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A Long Term Bio-stability Evaluation of Passivation Layer in the Silicon BiCMOS Technology for Bio-sensors Packaging

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ABSTRACT The silicon technology offers compact integrated read-out circuits and sensors on a single chip for a variety of bio-sensing applications. This includes applications such as glucose sensing, cancer cell detection and so on which rely on dielectric permittivity characterization. Such silicon sensors can provide the best sensitivity when the sensing element is in the closest proximity to the material under test. This translates to direct exposure of the passivation layer of silicon technology to various bio-materials. This work intends to evaluate the long term bio-stability and thus bio-compatibility of the silicon nitride passivation layer of the IHP's 130 nm SiGe BiCMOS technology. A 6-month evaluation is reported here and extrapolations has been deduced. The three types of silicon nitrides (based on the variation of silicon and nitride stoichiometry) has been developed and tested within IHP for the purpose of bio-stability evaluation. Three test fluids namely de-ionized water, glucose solution, and cell culture media (incubated at $37°C$) has been used for a continuous and long term attack on the passivation layer.

INDEX TERMS BiCMOS, bio-stability, cell-culture solution, passivation layer, SiGe, silicon nitride, and tribology.

I. INTRODUCTION

SILICON nitrides are constantly revisited due to their commercial relevance in a several applications wherein they are deployed as bio-materials for orthopedic implants spinal fusion [1], X, Y [4] or as passivation layers in silicon integrated circuits [5]. The widespread exploitation of silicon nitrides are largely due to their noteworthy mechanical properties, thermal stability, bio-compatibility, and xyz among others []. An important domain which benefits from the mechanical properties of the silicon nitride is the integrated circuits. Such silicon microwave and millimeter wave sensors are useful in numerous bio-medical applications such as blood-glucose level sensing, cancer cell detection, lymphatic disease detection and so on [5]– [8]. The fully integrated sensors and read-out structures are The silicon integrated sensing units are typically realized in the top most metal layer of the technology stack for a few important reasons. Not only does it offer the least parasitic sheet resistance but also the highest sensitivity due to closest proximity to the material under test (MUT). This is evident from the technology stack of IHP's 130 nm SiGe BiCMOS technology shown in Fig. 1. The metal layer stack, known as back-end-of-line (BEOL), illustrates the upper most metal layer (Top Metal 2) typically utilized for the sensor implementation. Such sensors implementations can be found in several works such as [5]– [8]. The silicon nitride passivation layer positioned above the top metal layer is the subject of investigation in this work.

The thickness of silicon nitride passivation layer is crucial in a few ways. While thin passivation layers enhance the sensitivity due to stronger penetration depth into the MUT, thick layers may be useful for mechanical stability and ruggedness characteristic to packaging entities. This makes

wear tribology of silicon nitrides predominantly relevant for packaging and sensing applications. (Bring out these points: How robust are they to common fluids ? How thin can SiN be made? How safe are the sensors?). This highlights the underlying significance of performing such long term and reliable study of the passivation layer for quantification of robustness/bio-degradablibilty of few relevant silicon nitrides developed with in IHP. A possible goal may also involve optimization of sensitivity of the sensor while maintaining thick enough layer for material longevity when deployed for extended durations and within organic environment.

A distinction can be made here between bio-compatibility and bio-stability of the silicon nitride. While bio-compatibility evaluates the silicon nitride cytotoxicity [**?**], the bio-stability assesses the physical wear-out of the layer [**?**] due to a variety of ambient impacts. Numerous studies such as [**?**] [**?**] [**?**] [**?**] and [**?**] maintain a consensus in concluding silicon nitride to be compatible with the bio-materials. On the contrary, very few works exist which present a rigorous account of bio-stability evaluation of the silicon nitride passivation layer. The chemical stability in water and wear resistances have been evaluated [**?**]. [2], []. Only few study can be found in literature which intends to perform physical for other materials and a possible electrical characterization scheme. For instance, [9] tested atomic layer deposited Al_2O_3 and Parylene C. Write about it, write about, write about it. Also find more works, also find more works, also find more works.

Evidently, a reliable and quantified long term evaluation on the physical/electrical degradation of the silicon nitride layers (and those developed for monolithic circuits as in IHP) is largely less studied, as far as the author's knowledge extends. The work presented in this article bridges the gap by providing the following valuable insights:

- Long term bio-stability evaluation of three types of silicon nitride layers developed in IHP.
- The evaluation conducted using three types of test fluids typical to bio-environment.
- A rigorous experimentation and measurement of the physical degradation of the nitride layer.
- Thorough dc electrical characterization (post experimentation) to detect any perceivable discontinuities in the metal lines.
- Simple and practical test concepts, characterization methods, and applicable measurement techniques for testing future materials.
- Cytotoxicity evaluation on the considered silicon nitride layer.

As mentioned above, the experimentation has been performed on three types of passivation layers developed within IHP: standard silicon nitride (SiN), silicon rich silicon nitride (SiRN), and silicon oxy-nitride (SiON). An overview of the experimentation conducted within this work is presented in Fig. 2. The tests have been divided into two categories viz. physical characterization and electrical characterization.

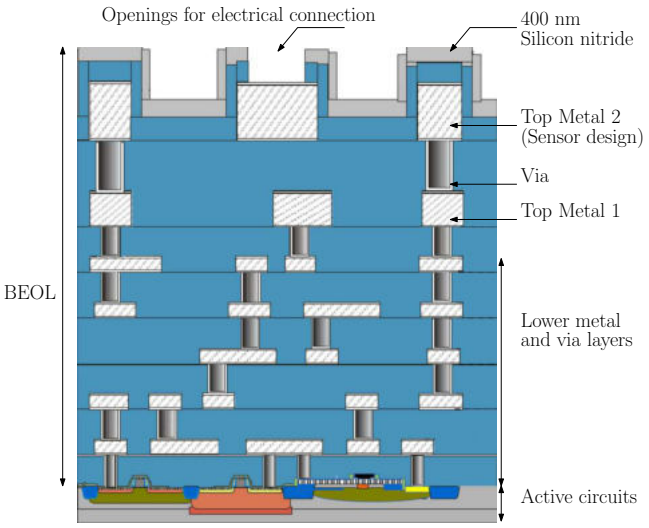

FIGURE 1. The metal stack of IHP's 130 nm SiGe BiCMOS technology node.

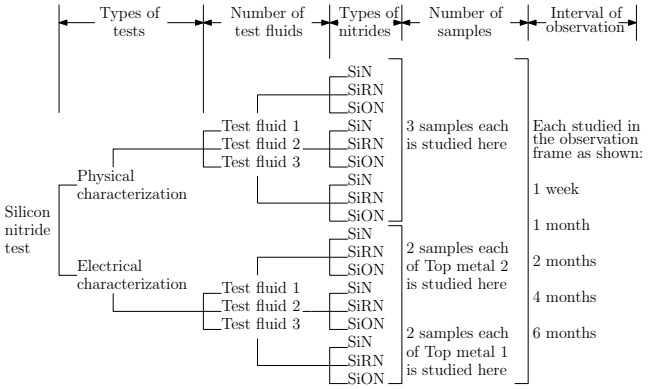

FIGURE 2. Overview of the tests, fluids, nitride types, quantity of the studied samples & observation window of the experimentation conducted in this study.

These terms have been elaborated as follows. Physical characterization revolves around the study of the physical degradation of the silicon nitride passivation layer over a long term observation frame. It has been carried out using ellipsometry and scanning electron microscope (SEM) analysis. The electrical characterization involves dc electrical measurements of the top most metal layers of the BEOL stack. It has been conducted using four-wire dc resistance measurements using dc wedges and a probe station. This test enables the study of test fluid impacts, such as physical cracks, on the metal lines to be used for sensor development. Although the sensor will be implemented on top metal 2 (TM2) layer, this study also includes top metal 1 (TM1) within the electrical characterization. This is intended toward studying damage depth arising due to all prospective effects such as adsorption and so on. Finally, one of the noteworthy aspects of this study is its long term study which is spread over a period of 6 months and comprising of the following observation segments: 1 week, 1 month, 2 months, 4 months, and 6 months. Such experimental results will be of significant relevance to

the scientific community working on medical implants using Si-technology. As mentioned earlier, numerous applications deploy silicon nitrides in their characteristic ways []– []. This, in turn, makes the presented study relevant and scalable to such prominent applications.

The remainder of this paper is organized as follows. Section II presents the three types of the silicon nitride used in the experimentation and includes details such as the physical conditions for fabrication among others. Section III describes the types of tests conducted viz. physical and electrical characterization. Subsequently, a brief account of the types of the test fluids used for creating the physical impact is outlined in Section IV. This is followed by the evaluation set up and experimentation results in Section V. Lastly, the results have been summarized along with the drawn conclusions in Section VI

II. SILICON NITRIDE PASSIVATION LAYER IN IHP'S BICMOS TECHNOLOGY

Silicon nitride is a chemical compound of the elements silicon and nitrogen. Si_3N_4 is the most thermodynamically stable of the silicon nitrides. Hence, Si_3N_4 is the most commercially important of the silicon nitrides as it maintains high-melting-point, largely chemically inert, hard, and has a high thermal stability []. The three types of silicon nitrides developed within IHP for the purpose of bio-compatibility evaluation are SiN, SiRN and SiON. *Theory on the physical aspects of the Nitrides and which of the three is expected to be robust to be included here. Possible points: Introduction to available Types, Chemical structure and expectations.*
If possible, a mention of clean room techniques to layer deposition could be made and how PECVD helps [2]. The SiN_x were deposited using plasma-enhanced chemical vapor deposition (PECVD) on 725 µm double-sides polished silicon wafers. PECVD enables low temperature and vacuum conditions for deposition, which is required for to avoid the impact on the technology stack. Additionally, the PECVD technology allows manipulation of the deposition parameters to circumvent stress arising due to dissimilar lattice constants of the silicon nitride and silicon. The set-up is an Applied Materials DxZ chamber installed on a Centura mainframe. The wafers contain of 1 nm native oxide layer prior to the SiN_x layer deposition. This, however, does not pose any adverse impacts for the subsequent steps. The fabrication process parameters and SiN_x layer information are summarized in the Table 1. The 13.56 MHz RF power is applied through capacitive coupling to the aforementioned chamber and combined with a fast working match. After a certain number of wafers, depending on the thickness and recipe, an in-situ clean is performed using a remote plasma source with NF_3 as fluorine source. *Some comments upon contributions of other factors in Table 1 could be included here. Comment upon possible sintering additives could be included here [13]. Useful to include formulae involved in Silicon nitride layer formation?*

$$E = mc^2. \tag{1}$$

$$E = mc^2. \tag{2}$$

The silicon nitride layer deposition rate is liner with respect to time. Thus, the thickness is controlled with great precision by controlling the time of deposition. The thickness is later verified through short-wavelength spectroscopic ellipsometry. Two parameters can be verified post production. These are surface roughness and layer thickness. The silicon nitride passivation layer roughness in IHP's technology is only of the order of a few nano-meter. A standard 49 point thickness measurement yields less than 1.3% standard deviation from the nominal value (400 nm) of the nitride thickness. This is also observed in other studies where in a combination of the atomic force microscope (AFM) and SEM measurements was evaluated. The surface roughness (RMS) obtained in an area of 2 µm x 2µm was 2.73 nm for the SiN, 1.92 nm for the SiON, and 3.12 nm for the SiRN [14]. Further, the extreme-ends (max-min) variation of 5% from the nominal value may be expected due to the fabrication tolerances across the complete wafer [15]. Evidently, the physical thickness evaluations can be sensitive to the location at which the measurement is done. Nevertheless, this variation is negligible within the sizes of the diced samples of the wafer.

III. TEST CATEGORIES

In order to acquire a wholesome view of the impact of the test fluids, two types of tests have been carried out in this study. These have been called physical and electrical characterization in this work. The physical characterization aims to characterize the physical degradation of the silicon nitride layer under a continuous long term impact of the test fluids. On the other hand, an electrical characterization is studied to isolate impacts on electrical performance under the long term exposure to the test fluids. Further details have been outlined below:

A. PHYSICAL CHARACTERIZATION: BLANKET WAFERS

The study of the physical removal rate of silicon nitride requires a blanket wafer. To server the purpose, a BEOL stack has been development with only Silicon base (725 µm double-sides polished silicon wafer) and a silicon nitride passivation layer above it. Thus, three different blanket wafers corresponding to three different silicon nitride have been developed and shown in Fig. 3 (left).

The wafer was diced into chips of 2 cms x 2 cms in dimensions. The wafer was diced into samples of such a size to aid experimentation and measurement process while creating enough samples to conduct a long term experiment. Additionally, these chips were laser scribed to create labels for a sample's unique identification. Three samples of SiN were glued on to a board laminate material as shown in Fig. 4. The laminate of FR4 material has been used here on account of its good thermal properties and moisture resistance [12]. The board dimension (in Fig. 4), of 7 cms x 2.6 cms allows the space required to glue the chips and also place the

TABLE 1. The process characteristics and the physical constants deployed for the three types of silicon nitride passivation layer deposition on the BEOL stack.

Type of silicon nitride	Chemical formula	Refractive index (n) (at 543.5nm)	Symbol used	Pressure (in Torr)	Temperature (in$^{\circ}C$)	Flow Rates (in sccm)					RF Power (in W)	Deposition rate (in nm/s)
						SiH_4	Ar	N_2O	NH_3	N_2		
Standard silicon nitride	Si_3N_4	2.03	SiN	4.7	400	360	-	-	145	4000	900	13
Silicon-rich silicon nitride	Si_3N_4	2.73	SiRN	4.7	400	500	2800	-	20	1200	650	10
Silicon-oxy nitride	Si_3N_4	1.634	SiON	5.0	400	107	-	90	-	4000	515	6

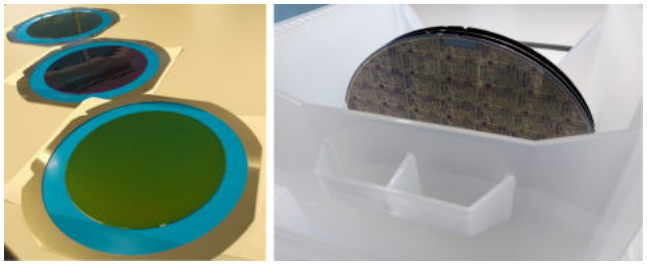

FIGURE 3. The three wafers which correspond to the three types of silicon nitrides for the physical thickness (left) and the dc electrical (right) evaluation.

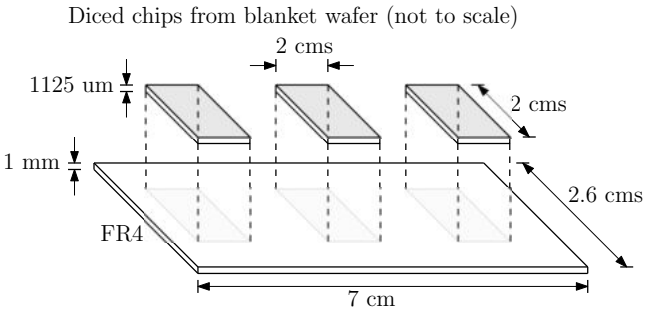

FIGURE 4. The diced chip dimensions from the blanket wafers and the board on which they were glued to perform the physical characterization in this study.

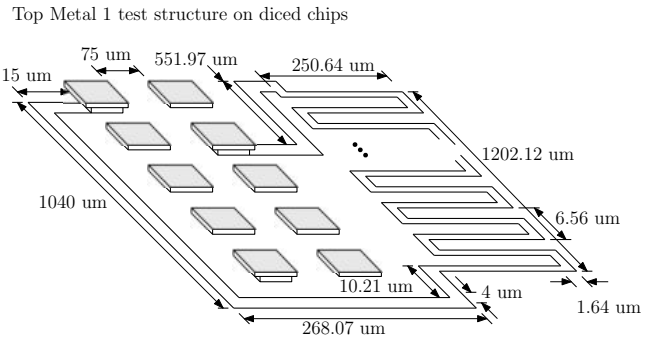

FIGURE 5. Top metal 1 electrical test structure in a serpentine configuration.

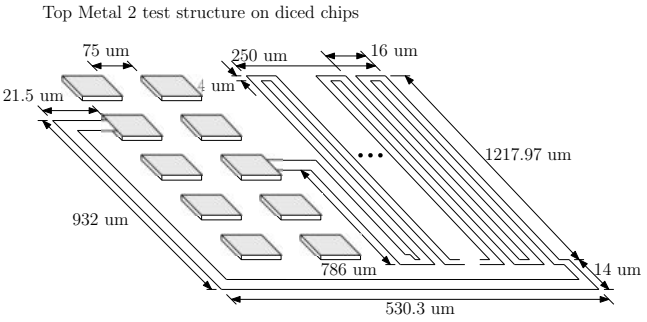

FIGURE 6. Top metal 2 electrical test structure in a serpentine configuration.

samples inside the test fluid container. Lastly, such a set of three samples glued on FR4 board were developed for SiON and SiRN.

B. ELECTRICAL CHARACTERIZATION: TECHNOLOGY TEST STRUCTURES

The key aspect of electrical characterization is the evaluation of DC resistance of the two upper most metal layers of the technology stack namely Top Metal 1 (TM1) and Top Metal 2 (TM2). These metal structures are made of Aluminum with sheet resistivity of . The focus here lies in verification of the intact-ness of these metals upon long term exposure to the test fluids. The passive microwave sensing unit along with other passive components is designed using these upper metal layers in the BEOL. Evidently, damages to this layer in terms of cracks, discontinuities, etc. on account of various possible factors such as adsorption etc. will be determined. This will facilitate the quantification of the prospective impact of biological fluids on the sensor. The TM1 test structure is of a horizontal serpentine configuration as shown in Fig. 5 and the TM2 test structure is a vertical serpentine configuration as shown in Fig. 6. The dimensions of these structures have also been indicated in their respective figures. The bond pads are 75 µm x 75 µm in size have a pitch of 50 µm. The serpentine configuration are standard structures typical in evaluation of process control for metal layer deposition [16], [17].

After etching the passivation layer consisting of Si_3N_4, the same lithographic mask was used for the Au lift-off process. Afterward 10 nm thick Ti and 100 nm thick Au layers were subsequently deposited by thermal evaporation. Au lift-off was performed in Acetone maintained at room temperature with sonication for 30 min, followed by rinsing with DI-water and drying with nitrogen gas.

IV. TEST FLUIDS

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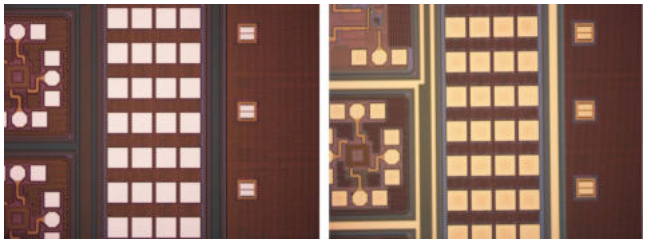

FIGURE 7. A layout portion presenting the pre-gold deposition (left) and the post-gold deposition (right) scenario on chips containing the test structures.

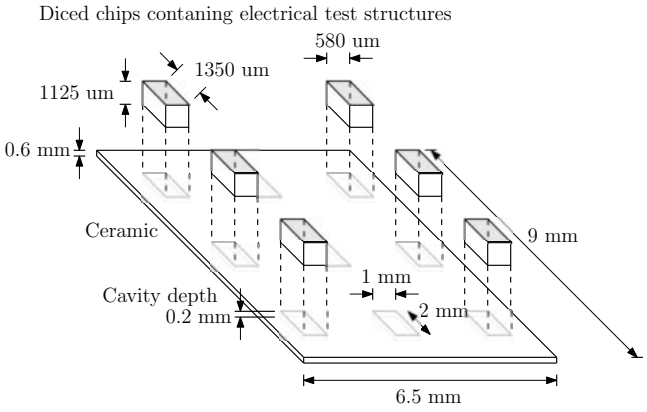

FIGURE 8. The diced chips out of the gold-deposited wafer and the board to which they were glued for the experimentation & dc electrical tests in this study.

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A. DI WATER

Human body comprises of 70% water and the impact of water alone (devoid of fluctuating ions) should be well quantified []. A laboratory grade de-ionized water is used here in order to isolate the impact of water (free from ions such as calcium, magnesium, iron, sodium, chlorides, sulphates, etc). More to be written. Suggestions welcome. More to be written. Suggestions welcome. More to be written. Suggestions welcome. More to be written. Suggestions welcome. More to be written. Suggestions welcome. More to be written. Suggestions welcome.

B. GLUCOSE SOLUTION

An industry grade glucose solution is used here. It contains D-glucose of a concentration of 200 g/L and a pH ranging between 6 to 8. This solution is applicable for mammalian cell culture. It is completely bio-degradable in waste water treatment and non-hazardous to environment. It is chemically stable during the observation period due to its durability of up to 12 months [18].

C. CELL CULTURE SOLUTION

The chips were sterilized in UV light for 30 min on all its sides and incubated in cell culture medium at $37°C$.

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V. SAMPLE PREPARATION AND EXPERIMENTATION

This experimentation demands a large scale manual measurements. This necessitates a well organized methodology in dealing with the samples for a few important and indispensable reasons. Firstly, the presented approach renders sample identification with utmost accuracy. Secondly, it allows for ease of conducting the experimentation. Lastly, it enables reliable, quick and the most convenient manner of conducting the measurements. These sample preparation techniques and the measurements conducted have been described below.

A. DESIGN FOR TESTABILITY APPROACH

In order to conduct the measurement with ease and also identify/organize the large set of samples, the chips are glued on a plastic board and identified using a chip which contains laser scribed naming on the passivation layer.

IHP's standard process qualification test-structures have been adapted for the evaluation of DC electrical resistance change. The resistance value of these test structures is recorded after the pre-defined intervals of time. Two chips of each of the three silicon nitrides have been considered. The chips contain gold covered bond pads so that they protect the metal layer at the pad opening from direct exposure to the tests fluids. The silicon chips are glued (using bio-compatible EPO-TEK 377) on a ceramic board as shown in Fig.9 (right) so as to facilitate probing through DC wedges and a probe station. The Fig. 9 shows a subset of the diced samples glued on a board which was developed for the physical (left) and the electrical (right) measurements. Every single board on 9 (left) contains three samples of the same type of silicon nitride. Similarly, each of the remaining two types of nitrides have been glued on distinct boards. The key reason for more than one sample (per experiment for each observation time frame) was to ensure accuracy through uniformity among multiple measurements. On the other hand, the 9 (right) presents one set created for each of the interval of observation, i.e. 1 week, 1 month, 2 months, 4 months and 6 months, exclusively for one of the test fluids. A total of three identical set of items shown in 9 (right) were created corresponding to three different test fluids. *Write about Fig 10, write about temperatures.*

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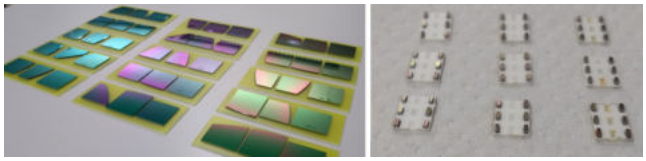

FIGURE 9. Diced samples of blanket wafers (left) & diced chips with electrical test structures (right) glued on the board using bio-compatible glue for the test.

FIGURE 10. The test samples placed in closed petri dish (left) with cell culture solution and incubated at $37^{\circ}C$ in the temperature controlled chamber (right).

B. MEASUREMENT TECHNIQUES, CALIBRATIONS AND RESULTS

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1) Physical characterization

The physical measurements has been conducted in a three fold manner. Firstly, all the diced and glued samples from the blanket wafer were measured using an ellipsometer prior to the commencement of the experimentation. This guaranteed the starting point thickness of each sample. Secondly, these samples were again measured at the end of each interval of observation. Lastly, a scanning electron microscope (SEM) measurement was carried out on the samples at the end of the 6 month period. The SEM measurements provide the highest accuracy of the physical thickness measurement among other techniques. These SEM measurements were used for a final re-assessment (requirement of a correction/calibration factor) on the accuracy of the ellipsometry measurements conducted on all samples. The apparatus used for ellipsometry is shown in Fig. 11. The ellipsometry is conducted at a wavelength of 543.5 nm for the measurement of the physical thickness of the passivation/nitride layer. The following two-layer model for measurement of the thickness of the nitride layer is defined based on the technology stack prepared for the physical thickness evaluation.

The SEM measurement results aat the end of the sixth month is shown in Fig. 13 through Fig. 15. The measured values of the nitride layer thickness at the end of each time interval is reported in Fig. 16. *Describe the results and account for the observation. Cite [10] and other works for temperature dependence in the results. Inputs from Marco, Christian for nitride dissolution dynamics*.

FIGURE 11. Physical thickness measurement of the silicon nitride passivation layer using the ellipsometer set-up at 534.5 nm wavelength of the incident light.

2) Electrical characterization

A four wire dc resistance measurement is performed here toward measuring the metal layer resistances [19]. The method is also know as Kelvin cross bridge resistance measurement which is a popular configuration deployed for resistance characterization in planar technologies []– []. In this measurement technique, current is forced into a pair of the probe terminals and the voltage is measured on the other pair of terminals. Subsequuently, the resistance is evaluated using Ohs law. However, this is accomplished by the multimeter and the computed resistance can be noted from the display. The Agilent 34411A multimeter is used here in the range 1 KΩ and in four-wire mode (Ω4W). *Write about residual (or contact) resistance calibration and BeCu wedges and cite IEEE papers.* The probe station and the dc wedges (used for probing the test structure) are shown in Fig. 12. Additionally, the set-up comprises of a multimeter to read the measured resistance out through a four-wire dc resistance measurement. A multi-contact wedge (MCW) with Beryllium-Copper (BeCu) dc needles (no capacitors/no resistors) and 50 um pitch is shown in Fig. 12 (left). The MCW part (#MCW-28-8911-A) has two BeCu dc needles (no capacitors/no resistors) built on a MCW-14 wedge body with a connector consisting of two rows of seven 0.025" square header pins on 0.1" centers []. The probe is for force (current) and sense (voltage) measurements. This needle is used to probe the gold covered contact pads (bond pads). The need for special BeCu needles arises due to a few important reasons. Firstly, a four-wire DC resistance measurement is to be conducted for the electrical characterization which requires reliable low resistance contacts. Secondly, the gold covered bond pads can be protected against damages due to probe touchdown. Lastly, its spring loaded tips provide reliable contacts even upon probing non-planar structures.

An initial calibration measurement was performed to identify/eliminate possible dc resistance offsets arising due to the probing needles. The "SHORT" calibration standard was used for this purpose. A gold-substrate short measurement as used in calibration techniques for on-wafer measurements has been used here. The measured dc resistances for each

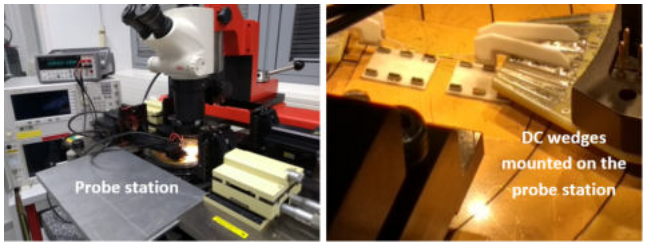

FIGURE 12. The probe station (left) using the BeCu probes (right) for the four wire dc resistance measurement to determine if the metal layers remain intact.

type of nitride at the end of various observation time frames is shown in Fig. 17 through Fig. 19. *Describe the results and account for the observation.*

VI. CONCLUSION

A six month long term bio-compatibility study on three types of silicon nitride passivation layer manufactured in IHP is studied here. The performance has been classified under a physical and an electrical characterization. Subsequently, simple and practical test concepts, characterization methods, and applicable measurement techniques has been discussed. This is also applicable for testing future materials.

Fully calibrated measurements have been conducted in the course of experimentation. For physical characterization, both ellipsometry and SEM was performed. For electrical measurements, an initial calibration measurement was performed to identify/eliminate possible dc resistance offsets arising due to the probing needles. A "SHORT" calibration standard was used for this purpose. The physical measurements reveal a degradation rate of 0.12 nm/day, 0.31 nm/day, and 0.12 nm/day for SiN, SiON, and SiRN, respectively, for the samples maintained at room temperature (between 22°C and 26°C) in DI-water. The degradation rates were 0.23 nm/day, 0.33 nm/day, and 0.01 nm/day for SiN, SiON, and SiRN, respectively, for the samples maintained at storage temperature (between 2°C and 5°C) in glucose solution. These rates were found to be 1.87 nm/day, 1.44 nm/day, and 3.2 nm/day for SiN, SiON, and SiRN, respectively, for samples incubated at $37°C$ in a cell-culture solution. On the other hand, it is observed in dc electrical resistance evaluation that metal lines remain intact post measurements period. This is observed in value of resistance in the experimentation time frame. It remains within the limits of measurement accuracy and fabrication tolerances. Based on several measurements across multiple chips, it is found that the metal lines in the silicon technology stack remain unaffected against common bodily fluids up to the reported period. *Comment on nitride dissolution dynamics (seek hints from Marco, Christain), comment on temperature dependence on degradation rate (see [10]) and comment on cytotoxicity (from Senectics)*

ACKNOWLEDGMENT

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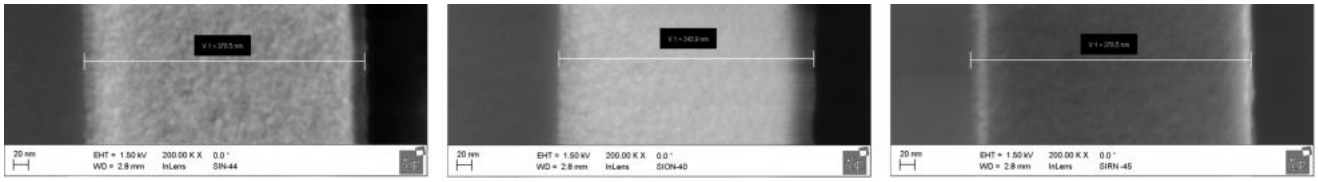

FIGURE 13. The SEM measurements performed on the samples post 6 months for SiN (left), SiON (centre), and the SiRN sample (right) placed in DI water.

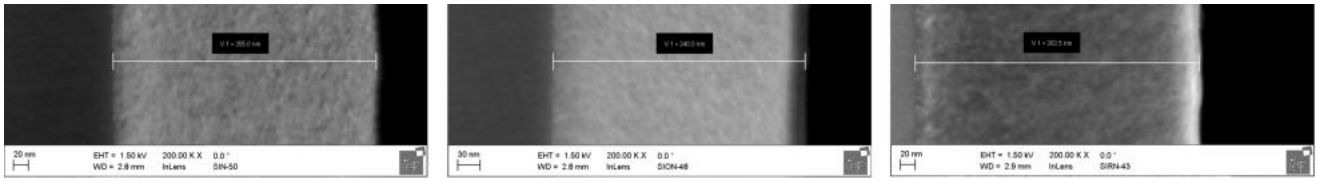

FIGURE 14. The SEM measurements done on the samples post 6 months for SiN (left), SiON (centre), and the SiRN sample (right) in the glucose solution.

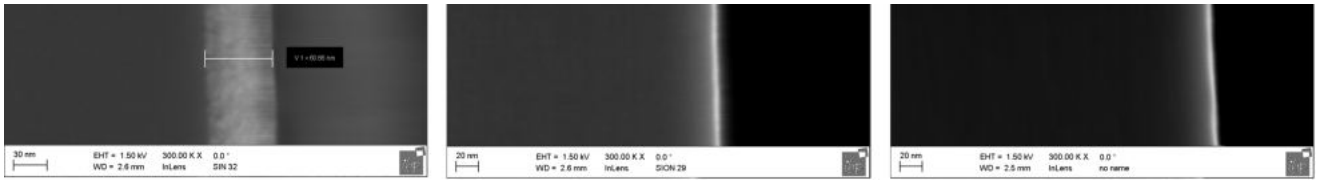

FIGURE 15. The SEM measurements performed post 6 months on the SiN (left), SiON (centre), and the SiRN sample (right) in the cell-culture solution.

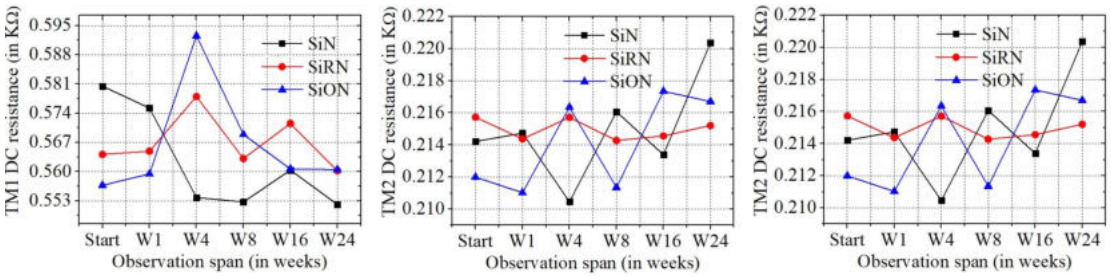

FIGURE 16. Under preparation: Bar graphs containing SiN, SiON, and SiRN with thickness on Y-axis and time on X-axis. One graph for each test fluid.

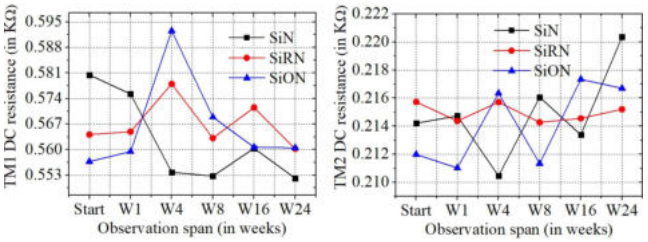

FIGURE 17. DC electrical resistances in SiN, SiON, and SiRN sample in the DI water. This graph will be replaced with bar graph.

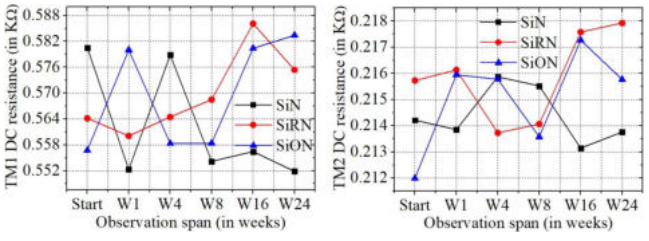

FIGURE 18. DC electrical resistances in SiN, SiON, and SiRN sample in glucose solution. This graph will be replaced with bar graph.

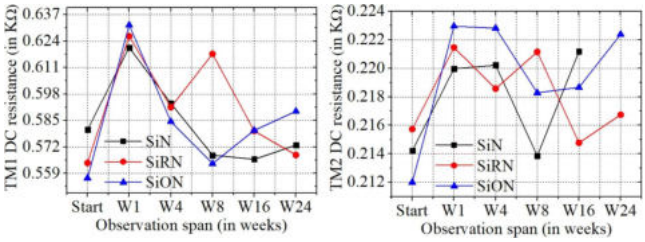

FIGURE 19. DC electrical resistances in SiN, SiON, and SiRN sample in cell-culture solution. This graph will be replaced with bar graph.