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Dielectric Spectroscopic Detection of Early Failures in 3-D Integrated Circuits

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Abstract

The commercial introduction of three dimensional integrated circuits (3D-ICs) has been hindered by reliability challenges, such as stress related failures, resistivity changes, and unexplained early failures. In this paper, we discuss a new RF-based metrology, based on dielectric spectroscopy, for detecting and characterizing electrically active defects in fully integrated 3D devices. These defects are traceable to the chemistry of the insulation dielectrics used in the through silicon via (TSV) construction. We show that these defects may be responsible for some of the unexplained early reliability failures observed in TSV enabled 3D devices.

Introduction

Emerging nanoelectronic devices are expected to operate at higher current densities, with lower voltage tolerances and higher electric fields, which make them more vulnerable to failure. This situation is further complicated by the introduction of new materials and integration schemes; for example, the commercial introduction of three dimensional integrated circuits (3D-ICs) has been hindered by reliability challenges, such as stress related failures, resistivity changes, and unexplained early failures[1, 2]. The performance demands of such devices appear to conflict with the need for reliability. Thus, such devices, with new designs and materials, require careful failure analyses to find new models for both individual failure mechanisms and the possible interaction between them. Proper tradeoffs in the early design stage are now a prerequisite for managing yields, despite being a dominating challenge.

Classically, basic models relate reliability to yield by assuming that the device is constructed out of perfect materials, with well-defined and uniform chemical properties. It is further assumed that reliability is correlated to packaged die yield because the same defects responsible for yield failures are most probably also responsible for reliability failures[3–6]. Thus, early reliability failures due to extrinsic failure modes, corresponding to defects are present in the test structure the majority of early field failures is supposed to be due to process defect density from random “out of control” processing incidents, and are well described by the Poisson yield model[7]. In reality, the reliability of electronic devices is

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constrained by materials of construction, use conditions and the structures of the devices. It is also known that intrinsic factors pertaining to the inherent nature of the materials of construction, the integration scheme and the resultant geometry can also contribute to early failures. Hence, there is the need for effective reliability analyses to determine the dominant failure mechanisms, and to predict appropriate device lifetimes[8].

In this paper, we discuss a tool for such an analysis, an application of broadband radio frequency (RF) in non-destructive identification and characterization of electrically active defects present in the materials of construction, which could be responsible for early reliability screening failures in integrated circuit devices[9]. We focus on how low frequency (< 300 MHz) RF signal scattering, specifically insertion losses (i.e., S_{12} and S_{21}), can be used to detect and monitor failure rate in such devices, without resorting to the traditional destructive physical analysis.

Electromagnetic (EM) waves interacting with material induces transient non-equilibrium that could result in non-equilibrium events such as polarization, vibration, reorientation, translation and rotation, etc. The macroscopic and local fields in material are modified by relaxation events such as surface charge depolarization fields that oppose the applied field, which results in net energy absorption reflected in the RF loss spectra. Such relaxation events could be leveraged to probe the nature of dominant processes involved in the polarization-depolarization dynamics of dielectric materials[10]. In amorphous materials, interfacial polarization results from the accumulation of charges at the interfaces between phases that have differences in electrical properties (e.g., differences in dielectric constant and conductivity), as such interfaces act as physical barriers that reduce charge displacement. Microwave and RF-dielectric material interactions are reasonably granular and uniquely suitable for studying the buried structures and material interfaces inherent in integrated circuit devices. Such metrologies are critical to the development of emerging nano-scale electronic devices, which are dominated by material interfaces[11, 12]. The RF based techniques offer the additional advantage of interrogating the entire device under test (DUT). Thus, detailed studies of broadband RF interaction with integrated circuit devices should, in principle, enable fast in-situ deduction of physical changes in the entire device, defects, failure modes and mechanisms without the need for physical failure mode analyses (FMA).

Experimental

For this study, SEMATECH built two-level stacked dies were used, and the details of fabrication are reported elsewhere[13]. The samples were stored at room temperature for more than nine months before being used for this experiment. Thermal cycling was used to induce material transformation and degradation in the devices as described elsewhere. In brief, the target samples were heated from 30°C to 150°C and then cooled to 30°C repeatedly in laboratory ambient.

The S-parameters measurements were first measured on the DUT in the “as-received” state, prior to thermal cycling, to determine their baseline RF signal characteristics. The DUTs were re-measured, ex-situ, after 500 thermal cycles, to monitor the consequences of the

treatment. RF scattering data was obtained based on a two-port measurement on a PNA N5230A vector network analyzer (Keysight, Santa Clara, CA)[§]. The through-reflect-line (TRL) calibration method was used to remove cables and connectors in order to establish the reference plane at the tip of the ground-signal-ground (GSG) probes. In this study, the transient insertion losses at the low frequency end of the broadband RF spectrum (below 300 MHz) were used to detect and characterize the response of the DUT. As discussed below, while changes were observed in both the reflective (S_{11}/S_{22}) as well as in the insertion loss (S_{12}/S_{21}), we chose to use the latter as metrics for monitoring the defects in the liner dielectric. In this report we arbitrarily define a failure criterion of 50% insertions signal loss ($S_{21} < 3\text{dB}$) at 210 MHz.

All the data in this study was obtained from a single 300 mm wafer, hence this report does not comprehend the other sources of variability, e.g., lot-to-lot, wafer-to-wafer, etc. However, since most advance fabrication processes use single wafer tools, we effectively have batches of one wafer. Thus, the variability we observed on the single wafer probably represents most of the expected variability for the process flow used in the fabrication of the prototypes studied.

Results and Discussion

The response of the DUT to broadband RF frequencies depends strongly on the intrinsic properties of the material composition and physicochemical nature of material interfaces. For an example, we have shown elsewhere that the local chemistry of the encapsulation dielectric films determines the mechanical properties of the encapsulant[14]. Specifically, high local silanol ($\text{R}_3\text{Si-OH}$) concentration and the resultant decreased Young's modulus of the encapsulating dielectric (SiO_2) afford fragile films with negative packaging yield consequences. In general, dielectrics such as, SiO_2 , are "difficult" materials from reliability perspectives, for reasons such as electron trapping, hot electron aging, and TDDB, possibly due to these dangling bonds and reactive functional groups (e.g., hydroxyl groups)[15, 16]. For a variety of manufacturability issues, sub-atmospheric chemical vapor deposited ozone-tetraethyl orthosilicate (SACVD O_3 -TEOS) films are frequently used as the isolation liner dielectric in the high aspect ratio vias of Cu-TSV's.[17] However, O_3 -TEOS films are notoriously hygroscopic,[18] and known to contain significant concentrations of electrically active defects.[19] There are numerous reliability challenges in using O_3 -TEOS; for example, the time dependent dielectric breakdown (TDDB) lifetime of such O_3 -TEOS liner is far below ten years due to the degradation of the liner, accompanied by the release of byproducts such as water and hydrogen. The liner material degradation byproducts suggest temperature driven chemical reactions in the O_3 -TEOS film, such as the condensation of the silanol ($\text{R}_3\text{Si-OH}$) functional groups, as contributors to the observed short TDDB lifetimes[20, 21].

Changes in RF scattering with thermal cycling have been described in detail elsewhere:[11] but for the purposes of this report, we focus on the changes occurring at the low frequency

[§]Certain commercial equipment, instruments, or materials are identified in this report in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

(< 300MHz) end of the spectrum. Figure 1 compares failure rates based on the insertion loss (S_{21}) magnitude, at 210 MHz, of 'as received' and up to 2000 thermal cycles, measured at 500 cycle intervals, of a prototypical TSV device from a 300 mm wafer. That the number of failing dies decrease with thermal anneal after 500 thermal cycles suggests transformation in the materials of construction with heat treatment. This is tantamount to changing the characteristic product failure distribution with post processing anneals. Interestingly, not all dies recovered from the 'anneal'. The hard failed parts showed very large RF signal loss. This difference is not an artifact of the measurement; it was observed on several samples using the same calibration procedure.

Figure 2 compares the locations of the failing devices in the 'as-received' and after 500 thermal cycles. The across wafer distribution of failing devices is reminiscent of the across wafer distribution of the local bond structure, which is determined by the details of the film formation process. For large substrates, such as a 300mm diameter silicon substrate, there is site-to-site variability in the process chemistry due to non-uniformities in the local gas compositions, substrate temperature, etc.[22, 23] We have shown elsewhere that the distribution of silicon oxide film modulus across a wafer depend on the local concentration of Si-OH in the film[11]. Based in part on the similarity of the geographic distribution of defects observed in this work (Figure 2) and the within wafer HARP film modulus distribution, we postulate that the changes in early failures and the subsequent annealing effects are due to dangling bonds from the incomplete condensation reactions within the liner dielectric film. In brief, the insertion loss (S_{21}) stems from the orienting effects of the terminated dangling bonds from the electric field of the microwave photons. The absorbed energy transfers between rotational and vibrational states in condensed phases, and affects the chemical equilibrium and the perfectly isotropic statistical orientation of the molecules in the dielectric film.

Amorphous silica (such as $O_3/TEOS$) is composed of bulk tetrahedral SiO_4 units, dangling bonds terminated with various functional groups, such as silanol (R_3Si-OH), siloxane bridge ($R_3Si-O-SiR_3$), and other hydrated surface functionalities. The early defects could be the result of the polarization of silanols in the dielectric; and the observed change after 500 thermal cycles is most probably due to condensation reactions involving silanols[24], such as depicted in equation 1. Applying heat slowly removes vicinal (hydrogen-bonded) silanols by condensation and elimination of water (Equation 2)[25, 26].

The silanols can also react with to generate well-defined charge transfer species (as depicted by Equation 2). In fact reactions are essential for metal barrier films to bind to the isolation liner dielectric[27]. Thus, the "hard failed" that persists after extensive annealing could also be silo-metal complexes formed by the condensation of silanols with the tantalum in the barrier layers, as depicted in equation (3)

Based on these observations we propose that the early failures involve RF scattering by dangling bonds in the liner oxide (ozone/TEOS film). The dangling bonds react upon modest heating to form siloxanes, with the elimination of water.

Conclusions

We have presented an approach of detecting and characterizing dangling bond defects in dielectrics, based on changes in the low frequency (300 MHz) dispersion of RF scattering. Unlike the current state of the art spectroscopic methods, this new method does not need ‘line of sight’ of the analyte, and can be used to interrogate buried structures and interfaces. The utility of the new method was demonstrated in characterizing ozone-TEOS films. While the immediate application has been in characterizing the reliability of prototypical 3D integrated circuits, the utility of this new technique could be extended to material characterization in general, for probing buried material interfaces in particular.

Based on the changes in the low frequency (300 MHz) dispersion of RF scattering, we propose an evolution of ozone-TEOS liner films under thermal cycling. The “as deposited” film contains silica domains surrounded by silanols, which react upon modest heating to form siloxanes, with the elimination of water. These siloxanes are subsequently incorporated into the silica domains, accompanied by the nucleation and growth of voids, with further heating. Furthermore, the silanols at the copper-barrier interface form charge transfer complexes with the metal, which are responsible for the metal bonding to the dielectric.

While the immediate application has been in characterizing the reliability of prototypical 3D integrated circuits, the utility of this new technique could be extended to material characterization in general, for probing buried material interfaces in particular. This low frequency RF technique could be optimized to compete favorably with other thin film metrology techniques, such as ellipsometry and FTIR, for mass production environments. Unlike the current state of the art spectroscopic methods, this new method does not need ‘line of sight’ of the analyte, and can be used to interrogate buried structures and interfaces.

References

1. Chen K-N, Tu K-N. Materials challenges in three-dimensional integrated circuits. *MRS Bulletin*. 2015; 40:219–222.
2. Jiang T, Im J, Huang R, Ho PS. Through-silicon via stress characteristics and reliability impact on 3D integrated circuits. *MRS Bulletin*. 2015; 40:248–256.
3. Kuper, F.; van der Pol, J.; Ooms, E.; Johnson, T.; Wijburg, R.; Koster, W., et al. Relation between yield and reliability of integrated circuits: experimental results and application to continuous early failure rate reduction programs; Reliability Physics Symposium, 1996. 34th Annual Proceedings., IEEE International; 1996. p. 17-21.
4. Van Der Pol JA, Kuper FG, Ooms ER. Relation between yield and reliability of integrated circuits and application to failure rate assessment and reduction in the one digit FIT and PPM reliability ERA. *Microelectronics Reliability*. 36:1603–1610. 11// 1996.
5. Evans, F.; Wall, J.; Hancock, B.; Brusius, P.; Mitchell, M. Reliability in VHSIC-level 1.25 μm radiation-hard CMOS IC devices; University/Government/Industry Microelectronics Symposium, 1991. Proceedings., Ninth Biennial; 1991. p. 107-112.
6. Prendergast, JG. Reliability, yield and quality correlation for a particular failure mechanism; Reliability Physics Symposium, 1993. 31st Annual Proceedings., International; 1993. p. 87-93.

7. Riordan, WC.; Miller, R.; Sherman, JM.; Hicks, J. Microprocessor reliability performance as a function of die location for a 0.25 μ m, five layer metal CMOS logic process; Reliability Physics Symposium Proceedings, 1999. 37th Annual. 1999 IEEE International; 1999. p. 1-11.
8. Wyrwas, E.; Condra, L.; Hava, A. Accurate quantitative physics-of-failure approach to integrated circuit reliability. Las Vegas, NV: IPC APEX Expo; 2011. p. 13
9. Bezat AG, Montague LL. The effect of endless burn-in on reliability growth projections: A. G. Bezat and L. L. Montague. Proc. IEEE Reliability Symposium, Washington D.C., January 23–25, p. 392 (1979). Microelectronics Reliability. 20:756–757. // 1980.
10. Jonscher AK. Dielectric relaxation in solids. Journal of Physics D: Applied Physics. 1999; 32:57.
11. You L, Okoro CA, Ahn J-J, Kopanski J, Franklin RR, Obeng YS. Broadband Microwave-Based Metrology Platform Development: Demonstration of In-Situ Failure Mode Diagnostic Capabilities for Integrated Circuit Reliability Analyses. ECS Journal of Solid State Science and Technology. 2015 Jan 1.4:N3113–N3117. 2015.
12. You, L.; Ahn, JJ.; Hitz, E.; Michelson, J.; Obeng, YS.; Kopanski, JJ. Electromagnetic Field Test Structure Chip for Back End of the Line. presented at the 2015 IEEE Intl/Conf. on Microelectronic Test Structures; Phoenix, AZ, US. 2015.
13. Olson, S.; Hummler, K. TSV reveal etch for 3D integration; 3D Systems Integration Conference (3DIC), 2011 IEEE International; 2012. p. 1-4.
14. Okoro CA, Obeng YS. A case study on the impact of local material chemistry on the mechanical reliability of packaged integrated circuits: Correlation of the packaging fallout to the chemistry of passivation dielectrics. Thin Solid Films. 520:5060–5063.
15. Chua L-L, Zaumseil J, Chang J-F, Ou ECW, Ho PKH, Sirringhaus H, et al. General observation of n-type field-effect behaviour in organic semiconductors. Nature. 434:194–199. 03/10/print 2005. [PubMed: 15758994]
16. Mathijssen SGJ, Kemerink M, Sharma A, Cölle M, Bobbert PA, Janssen RAJ, et al. Charge Trapping at the Dielectric of Organic Transistors Visualized in Real Time and Space. Advanced Materials. 2008; 20:975–979.
17. Li Y, Oba Y, Wu C, Van Huylbroeck S, Van Besien E, Vereecke G, et al. Hydrogen outgassing induced liner/barrier reliability degradation in through silicon vias. Applied Physics Letters. 2014; 104:142906.
18. Obeng YS, Steiner KG, Velaga AN, Pal C-S. Dielectric Materials for Advanced VLSI and ULSI Technologies. AT&T Technical Journal. 1994; 73:94–111.
19. Kim HU, Rhee SW. Electrical Properties of Bulk Silicon Dioxide and SiO₂ / Si Interface Formed by Tetraethylorthosilicate-Ozone Chemical Vapor Deposition. Journal of The Electrochemical Society. 2000 Apr 1.147:1473–1476. 2000.
20. Nguyen S, Dobuzinsky D, Harmon D, Gleason R, Fridmann S. Reaction Mechanisms of Plasma- and Thermal-Assisted Chemical Vapor Deposition of Tetraethylorthosilicate Oxide Films. Journal of The Electrochemical Society. 1990; 137:2209–2215.
21. Huffman M, McMillan P. Infrared and raman studies of chemically vapor deposited amorphous silica. Journal of Non-Crystalline Solids. 76:369–379.
22. da Silva ANR, Morimoto NI, Bonnaud O. Tetraethylorthosilicate SiO₂ films deposited at a low temperature. Microelectronics Reliability. 40:621–624.
23. Morimoto N, Swart J. Development of a Cluster Tool and Analysis of Deposition of Silicon Oxide by TEOS 2 PECVD. MRS proceedings. 1996:263.
24. Jeong S, Kim D, Park BK, Lee S, Moon J. Influence of silanol groups on the electrical performance of organic thin-film transistors utilizing organosiloxane-based organic–inorganic hybrid dielectrics. Nanotechnology. 2007; 18:025204.
25. Mania P, Conrad S, Verel R, Hammond C, Hermans I. Thermal restructuring of silica-grafted -CrO₂Cl and -VOCl₂ species. Dalton Transactions. 2013; 42:12725–12732. [PubMed: 23752559]
26. Mania P, Verel R, Jenny F, Hammond C, Hermans I. Thermal Restructuring of Silica-Grafted TiCl_x Species and Consequences for Epoxidation Catalysis. Chemistry – A European Journal. 2013; 19:9849–9858.

27. Satta A, Schuhmacher J, Whelan CM, Vandervorst W, Brongersma SH, Beyer GP, et al. Growth mechanism and continuity of atomic layer deposited TiN films on thermal SiO₂. *Journal of Applied Physics*. 2002; 92:7641–7646.

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Figure 1.
Fraction of Device Population Failing -3dB (50% Insertion losses (S_{21})) criterion

0 Thermal Cycles

500 Thermal Cycles

Figure 2.

Comparison of the geographic distribution of failed dies in 'as-received' and post 500 thermal cycles dies (Y = 19 is the bottom row, and -X is the left of the wafer. The failing dies after 500 thermal cycles are concentrated in the bottom half of the wafer.