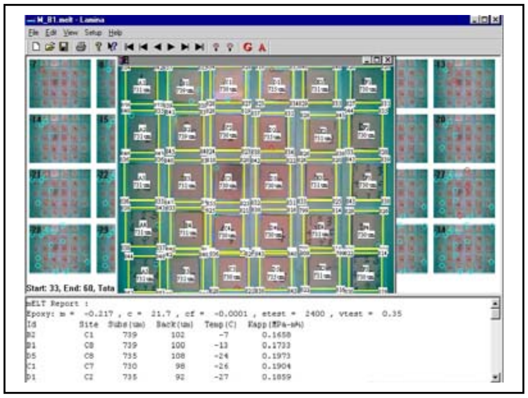


Figure7: Image Capture (After delamination)



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mELT Report :
Epoxy: m =  -0.217 , c =  21.7 , cf =  -0.0001 , etest =  2400 , vtest =  0.35
Id         Site  Subs(um)  Back(um)  Temp(C)  Kapp(MPa-m½)
B2          C1      739       102       -7       0.1658
B1          C8      739       100      -13       0.1733
D5          C8      735       108      -24       0.1973
C1          C7      730        98      -26       0.1904
D1          C2      735        92      -27       0.1859
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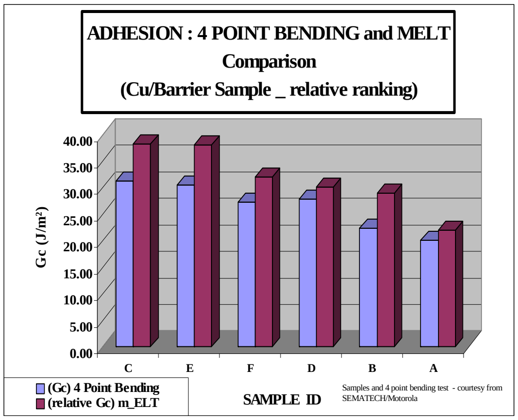
Figure 8 : Image Analysis and reporting

It is possible to screen various low k/copper/ barrier film stacks with a throughput of up to 100 wafers per week. Test coupons are examined for their loci of failure at the onset of delamination. This step reveals which layer on the film stack has failed and whether the failure is an adhesive or a cohesive failure. Supporting tools include optical microscopes, AFMs, XPS. Several results have been reported recently for thin film adhesion testing using the MELT methodology in the literature [1] [10,11,12,13].

The results for a set of Copper film/ barrier material silicon wafers obtained through the MELT test was compared with results obtained from the 4 point bending technique [14, 15] . The 2 techniques showed strong correlation in their relative adhesion strengths. (Figures)



Samples and 4 point bending test - courtesy from SEMATECH/Motorola

Repeatability expected from the MELT technique is generally within the order of 10%.

Conclusions

Two complementary tools for the rapid evaluation and characterization of thermal mechanical properties low k and copper have been discussed. Properties like film adhesion strength, thermal stability, stress hysteresis, thermal desorption (outgassing), in situ film shrinkage, reflectivity and sheet resistivity changes can be obtained.

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