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Interface Engineering for High Interfacial Strength between SiCOH and pSiCOH ILDs and Diffusion Caps

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ABSTRACT

The integration of low and ultralow-k SiCOH dielectrics in the interconnect structures of VLSI chips involves complex stacks with multiple interfaces. Successful fabrication of reliable chips requires strong adhesion between the different layers of the stacks. A critical interface in the dielectric stack is the interface between the SiCNH diffusion cap and the SiCOH intra- and interlevel dielectric (ILD). It was observed that, due to the original deposition conditions, the interface layer was weakened both by a low adhesion strength between SiCNH and SiCOH and by the formation of an initial layer of SiCOH with reduced cohesive strength. The manufacturing process has been modified to engineer this interface and obtain interfacial strengths close to the cohesive strengths of the bulk ILDs. The paper discusses the causes for the original low interfacial strength and presents an approach for enhancing it by engineering the interface to the cap for both the dense SiCOH and porous pSiCOH ILDs.

INTRODUCTION

The replacement of silicon dioxide ULSI interconnect dielectric (ILD) with low-k [1-3] and ultralow-k dielectrics [4, 5] added significant complexity to the integration of the interconnect structure. This back end of the line (BEOL) structure is built of a complex stack with multiple interfaces, therefore successful fabrication of the integrated devices requires high interfacial strength between the different layers in contact in the stack. Such layers comprise the interconnect Cu metal, metal barriers, the insulating dielectric, dielectric diffusion barriers, hardmasks.

In the interconnects of older technology generations (pre 90 nm), which used as the ILD silicon dioxide characterized by high cohesive strength, high modulus and hardness, the adhesion between the layers of the stack was strong and did not pose a special integration problem. Replacement of the oxide with the SiCOH dielectric [1] introduced in the BEOL a material with significantly reduced mechanical properties and a cohesive strength inferior to that of the oxide. In spite of these weaker mechanical properties, the initial integration stages of SiCOH in the BEOL did not reveal any adhesion problems and the structures passed the wafer level reliability testing. However, when the individual chips were diced and packaged, delaminations occurred in the BEOL structure due to the additional stresses imposed on the structure during the mechanical dicing and by the packaging processing and materials [6]. Analysis of the delaminated interfaces showed that the delamination occurred between the SiCNH diffusion cap and the SiCOH ILD.

This paper will discuss the identification of the causes of the interfacial failure and will then present a solution for improving the interfacial strength between the SiCNH cap layer and the SiCOH dielectric based on the understanding of the failure mechanism.

EXPERIMENTAL

The studies have been performed using blanked SiCNH, SiCOH and porous pSiCOH films deposited on 300 mm Si wafers. The SiCNH/SiCOH or SiCNH/pSiCOH stacks have been prepared using manufacturing tools and recipes. The preparations of SiCOH and porogen based porous pSiCOH films have been discussed elsewhere [1, 4, 7-9]. Cross-sections of the delaminated BEOL stack showed that the delaminations occurred between the SiCOH dielectric and the underlying SiCNH Cu diffusion cap [6] indicating that this interface is characterized by the weakest interfacial strength. In order to evaluate the effects of different approaches for adhesion improvement, the adhesion strength was tested in each case using the 4 point bending method described elsewhere [10], using a structure illustrated in Figure 1.a. The delamination can occur during this test in two different modes: (i) adhesive failure, i.e. clean interfacial delamination between the two materials; (ii) cohesive failure in the bulk of one of the films. The two failure modes are illustrated in Figure 1.b.

Electron microscopy techniques were used initially to shed some light on the failure mechanism. The identification of the type of failure was done by analyzing both surfaces of the delamination (both the top and the bottom delaminated surfaces) of the SiCNH/SiCOH stack using surface analytical techniques. Time-of flight TOF-SIMS was found to have the best resolution and be the most useful technique for characterizing the delaminated layers and distinguish between the two types of failure modes. TOF-SIMS

sputter depth profiling analysis was carried out using a model TOF IV, Time of Flight SIMS instrument manufactured by Ion TOF GmbH. Sputter depth profiling was accomplished using 500 eV or 1KeV Cs sputtering with the profiles being collected in the negative ion mode.

A number of different techniques have been tried to improve the interfacial strength between the SiCNH and SiCOH layers, such as: (i) modifications of the top surface of SiCNH; (ii) use of intermediate adhesion layers between SiCNH and SiCOH; (iii) combinations of (i) and (ii). Surface modifications of type (i) included the treatment of the SiCNH surface with inert plasmas of Ar or He, or treatment with reactive plasmas of O₂, CO₂, N₂, or SiH₄ before the deposition of SiCOH, or thermal exposure of the SiCNH to the SiCOH precursor before initiating the deposition of the SiCOH by PECVD. Modifications of type (ii) included the deposition of an amorphous a-Si layer or a layer of HOSP between SiCNH and SiCOH, or in-situ grading the SiCOH layer from SiCNH to bulk SiCOH compositions.

RESULTS and DISCUSSIONS

Interfacial failure mechanism

Figure 2 presents the depth profile obtained with TOF-SIMS from the SiCNH site of a delamination of SiCOH deposited directly on SiCNH without any additional treatment. The figure shows that the analyzed layer on top of the Si substrate has a uniform composition corresponding to the bulk SiCNH and there is no evidence of any residual material from SiCOH on the analyzed surface. A similar analysis of the SiCOH side of the delamination showed no residual SiCNH on the SiCOH film, indicating that in this case the failure during this adhesion test was of adhesive type. The 4 point bending test measured in this case an adhesion strength of only $\sim 2.0 \text{ J/m}^2$ compared to the cohesive strength of $\sim 6.0 \text{ J/m}^2$ for the SiCOH film with a dielectric constant $k=3.0$. Similar adhesive strength was obtained for SiCOH films prepared by different processes.

Some of the type (i) SiCNH surface modification treatments did improve the interfacial strength, especially those that oxidized the surface and increased the number of SiO bonds at the surface of SiCNH. Adhesion strength values as high as 3.7 J/m^2 were obtained with such surface modifications, but these values were still significantly lower than the cohesive strength of bulk SiCOH. TOF-SIMS analysis of the SiCNH side of the delamination of such a stack is presented in Figure 3 which shows the composition profile into the bulk of the SiCNH.

The surface of the SiCNH film, which has been treated in this case with oxidizing plasma, has been converted to an oxide, as indicated by the oxide peak. The carbon content in the oxidized layer has been reduced to a minimum. Figure 3 shows the presence of traces of increased carbon content on top of the oxide layer indicating that residue from SiCOH was left on the SiCNH side of the delamination. The failure in this case was of cohesive type, inside the SiCOH film. The measured interfacial strength ($\sim 3.7 \text{ J/m}^2$), being much lower than the cohesive strength of bulk SiCOH (6 J/m^2), indicates that the delamination occurred in a SiCOH layer of reduced cohesive strength.

TEM analysis of delaminated SiCNH/SiCOH stacks showed that the delaminations occurred at about 50 - 100Å into the SiCOH film. TEM was used to shed some light into the differences between the observed adhesion values of SiCOH to the untreated and

oxidized SiCNH surfaces. Figure 4 compares TEM analysis of FIB prepared cross-sections of SiCNH/SiCOH structures having two different interfacial strengths. Figures 4.a & b show the micrographs of the cross-sections while Figures 4.c & d. show transmission line scans of the intensity across the structures in the two specimens.

Both investigated samples showed a contrast peak near the interface between the SiCNH and SiCOH films. An intermediate brighter layer, indicating material of lower density, is observed in SiCOH near the interface with the SiCNH cap (Figures 4.a & b). The intensity line scans in Figures 4.c & d, confirmed this lower density in the intermediate layers, showing higher transmission through the brighter layers in Figure 4 a. & b. It was further observed that the width of this transition layer of lower density is larger in the structure characterized by lower interfacial strength (Figure 4.c) than in the structure characterized by higher interfacial strength (Figure 4.d). Analysis of the composition across the structures shown in Figure 4 by electron energy loss spectroscopy (EELS) indicated that the transition layer contained higher C concentration than the rest of the SiCOH dielectric.

The observations described above can be summarized as follows:

- Deposition of SiCOH directly on precleaned SiCNH leads to adhesive failure indicated by a clean SiCNH delaminated surface
- Surface treatments of SiCNH that cause the formation of an oxide interlayer lead to improved interfacial strength with cohesive failure in SiCOH near the interface with SiCNH
- The weak interfacial strength was identified to be associated with the existence of a layer of SiCOH of lower cohesive strength associated with an increased C concentration in this layer. As will be explained next and illustrated in Figure 5, this C-rich SiCOH layer near the interface is created by the PECVD process used for the deposition of SiCOH.

Interface engineering for improved interfacial strength of SiCNH/ SiCOH

In a typical PECVD deposition procedure the precursors for film deposition are introduced first in the PECVD reactor and the RF power that ignites and sustains the plasma is applied after the flow and pressure in the reactor are stabilized. As illustrated in Figure 5.a, the RF power does not actually reach its preset value instantaneously but only after an initial ramp (that can be very short). During this initial ramp, until the RF reaches full power, the precursor is only partially dissociated in the plasma and, as a result, more C is incorporated in the initial layer of the SiCOH film than in its bulk.

In order to prevent the formation of the weak layer with increased C content we modified the deposition sequence, as illustrated in Figure 5.b, whereby the SiCOH precursor is introduced in the reactor after the RF power reaches the preset value. Under such conditions, the precursor is dissociated at controlled and constant RF power and the film has uniform composition without an excess C content. We further optimized the SiCOH deposition process to create a graded layer wherein the C concentration increased from about zero near the SiCNH surface to its concentration in bulk SiCOH: we deposited first a thin initial PECVD oxide-like layer, then adjusted the deposition conditions to gradually reach the bulk SiCOH composition and continued with the deposition of the SiCOH film to the desired thickness. The described transition layer is

deposited using the same chemicals as used for the deposition of bulk SiCOH but with different concentrations in the gas feed, i.e. higher oxygen and lower SiCOH precursor flow. A typical transition layer consists of 2-5 nm of oxide and 20-30 nm of graded film.

The graded transition layer has a higher dielectric constant than the SiCOH film, but its impact on the effective dielectric constant can be minimized by minimizing the thickness of the graded layer. It is therefore important to control the deposition process to minimize the final thickness of this layer. The composition of a typical SiCNH/SiCOH structure with a graded layer is shown in Figure 6 which presents the TOF-SIMS profile through such a structure. The different layers of the stack are marked in the figure. An interfacial strength of 5.0 J/m² has been obtained in the 4 point bending test for such an optimized structure. The TOF-SIMS analysis of the locus of failure showed that the delamination occurred through cohesive failure in SiCOH at the top of the oxide-like layer, as shown in Figure 7. The C peak on the surface of the oxide layer is an indication of SiCOH resides on the SiCNH side of the delamination.

The transition layer developed for dense SiCOH with a dielectric constant $k=3.0$ has been applied also for a modified SiCOH with a dielectric constant $k=2.7$ [3] resulting in this case too in a significant improvement in its interfacial strength to SiCNH, as shown in Table I.

Improved interfacial strength for SiCNH/ pSiCOH.

The adhesion strength of porous pSiCOH deposited on pristine SiCNH was again only 2.0 J/m², compared to pSiCOH's cohesive strength of 3.5 J/m² (see Table I). The SiCNH/pSiCOH interfacial strength has been enhanced too by introducing a transition layer, similar to the one described for dense SiCOH, between SiCNH and pSiCOH. Because the pSiCOH is prepared by addition of a porogen precursor to the SiCOH precursor in the deposition process [8, 9], the fabrication of the transition layer is more complicated. The deposition of the transition layer for pSiCOH has to be carefully controlled and the porogen must be introduced into the reactor chamber by ramping it at an optimal rate.

Using the proper deposition conditions, a composition profile similar to the one shown in Figure 6 is obtained for SiCNH/transition layer/pSiCOH structure. The TOF-SIMS composition profile of the SiCNH side of the delamination in a 4-point bend test, presented in Figure 8, shows that delamination occurred in pSiCOH close to the graded layer. The interfacial strength for this optimized structure is 3.5 J/m², equal to the cohesive strength of bulk pSiCOH (Table I). If the deposition conditions are not optimized, a layer of excess C can form in the initial pSiCOH layer, resulting in an interfacial strength of only 2.5 J/m².

Manufacturability issues

The formation of the transition layer needed for the improvement of the interfacial strength between the SiCOH or pSiCOH interconnect dielectric and the SiCNH cap adds complexity to the fabrication of the stack. The space of proper deposition conditions for each layer is limited by manufacturability issues. Formation of foreign material (FM) on the processed wafers must be limited to a minimum for maintaining high yield in

manufacturing. FM has been observed to form on the wafers when non-optimized processing conditions of the transition layer were used, as a result of gas phase nucleation or plasma instabilities. Further tuning of the deposition conditions, especially the ramp and flow rates of the SiCOH precursor, oxygen and porogen, during the fabrication of the transition layer has enabled the elimination of the gas phase nucleation and strong reduction of FM.

SUMMARY

The interfacial strength of SiCOH or pSiCOH interconnect dielectrics to underlying SiCNH caps has been optimized to values close to the cohesive strengths of the corresponding ILD dielectric films. This has been accomplished by preventing the incorporation of excess carbon at the interface between the two materials using a transition layer between the cap and the ILD comprising an oxide-like layer and a layer with graded carbon content.

The processing conditions for the fabrication of graded layer have to be carefully controlled to obtain the right composition of the transition layer and, in the same time, prevent plasma instabilities or formation of FM. Special attention has to be given to the control of the ramp and flow rates of the SiCOH precursor, oxygen and porogen during the fabrication of the transition layers.

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Table I: Improvement of interfacial strength to SiCNH for different SiCOH films

SiCOH version	Bulk-only SiCOH (J/m²)	Substrate treatment (J/m²)	Optimized transition (J/m²)	Cohesive strength (J/m²)
k=3.0	2.0	3.0-4.0	5.0	6.0
k=2.7	2.0	N/A	4.5	5.3
k=2.4	2.0	N/A	3.5	3.5

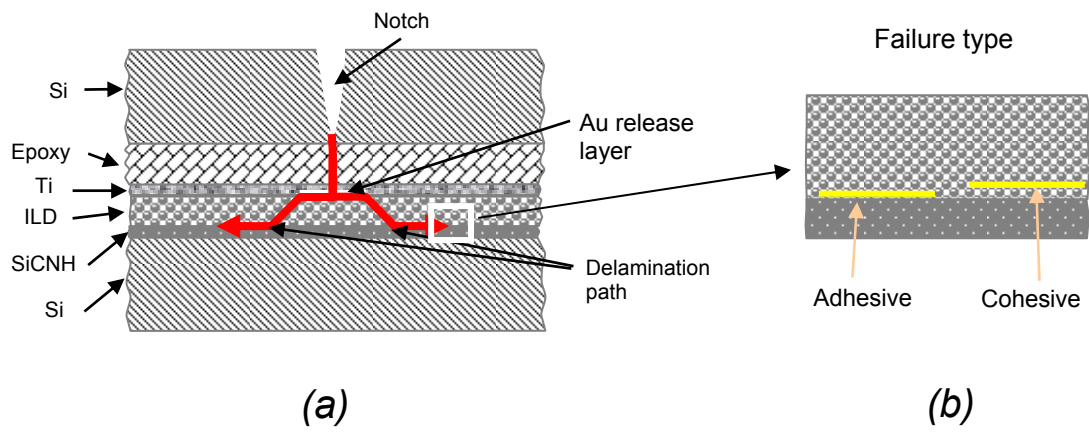


Figure 1: a) Structure used for the 4 point bending test. b) The types of failure occurring during the 4 point bending test.

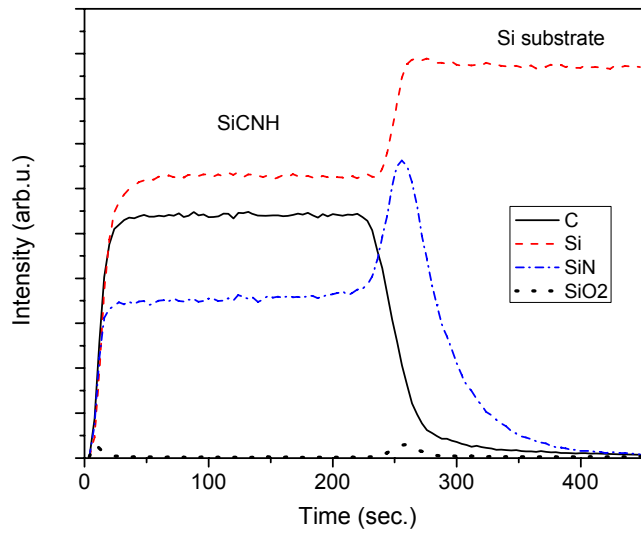


Figure 2: Composition profiles of locus of failure of SiCNH/SiCOH for SiCOH deposited on unmodified SiCNH surface: TOF-SIMS of SiCNH side.

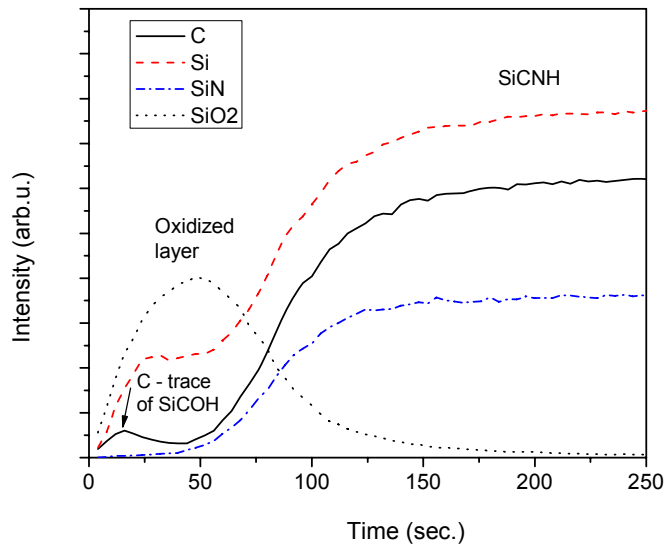


Figure 3: Composition profiles of locus of failure of SiCNH/SiCOH for SiCOH deposited on oxidized SiCNH surface: TOF-SIMS of SiCNH side.

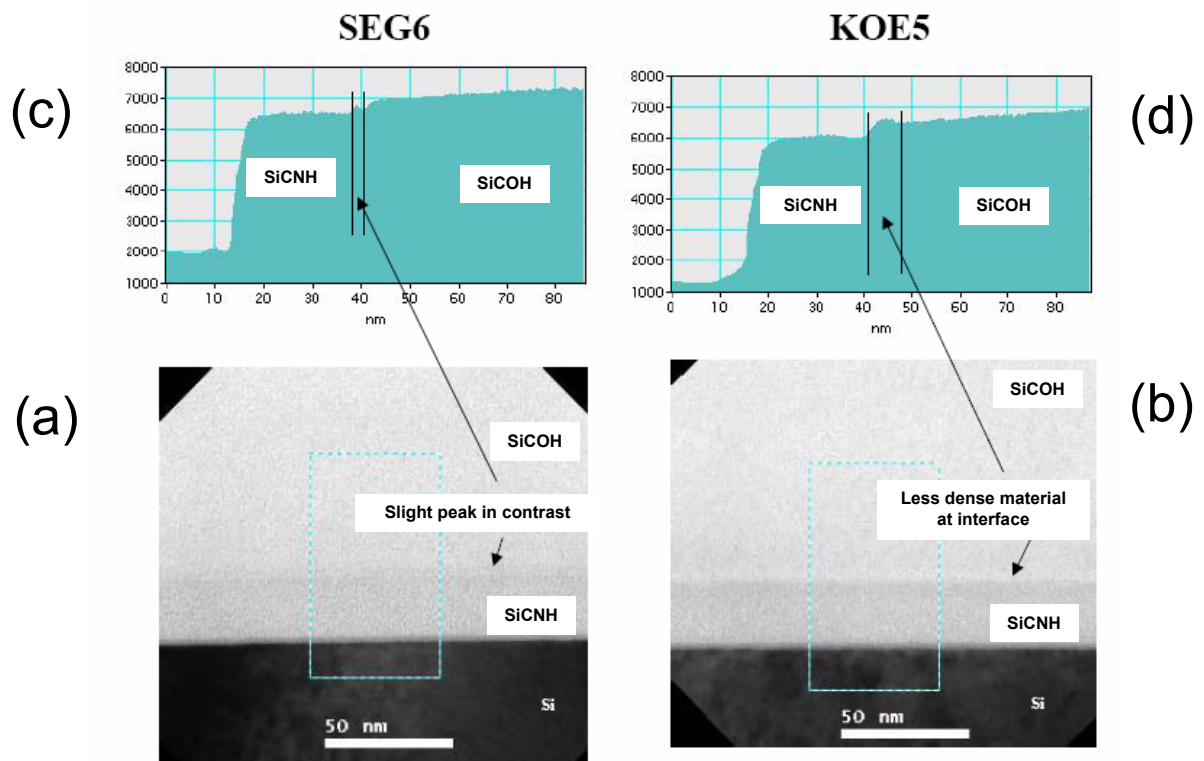
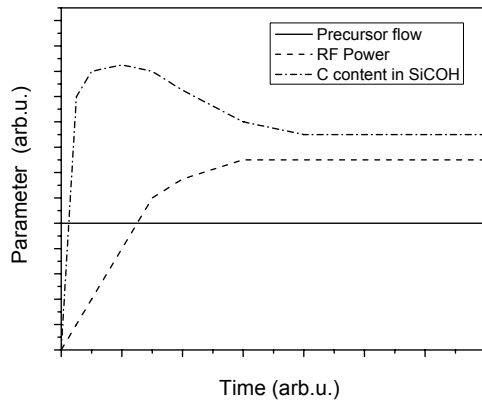
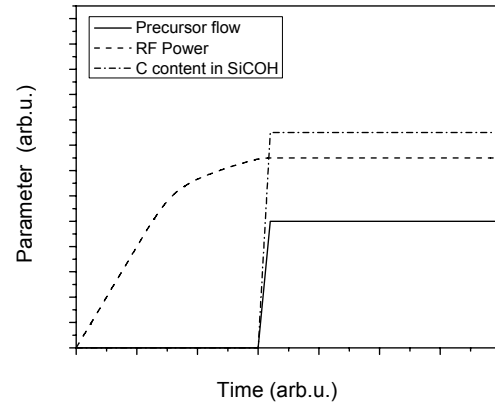


Figure 4: TEM characterization of SiCNH/SiCOH interface. a & b) transmission micrographs through FIB specimens; c & d) intensity profiles across the structures in a & b. (TEM by L.Gignac & J. Rullan)



(a)



(b)

Figure 5: Diagram of PECVD processing flow and the effects on carbon distribution in SiCOH.

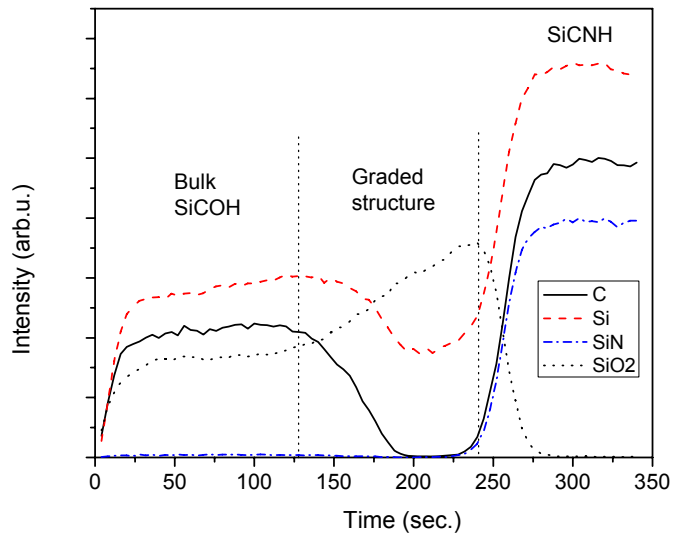


Figure 6: TOF-SIMS profile of optimized SiCNH/SiCOH stack with graded transition layer.

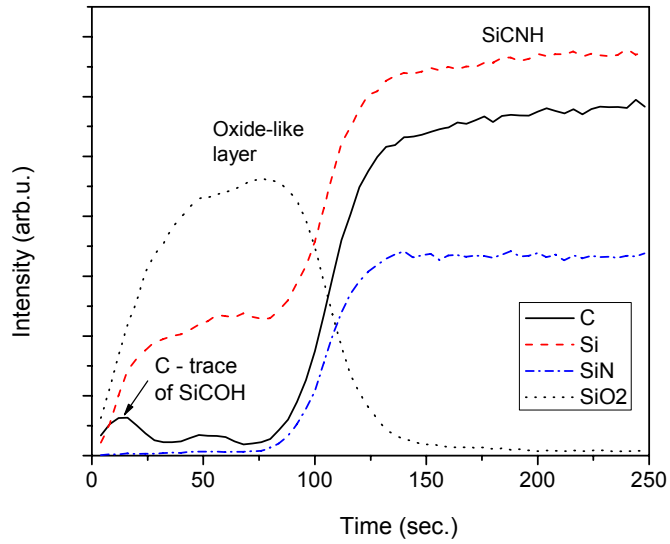


Figure 7: Composition profiles of locus of failure of SiCOH on SiCNH with graded layer – TOF-SIMS of SiCNH side. Trace of SiCOH is observed on the delaminated SiCNH surface.

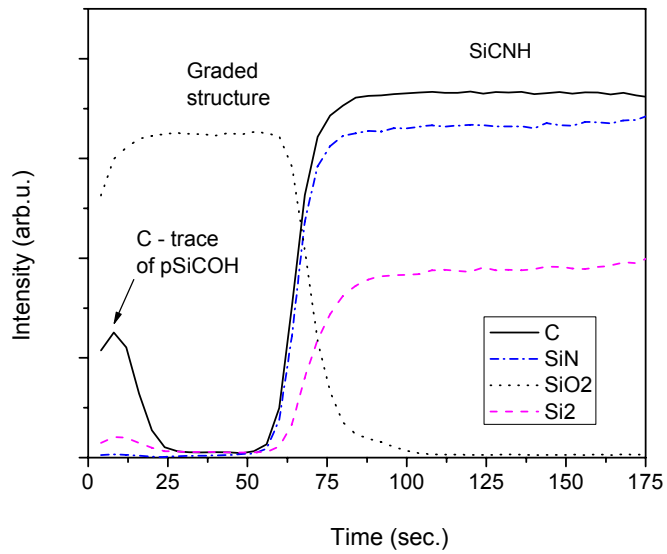


Figure 8: TOF-SIMS of locus of failure of SiCNH/pSiCOH stack with graded transition layer – SiCNH side. The optimized interfacial strength of this structure was $\sim 3.5 \text{ J/m}^2$.