# Characterization on pH-ISFET Poly Bisphenol Hydrophobic Encapsulation Material

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Nowadays the microsensor is playing the big role our modern life. The Ion Sensitive Field Effect Transistor or pH-ISFET sensor is one the said microsensor. The use of microsensors for in-field monitoring of environmental parameters is gaining interest due to their advantages over conventional sensors. The fabrication of pH-ISFET sensor are using standard transistor process i.e through semiconductor process. The IC's type pH sensor contributes further advantages such as small size and robustness. The differences between pH-ISFET sensors with other electronic Integrated Circuit (IC's) is the encapsulation/package use to protect the membrane/sensing area. The pH-ISFET sensor required an opening at sensing area or membrane area. Thus, it can't follow the standard IC's packaging using the Joint Electron Device Engineering Council (JEDEC) encapsulation which only offers fully enclosure. As of today there is no standard package available for pH-ISFET sensor. In this research work, the robustness test for pH ISFET encapsulation using Poly Bisphenol hydrophobic materials have been discussed. The experiment such as leakage test, high temperature test, fatigue test and bending test show the characteristics of encapsulation and pH-ISFET device. From the result, Poly Bisphenol encapsulation material able to protect the sensitive area and give minimum leakage current below 1 uA. The temperature test shows the encapsulation work well with Malaysia climate either at lowest temperature 10°C and highest temperature 40°C. For fatigue and bending, the testing able to verify the maximum limit can be absorb by the pH ISFET package before failed.

Keywords: pH-ISFET, Encapsulation, pH Sensor, leakage test, structural integrity test

## I. INTRODUCTION

The field of microfabricated chemical sensors research based on Ion-sensitive Field Effect Transistors (ISFETs) and related sensors has been dynamic since Bergveld introduced the ISFET concept in 1970 (Bergveld P. 1970). From then many scientific papers as well as some interesting reviews concerning such semiconductor based sensors have been published (Bergveld P. 2003). The initial interest came from

the advantages of ISFET over conventional ion-selective electrode (ISE) such as small size and solid-state nature, mass fabrication, short response time and low output impedance. Other features such as the integration of compensation and data processing circuits in the same chip offered also new perspectives for these sensors (Bergveld P. 2003). The main problem that causes the difficulty of building pH-ISFET sensors that can be used continuously is due to the encapsulation of the sensor itself (Jimenez C. et al 2010).

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Although ISFET history began in the seventies, commercialization of probes with ISFETs only started in the nineties. This fact can be explained due to a few practical limitations related to the inherent properties of the devices, such as drift, temperature and light sensitivity, and technological limitations such as encapsulation and the need for a stable miniaturized reference electrode (Jimenez C. et al 2006). In this experiment, the encapsulation using polybisphenol material has been identified to protect the sensitive circuit parts that need to be closed from the liquid during the experiment (Mamat H. et al 2012). Several progress on the encapsulation of the pH-ISFET have been reported to address few issues and become pints of interest.

The encapsulation for PH-ISFET sensor is quite tricky because it cannot use the standard JEDEC package due to sensing membrane opening area requirement, thus to developed such protection for PH-ISFET sensor required extensive study on the suitable materials (Mamat H. et al 2012). The difficulties with pH-Isfet sensor are the standard operation require the sensor to dip or submerged into the solution. The measurement can be in minute, hours or continuous for years. So the robust, reliable and cheaper material is presented in this paper. The pH ISFET chip was design and fabricated at Mimos wafer fabrication lab. Then the ISFET chip being test for standard parametric test. After grinding and dicing process, the ISFET chip encapsulated with few epoxy materials. The ISFET place on the PCB together with Temperature sensor and connector. To avoid leakage from other area, the encapsulation apply to all PCB area in front and backside of PCB excluding the sensor membrane area as per Figure 1. To minimize the hazard, pH operational testing using buffer solution to replace actual acidic and alkaline liquid. The encapsulation package was design to operate in solution, water and fertilizer without degrading the sensor performance and able to minimize the gate current leakage. In order to further assess the performance of the pH-ISFET sensor, this paper focuses on the characterization of the I-V properties. This includes the effect of leakage current, type of encapsulation materials and the response toward different pH environment.

#### II. METHODOLOGY

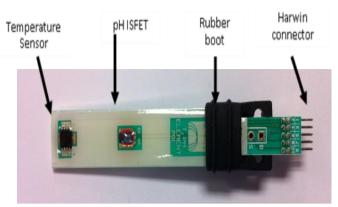


Figure 1. Actual PH-ISFET sensor

The experiments below are to confirm the encapsulation material is good for operation in water, able to provide low leakage current and maximum strength. The duration of the test on the encapsulation material is approximately 140 days. There are 4 type of encapsulation materials were test to find the best encapsulation material with lowest leakage current such as Poly Bisphenol, PTA, Silicon and Asacast. Leakage testing was carried out to determine the operational level of the pH sensor package. The voltage range used is from oV to 5V and the current supplied is 100mA. Measurements for leakage current Ig is the current supplied at the gate (reference electrode) with reference to bulk. Basically if the Ig gate current is higher than 1uA then the package is not good because it will cause the use of high battery power and will cause poor pH sensitivity or low sensor sensitivity. Depend on usage, some pH sensor is intended for permanent use in the field, so the use of the battery power should be at the minimum. The optimum voltage for this silicon pH sensor is designed at 3.3V. The ideal leakage current at Ig is oA and it is impossible to achieve the number due to pH sensor operating condition is in the water (Figure 2). The pH-ISFET sensor and reference electrode are placed in a pH 4 buffer solution, while pH 7 and pH 10 solutions for sensitivity tests. The leakage testing was done in black box to avoid light effect. The good pH-ISFET sensor can be determined with low leakage current under 1 uA and high sensitivity toward different pH solution. There were 2 separate testing done i.e different encapsulation materials and testing with 40 units of sample.

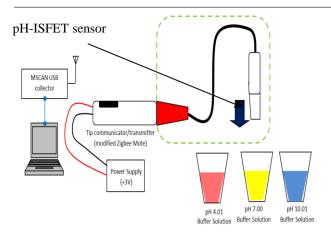


Figure 2. Leakage Test simulation

## III. RESULTS AND DISCUSSION

Figure 3 shows the result of leakage testing on different encapsulation materials (a) Poly Bisphenol, (b) PTA, (c) Silicon and (d) Asacast. The result proves that the Poly Bisphenol material with leakage current less than 1 uA is able to perform well in solution compare to other materials. This is the most important test for the pH-ISFET sensor package. If the leakage current is high more than 1 uA definitely the sensitivity of pH will failed. Summary of leakage current for each material are shown in Table 1.

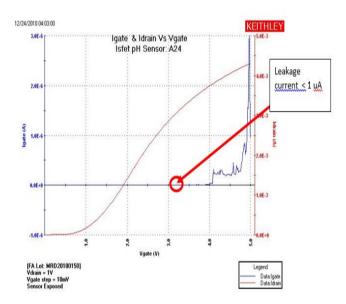


Figure 3a. Poly Bisphenol Leakage Current

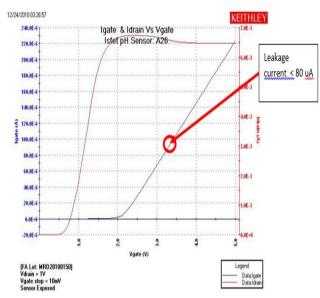


Figure 3b. Asacast 2:1 encapsulation Leakage Current

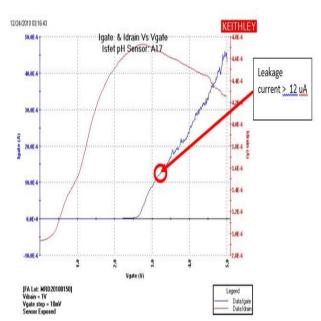


Figure 3c. PTA encapsulation Leakage Current

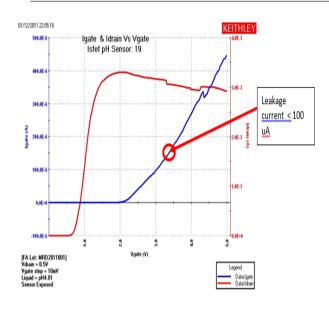


Figure 3d. Silicone encapsulation Leakage Current

Table 1 shows that only Bisphenol encapsulated materials has passed the leakage current of low than < 1uA. For Asacast, PTA and silicone encapsulated materials have indicated the value of 70uA, 10uA and 100uA, respectively. The observation from all the hydrophobic encapsulation material used suggest that only Poly Bisphenol material able to operate in a solution or water with a low leaking current of less than 1 uA.

Table 1: Comparison different encapsulation material

No	Encapsulation Material	Leakage Current (Ig)
1	Bisphenol	< 1UA
2	Asacast	70uA
3	PTA	10uA
4	Silicone	100UA

In summary, pH-ISFET-sensor is a device that progressively improved over time and it's getting smaller, from micro to nanometer range. Currently micro packaging becomes an issue for the commercialization devices. In conventional packaging of integrated circuits IC contributes to one-third of the cost of manufacturing. PH-ISFET sensor packaging has stringent requirements due to the microstructure of the fragile and they are deemed the most expensive in the production of devices. As reported by Jimenez C. 2010, if the

current leakage test exceeds 10nA (pH Isfet package rejected) and the pH-ISFET using polymer based encapsulation work continuously in the field is 7 months to a year. From the experiment above, one can confirm leakage current, effect to extreme temperature can be summarized that the pH sensor with the encapsulation of Poly Bisphenol have the ability to protect the pH-ISFET electric circuit from the leaking or short circuit while operating in the liquid. With leakage current *Ig* measured in nano ampere make the pH Isfet sensor operating well and able to save power during long operation in field. The sample tested at temperatures ranging from 10°C to 40°C, pH-ISFET sensor with Poly Bisphenol package works well without any failure.

#### IV. CONCLUSIONS

From observation on ability of the package to operate in water or a solution, it can confirm that the Poly Bisphenol is a suitable material for the pH-ISFET sensor. Comparison with other materials such as PTA, Silicon and Asacast shows only Poly Bisphenol A package are capable of operating in a solution or water with leakage current Ig less than 1 uA. This will allow the pH detector to operate with optimum battery power and operate longer in the field.

## V. ACKNOWLEDGMENTS

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#### VI. REFERENCES

Al Hardan, NH et al 2016, High sensitivity pH sensor based on porous silicon (psi) extended gate field-effect transistor, *MDPI Journals Sensors*, vol. 16, no. 6, pp. 839.

Bergveld, P 2003, Thirty years of ISFETOLOGY—what happened in the past 30 years and what may happen in the next 30 years. *Sensors and Actuators B: Chemical* vol. 88, no. 1, pp. 1-20.

Bergveld, P 2003, ISFET, Theory And Practice, Proceedings of IEEE Sensor Conference Toronto, pp. 1-26.

Deshpande AW 2005, Study and characterization of plastic encapsulated packages for MEMS, MSc Thesis, Worcester Polytechnic Institute.

Huang X et al 2015, A dual-mode large arrayed CMOS ISFET sensor for accurate and high throughput pH sensor in biomedical diagnosis, *IEEE Transactions* on *Biomedical Engineering*, vol. 62, no. 9.

Jimenez C. et al, ISFET based microsensors for environmental monitoring, *Sensors*, vol. 10, no. 1, pp. 61-83; <a href="https://doi.org/10.3390/s100100061">https://doi.org/10.3390/s100100061</a>.

Jimenez, C et al 2006, ISFET Based Sensors: fundamentals and Applications. In *Encyclopedia of Sensors*; Grimes, C.A., Dickey, E.C., Pishko, M.V., Eds.; American Scientific Publishers: Stevenson Ranch, CA, USA, 2006; vol. 5, pp. 151-196.

Kenneth S et al, 2016, Deep-sea DuraFET: a pressure tolerant pH sensor designed for global sensor networks, *Anal. Chem.*, vol. 88, no. 6, pp. 3249-3256.

Mamat H. et al 2012, *Characterisation of moisture* 

sensor using FR4 materials, Proceedings of ICME Jun 2012.

Mamat H. et al 2013, The characterization of pH sensor over temperature, *Proceedings of the 2nd International Conference on Mechanical, Electronics and Mechatronics Engineering (ICMEME'2013)*.

Mamat H. et al 2017, The study of bending and fatigue testing of pH ISFET encapsulation using hydrophobic material epoxy, *Proceedings of ICOS International Conference on Computer Science 2017, Miri Sarawak.* 

Mamat H. et al, *High energy implant effect to resist thickness*, /www.scientific.net/AMR.230-232.718
Nov 2011.

Manjarres. C et al 2016, Chemical sensor network for pH monitoring, *Journal of Applied Research and Technology*, vol. 14, no. 1, pp. 1-8.

Nakata, S, Arie, T, Akita, S & Takei, K 2017, Wearable, flexible, and multifunctional healthcare device with an ISFET chemical sensor for simultaneous sweat pH and skin temperature monitoring, ACS Sens. vol. 2, no. 3, pp. 443-448.

Norzin, MS et al 2018, ph sensing characteristics of silicon nitride as as sensing membrane based ISFET sensor for artificial kidney, *Proceedings of 2018 IEEE International Conference on Semiconductor Electronics*, *ICSE 2018*, pp. 124-127.

Parizi, K et al, ISFET pH sensitivity: counter-ions play a key role, 02 February 2017, Scientific Reports, vol. 7, pp. 41305.

Qin Y et al 2015, Microfabricated electrochemical ph and free chlorine sensors for water quality monitoring: recent advances and research challenges, RSC Advances, no. 85.

Sabah FA et al 2016, Sensitivity of CuS membrane pH sensor with and without MOSFET, The Journal of The Minerals, Metals & Materials Society, vol. 69, no. 7, pp 1134–1142.

Sabah FA et al 2016, Sensitivity of CuS membrane pH sensor with and without MOSFET, The Journal of The Minerals, Metals & Materials Society, vol. 69, no. 7, pp 1134–1142.

Sabah, FA et al 2016, Sensitivity of CuS and CuS/ITO EGFETs implemented as pH sensors, Applied Physics A, vol 122, pp. 839.

Sabah, FA et al 2016, Sensitivity of CuS and CuS/ITO EGFETs implemented as pH sensors, Applied Physics A, vol 122, pp. 839.