Heterogeneous Cubic Multi-Dimensional Integrated Circuit for Water and Food Security in Fish Farming Ponds

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Keywords: Integrated circuit; packaging; silicon; Internet of Things, water and food security.

Among major food production sectors, world aquaculture shows the highest growth rate, providing more than 50% of the global seafood market. However, water pollution in fish farming ponds is regarded as the leading cause of fish death and financial losses in the market. Here, an IoT system based on a cubic multi-dimensional integration of circuit (MD-IC) is demonstrated for water and food security application in fish farming ponds. Both faces of the silicon substrate are used for thin-film based device fabrication. The devices are interconnected via through-silicon-vias, resulting in bi-facial CMOS-compatible electronics system. The demonstrated cubic MD-IC is a complete, small and lightweight system that can be easily deployed by farmers with no need for specialists. The system integrates on its outer sides simultaneous air and water quality monitoring devices (temperature, electrical conductivity, ammonia and pH sensors), solar cells for energy-harvesting and antenna for real-time data-transfer, while data-management circuitry and a solid-state battery are integrated on its

internal faces. Microfluidic cooling technology is used for thermal management in the MD-IC. Finally, a bio-friendly polymeric encapsulation is used to waterproof the embedded electronics, improve the mechanical robustness and allow the system to float on the surface of the water.

Fish is an excellent source of protein, vitamins, and minerals with uncountable benefits to the human health; as a result, the yearly per capita consumption of seafood has increased over time from 20 pounds in 1961 to more than 45 pounds today.^[1-2] With the fast-increasing demand for fish protein, aquaculture grew to compensate for the reduced fish abundance due to the overfishing in the wild fisheries.^[3] Currently, aquaculture provides more than 50% of the globally consumed fish, and during the last 10 years, the yearly global returns for fish farming grew from \$60 billion to more than \$231.6 billion. The corresponding total production is more than 80 million tonnes of fish.^[1] Nevertheless, water pollution in the ponds is considered as the major cause of financial losses in the market especially in the rapidly urbanizing areas. For instance, the aquaculture in China suffered from a \$1 billion loss in 2010 due to water pollution and consequent fish death.^[4]

Pollution in ponds water can be due to biological, chemical and atmospheric reasons, all of which are inter-related, leading to the reduction in fish yield. For instance, the increased nutrient concentration in the ponds due to fertilizers, pesticides or increased urbanization and resultant industrial waste with high amounts of phosphates and nitrogen result in a phytoplankton and algal heavy bloom.^[5] When the algae is decomposing, it excretes toxic levels of ammonia and consumes the oxygen in the water resulting in fish asphyxia. Human agricultural activities can also increase the heavy metals and salts concentration in ponds to reach toxic levels. Moreover, the quality of the air surrounding the pond greatly contributes to the water quality where high levels of sulfur oxides and nitrogen oxides generated from combustion of fossil fuels and transportation can either dissolve or deposit on the surface of the

water, leading to the deterioration of the water quality.^[6-7] Hence, preventive measures can be taken by the farmers to protect the fishes when toxic levels of air pollutants are detected. Other key factors that affect the fish production are the sunlight exposure and the temperature of the water. Sunlight is critical for the regulation of the water temperature – a too high temperature reduces the density of oxygen while a too low temperature reduces the fish metabolism – and for the production of the natural food in the ponds – plankton and photo-synthesis.^[8-9] Therefore, real-time monitoring of water quality is essential for protecting and further fostering the aquaculture industry growth.

Commercial systems for observing water quality do exist, however, they can monitor one characteristic at a time such as conductivity meters, ammonia sensors, oxygen meters, etc.^[10] However, this is considered as an expensive and time consuming solution for the farmer. Moreover, the commercially available and previously reported IoT multi-sensory systems for water monitoring are mostly bulky, expensive and require installation by experts.^[10-13] Here, we report a prototype of a small and lightweight system using a multi-dimensional integration of circuit (MD-IC) with the application of water security in fish farming ponds. The MD-IC technology merges the benefits of the two most successful integration and packaging technologies: system-on-chip (SoC) and system-in-package (SiP) (**Supplementary Figures S1, S2, Supplementary Table S1**).^[14] In fact, the MD-IC exceeds the restricted functionalities of the SoC technology and the bulky dimensions and inefficient area consumption in the SiP approach. The cubic system comprises thin-film based sensors on its outer sides for simultaneous air and water quality monitoring while data management circuitry and a solid state battery are integrated on its internal faces, all of which are interconnected using through-silicon-vias (TSVs) and side interlocks. The integrated system is then encapsulated using a polymeric material to waterproof the embedded microcontroller and battery, improve the mechanical robustness of the system and enable it to float on the surface of the water for solar energy harvesting and simple wireless data transfer. A Bluetooth low energy unit (BLE) and light

emitting diodes (LEDs) are employed to provide the farmer with fast and real-time water monitoring results. Finally, silicon solar cells are embedded in the MD-IC for achieving a complete, compact-packaged, lightweight and high performance multi-sensory system for water and food security application.

The development of the cubic MD-IC is realized using a CMOS based processes. The different sides are fabricated using CO₂ laser patterning followed by silicon deep reactive ion etching (DRIE) Bosch process etching (**Figure 1a**). Every MD-IC side includes multiple metal-coated interlocks to electrically interconnect the neighboring sides in a puzzle-like fashion. In addition, TSVs connect the top and bottom faces (inner and outer faces) of each MD-IC side (**Figures 1b-d**). The MD-IC dimensions could be modified based on the species inhabiting the pond where larger cubes would be deployed when large fishes are being farmed to avoid their mistaken consumption (**Figure 1b**). Electronic devices that necessitate active materials other than Si can be integrated on the unpolished of the substrate, while data management devices could be integrated on the polished Si face with lower defects density (**Figures 1c-d**). Different water quality sensors are fabricated on a single outer side of the MD-IC that will be in direct contact with the water. The pH level sensor is based on an interdigitated electrodes structure with a monolayer of graphene (**Supplementary Figure S3**). The temperature detector uses the resistive temperature detection technology, the salinity sensor is based on interdigitated electrodes and the ammonia sensor uses 3D graphene foam (**Supplementary Figure S4**) with high surface area-to-volume ratio as the sensing medium. On the opposite side (top side of the MD-IC), a monocrystalline silicon solar cell with high efficiency (19%) is integrated to harvest energy and charge the battery of the system. It is worth to note that the MD-IC technology enables the integration of heterogeneous materials, thus, a Ge substrate could be used for the fabrication of higher efficiency III-V solar cells^[15] instead of the used silicon substrate. On the rest of the outer sides of the system, a copper ring antenna is fabricated in addition to air quality sensors. The air quality sensors enable the detection of potential contamination of the water by

the air pollutants. On the inner sides, a microcontroller IC (16 MHz, $2.6 \times 2.5 \times 0.28 \text{ mm}^3$) and solid-state battery are integrated (**Figure 1c**, **Supplementary Table S2**). Finally, all the interconnects are e-beam evaporated with 500-nm of silver. It is worth to note that the MD-IC technology enables an improved yield at a reduced cost because it allows the development and testing of the different sides independently prior to merging them into a compact-packaged system.

The proposed MD-IC is designed to float on the surface of the water for several reasons: 1) to monitor the quality of water and air simultaneously, 2) to harvest solar energy and 3) to enable simple and real-time wireless transfer of data. A bio-friendly polymer polydimethylsiloxane (PDMS) is therefore used to completely encapsulate the MD-IC resulting in a system that is less dense than water due to the inner trapped air, as a result, it floats on the surface of the water (**Figures 2a-d**). Moreover, the MD-IC includes a single face with embedded water quality sensors, thus, whenever an external force (e.g. contact with fishes) causes the rotation of the cubic system, it must always flip back such that the desired side is in contact with water. The remaining outer sides can include thin-film based air quality sensors^[16-18] in addition to the energy harvesting devices and antenna. To achieve this, the PDMS around the side with water sensors (bottom side) is made thicker and asymmetric which would shift the center of gravity of the MD-IC downward and achieve stable equilibrium when the desired side is immersed in water (**Figures 2a-b**, **Supplementary Figure S5**, **Supplementary Video S1**). As a result, the farmer can easily deploy the cubic system by simply throwing it into the pond water, the MD-IC will then autonomously adjust its orientation owing to the PDMS encapsulation design. The PDMS was next patterned using CO₂ laser to expose the active area of the different sensors to the outer environment, and thus to be in direct contact with the water/air (**Figure 1d**). In addition, the encapsulation of the MD-IC enhances the mechanical robustness of the system by absorbing any mechanical shock (**Supplementary Video S2**) and protects the embedded electronics such as the solar cell and the microcontroller due to its water

resistant characteristic. It is worth to mention that, according to the FAO,^[1] the optimum depth of fish farming ponds is around 2 m and that the required oxygen level in the water is usually maintained by using different aerators such as waterfalls or spitters in the ponds. These aerators contribute to the mixing of the water and reducing the inhomogeneity in water properties throughout the pond. Therefore, even though the MD-IC system is floating on the surface of the water, it can provide reliable measurements of the water properties.

To ratify the reliable operation of the proposed system, the different embedded sensors and electronics are characterized, in addition, multiple reliability tests are conducted including un-biased highly accelerated stress test (μ HAST), high temperature storage test (HTS) and environmental tests. The temperature sensor is characterized by measuring the patterned metal resistance at different temperatures. A sensitivity of $0.021 \text{ } \Omega/^{\circ}\text{C}$ and a temperature coefficient of resistance (TCR) of $1.34 \times 10^{-3}/^{\circ}\text{C}$ are obtained (**Figure 3a**).^[19] The same measurements are conducted when probing the sensor from the back contacts (through the via) and from the interlocks on an adjacent side; upward shifts of $0.2 \text{ } \Omega$ and $11.1 \text{ } \Omega$ are observed, respectively, which is due to the added resistance of the metal contacts, however almost the same sensitivity is preserved (**Figure 3a, Supplementary Figure S4**). The pH level sensor, based on a graphene monolayer (**Supplementary Figure S3**), shows a reduced resistance in solutions with higher pH level (**Figure 3b**). The pH sensors are tested when exposed to solutions with pH going from 1.5 to 8.5 (acid to alkaline) and vice versa, from 8.5 to 1.5 (alkaline to acid). A small hysteresis is observed as expected (Figure 2). In fact, the sensing mechanism in graphene is known to be nonfaradaic (capacitive), which means that charges do not transfer across the graphene/solution interfacial boundary (no redox reaction).^[20] Rather, the development of an adlayer occurs that change the interfacial surface and result in a “doping effect” (Figure S4). Owing to the nonfaradaic nature where no exchange of charges occurs, charges are highly reversible (reversible adsorption),^[21] as a result, when going from acidic to alkaline solutions and vice versa, a negligible hysteresis is expected.^[22] Moreover, adsorbed OH^- ions (in alkaline solution)

are found to induce a larger conductivity in graphene than H_3O^+ ions (in acidic solution)^[23] which is consistent with the measured smaller resistance of graphene at higher pH levels (**Figure 3b**).

The employed salinity sensor is based on conductivity measurements in order to measure the concentration of different undesirable salt ions in the fish farming ponds. Traditionally, a salinity sensor is based on a simple design with two electrodes separated by a specific distance. In the demonstrated sensor, interdigitated electrodes are used instead of parallel electrodes. In fact, interdigitated electrodes enable a smaller cell factor which is needed for measuring low levels of conductivity. The results show that the conductivity of the saline solution increases with higher concentration of salt and with temperature. In fact, at higher concentrations of salt, more ions (anions and cations) are introduced into the solution which contribute to the conductivity of current. In the experiment, table salt with different concentrations is diluted in DI water, thus, $\text{NaCl} \rightarrow \text{Na}^+ + \text{Cl}^-$. While at higher temperatures, the ions in the solution (anions and cations) gain more energy and move faster resulting in an increase in the measured conductivity (**Figure 3c**).^[24] Therefore, the measurement of both salinity and temperature of the water is required to extract the correct salinity level.

The ammonia concentration is measured using 3D graphene foam-based sensors. Graphene generally shows p-type characteristic under ambient conditions due to the electron extracting nature of oxygen and water functionalities.^[25] This means that electrons in graphene are minority carriers while holes are majority ones. NH_3 molecules have strong electron-donor properties and are physically adsorbed on the graphene surface. Once the NH_3 molecules are adsorbed, the electron charge is partially transmitted to the graphene surface, and the holes in graphene are trapped in the electrostatic gravity of the electrons.^[26-27] As a result, the mobility of the hole carriers is reduced causing an increase in the resistance of the sensor.^[28] It is important to note here that the exposure of graphene to ammonia at ambient conditions for long durations does not functionalize graphene with nitrogen which could otherwise affect the

reliability of the device, instead functionalization requires exposure to high energy and chemically active nitrogen^[29] such as annealing the graphene at high temperatures ($\geq 300\text{ }^{\circ}\text{C}$) in NH_3 ambient and for long durations (several hours)^[30] or exposure to ammonia plasma.^[31] Moreover, the employed graphene has a porous 3D foam structure with thickness of around $50\text{ }\mu\text{m}$ (**Figure 3d**) with an RMS roughness of around $4\text{ }\mu\text{m}$ (Figure 3d, inset). It has been reported that defective graphene shows a higher adsorption energy and charge transfer than its pristine graphene counterpart.^[32] The used 3D foam graphene is defective as confirmed by the broad Raman peaks^[33] (Figure S5) which enhances the response characteristic of the sensor. Two designs are studied for the ammonia sensor, square and spiral (Figure 3e, inset). The higher surface area-to-volume ratio in the 3D graphene with spiral structure enables a higher efficiency in absorbing the ammonia, resulting in a higher sensitivity (**Figure 3e**) and faster response compared with the square design (**Figure 3f**).

The recovery behavior of the graphene foam is studied by exposing the sensor to a solution of ammonium hydroxide with a concentration of 7% NH_3 for around 15 min then immediately dipping it in deionized (DI) water. A cycling test up to 4 cycles is conducted on the sample with spiral design (**Figure 3g**). The results show that once the sensor detects ammonia and NH_3 are adsorbed on the surface of the graphene, the resistance is increased until it reaches a plateau, allowing a reliable reading of the sensor. Then, when the sensor is immediately placed in DI water, its resistance does not return back to its initial value. Instead, it shows a shift that becomes larger as the number of NH_3 exposure cycles is increased, suggesting that residual ammonia molecules did not desorb completely from the sensing device. Moreover, the results show that the recovery characteristic is slow ($\sim 10\text{ min}$) which is in agreement with reported recovery behavior of ammonia gas sensors based on graphene.^[34-35] This can be due to the large thickness and porosity of the 3D graphene, where some NH_3 residuals are left on the surface after the tested ammonia solution is replaced with deionized water. To solve this issue, heat is introduced during the desorption process ($80\text{ }^{\circ}\text{C}$) to assist the

complete recovery of the sensing element. In fact, it has been previously confirmed that introducing heat/annealing during the recovery period enhances the desorption characteristic of the adsorbates from the graphene.^[34-35] The results show that the resistance of the graphene sensor reduces until it reaches its initial state (**Figure 3h**). The desorption process takes around 10 min. Even when the sample is placed back in room temperature DI water, the sensor continues to show the same initial resistance which confirms that the NH₃ molecules are completely desorbed from the surface of the graphene.

Therefore, in the proposed MD-IC, a heater will be fabricated on the backside (inner side) of the silicon face with the ammonia sensor. A heater is designed and fabricated by sputtering 1 μm of copper (inset of Figure 3h). When biased with 3 V, the heater gets heated up to 80 °C within seconds (Supplementary Video S3, left side). It is also worth to mention that the microcontroller provides an output of 3.3 V on its output pins which is suitable for triggering the fabricated heater (Table S2). Finally, it is important to note that 80 °C is considered as a low temperature which should not cause any damage to any of the embedded sensors or devices. In summary, the following scenario is suggested in the final MD-IC system which can be achieved by programming the embedded microcontroller with the corresponding algorithm: when the system detects an unacceptable ammonia level in the fish farming pond, a message is sent to the farmer. Next, the heater will be triggered by the microcontroller in order to heat up the ammonia sensor and reset it to its initial resistance value. The process which consists of a) heating, b) recovery and b) cooling down is expected to take around 15-20 min in total since the heating process is very fast (less than 20 s) while the recovery process takes around 10 min. In addition, the silicon face with water quality sensors is in direct contact with cold water which assists the heat dissipation and cooling down process. Therefore, during this “RESET” period, the output from the rest of the water quality sensors can be either neglected since they will be affected by the heater, or their output can be corrected taking into consideration the temperature provided by the temperature sensor. Finally, once the temperature sensor detects the last-

recorded temperature value before triggering the heater (which means that the heat is fully dissipated and the system is cooled down), the ammonia sensor is considered as reset and all the water quality sensors can be back to normal operation.

The embedded monocrystalline silicon solar cell is finally characterized using a solar simulator under 1 Sun AM 1.5G, the measured efficiency of the solar cells is ~19%, with a current density (J_{sc}) of ~39 mA/cm², an open circuit voltage (V_{oc}) of ~0.64 V and a fill factor of ~75.8 (**Figure 3i**).^[36] As a result, a complete multi-sensory system with energy harvesting/storage devices, data management circuitry and data transmission is obtained. The detected data would be synchronously sent to a phone wirelessly. In fact, the output of the various sensors would be connected to the input pins of the microcontroller, which would read and process the data based on the programmed algorithm (i.e. equations, correction factors, sensors sensitivities, etc.). As a proof-of-concept, the temperature and salinity sensors data are synergistically sent via the Bluetooth Low Energy unit to a mobile phone (Supplementary Video S4).

The MD-IC passed preliminary component-level reliability tests including the μ HAST at 200°C and 300°C at 60% RH humidity for 48 hours (**Figure 4a**), HTS at 200°C and 300°C for 48 hours (**Figures 4a-b**) and environmental tests (immersion in DI and saline waters) for 48 hours (**Figure 4a**). In fact, a DC test is conducted on the microcontroller to measure the current draw during standby mode after performing the reliability tests. The measured current consumption is similar to those obtained in reference ICs (**Figures 4a-b**) which confirms that there are no defects in the multilayered PDMS causing any leakage of the fluids into the system. Moreover, although the maximum allowed storage temperature of the microcontroller IC is 150°C, Figure 4b shows that the current consumption during standby mode (leakage current) did not increase after the exposure of the embedded microcontroller IC to elevated temperatures, which suggests that the system could sustain harsh environment conditions. In addition, the poor thermal conductivity of the PDMS considerably reduces the exchange of

thermal energy across the encapsulation constituents ^[37]. This is confirmed using infrared thermal imaging (**Figures 4c-d, Supplementary Video S3**). As a result, if one side of the MD-IC gets heated up (e.g. side with encapsulated antenna gets heated up due to sun exposure), the adjacent sides will experience a strongly attenuated effect due to the low thermal conductivity of PDMS, which would otherwise result in faulty readings from the sensors.

A system-level reliability test is also performed on the cubic standalone system where the embedded LED shines light as the system temperature rises above a threshold value of 30°C. This is achieved by programming the microcontroller input and output pins connected to the temperature sensor and LED, respectively (**Figure 4e**). It is worth to note that in this test, the devices are integrated on various sides and both faces of the cubic system to include the effect of the side interconnects and TSVs. The MD-IC passed the system-level reliability test where the microcontroller current output received by the LED is similar to reference values (Figure 4e). Finally, microfluidics based cooling is used to dissipate heat generated within the electronic devices such as the solar cell. In fact, silicon solar cell's efficiency degrades by 0.5% for each 1°C rise in temperature. Since the MD-IC is in constant contact with water, a separate reservoir of cold water is not needed. Micro-channels in the PDMS encapsulation are fabricated using CO₂ laser ablation which are then capped with another flat PDMS layer (**Supplementary Figure S6**). An electric thin-film based heater is fabricated to emulate a hot spot and test the efficiency of the microfluidic channels in dissipating heat; the results show a ~9°C reduction in temperature compared to the hot spot with no embedded microfluidic channels in its PDMS encapsulation layer (**Figure 4f**).

In conclusion, a complete, small and lightweight IoT multi-sensory system for simultaneous air and water-quality monitoring based on the MD-IC technology is demonstrated. The system integrates thin-film components on semiconductor substrates which enables the integration and packaging of high density and high performance heterogeneous devices. In the demonstrated cubic MD-IC, water quality sensors are embedded on a single

outer face which is in constant contact with water while the remaining outer faces include air quality sensors in addition to solar cells for energy harvesting and an antenna for wireless data-transfer. On the inner faces, data management circuitry and solid-state battery are embedded. To enable the system to float on the surface of the water with the same orientation, an asymmetric PDMS encapsulation is used. The encapsulation assists in waterproofing the electronic devices such as microcontroller and solar cells, in addition, it improves the mechanical resilience of the system. As a result, the farmer can easily deploy the MD-IC system by throwing it into the water which will autonomously adjust its orientation owing to the PDMS encapsulation design. Finally, microfluidic channels in the PDMS encapsulation layer are used to dissipate the heat generated within the system. The system then provides real-time monitoring results to the farmer. The demonstrated prototype shows the potential of the MD-IC in IoT applications and can be further enhanced by integrating higher-efficiency III-V solar cells and additional water and air quality sensors.

Experimental Section

Development of the MD-IC: Initially, a photoresist is spin-coated on a 4 inch Si (100) wafer. Next, a polymeric kapton tape is used as a hard mask. A CO₂ lasing system with a power of 23 W and height of 1 mm is then employed to design the various sides of the cubic system. The exposed areas are then completely removed via Bosch process etching in an Oxford deep reactive ion etching (DRIE) tool with sulfur hexafluoride (SF₆) and carbon fluoride (C₄F₈). The obtained isolated sides are immersed in acetone to lift off the kapton, and the interconnects are e-beam evaporated with 500-nm silver.

Development of the integrated devices: Initially, SiO₂ with 300-nm thickness is grown using an Oxford plasma enhanced chemical vapor deposition (PECVD) system. Next, 10 nm/180 nm Ti/Au is sputtered. The temperature detectors are patterned by photolithography and RIE-based etching. The graphene monolayer is synthesized on copper foil using a chemical

vapor deposition system and is then transferred onto the silicon substrate by first etching the copper using FeCl_3 for 24 hours. The graphene is next immersed in deionized water and scooped on the metallic electrodes of the pH detector. The monolayer is then left to dry at ambient conditions for 24 hours. The salinity sensor is fabricated by sputtering 200-nm platinum and patterning interdigitated electrodes using photolithography. The ammonia sensor is based on 3D graphene foam which is induced by CO_2 laser ablating a polyimide sheet and transferred onto PDMS. The ammonia sensor is a two terminal device, the resistance change is extracted by applying a 0.6 V and reading the output current. The antenna is fabricated by first sputtering 200-nm copper on a ring-patterned photoresist using photolithography followed by lift off. The photovoltaic device uses monocrystalline Si and tin coated copper interdigitated back electrodes. On the internal faces of the cubic system, a microcontroller chip (pre-fabricated bare-die) is integrated in addition to a solid-state battery (micro-lithium-ion based).

Fabrication and characterization of the heater: A heater is designed and fabricated by sputtering 1 μm of copper (inset of Figure 3h). When biased with 3 V, the heater gets heated up to 80 °C within seconds (Video S3, left side). The silicon sample with the heater is placed on a metallic substrate (22 °C) which has a higher thermal conductivity than water in order to ensure that the heater will be able to heat up the sample to at least 80 °C even when the silicon face is in direct contact with water (instead of the metal). The temperature variation is measured using an infrared microscope.

Encapsulation: Polydimethylsiloxane (PDMS) is arranged by mixing 1:10 PDMS curing agent:silicone elastomer followed by degassing in a vacuum mixer for 4 minutes at a 1.1 kPa pressure with a speed of 700 rpm to get rid of any trapped air. The cubic system is next covered with the encapsulant and cured at a temperature of 60°C for 24 hrs. The MD-IC was encapsulated with 6 layers of PDMS to make sure that there are no defects in the encapsulation that could lead to the leakage of water from the fish farming pond into the MD-IC. To make an asymmetric PDMS encapsulation, the cubic system is placed in a Poly(methyl methacrylate)

(PMMA) based mold with depth of 6 mm. 6 layer of PDMS are then poured onto the cubic system and cured at 60 °C for 24 hrs. Once cured, the bottom PDMS with 6 mm thickness is cut in an asymmetric way as shown in Figure 2b (wider PDMS on the left side) such that the weight is distributed asymmetrically. This ensures that the system will always flip such that the heaviest side (with thickest PDMS) will be at the bottom and in contact with water. This means that when the farmer throws the MD-IC into the water or if the system receives a mechanical shock by a fish which may cause its rotation, the MD-IC will autonomously adjust its orientation such that the face with water quality sensors is always in contact with water while the face with solar cell (opposite side) is always the top side of the MD-IC (as shown in Video 1).

MD-IC design rule: The face with heaviest PDMS (asymmetrical PDMS) is the bottom face to be in direct contact with water therefore it includes the water quality sensors. On the inner side of the bottom face, a heater will be embedded in order to reset the ammonia sensor. On the opposite side (top side), the solar cell will be embedded in order to capture sunlight most efficiently. PDMS microfluidic cooling will be used to cool down the solar cells and reduce the heat effect, where the efficiency drops by 0.4-0.5% for every 1 °C rise in temperature. On the inner face of the top side, the battery will be included to store the energy harvested using the solar cells. The remaining sides are symmetrical (vertical sides) and are used for embedding an antenna in addition to air quality sensors. It should be noted that optical sensors could be embedded on the outer vertical sides in order to detect the deposition of any solid pollutant on the surface of the water such as plastic bag, cans, etc. On the inner faces, the microcontroller (microprocessor and memory) would be embedded.

Microfluidics channel fabrication: CO₂ laser is used to pattern microchannels in PDMS for fluid flow to achieve microfluidics based cooling. The used CO₂ laser is based on the Universal Laser Systems PLS6.75 system with 75 W maximum power. The tool can achieve a

resolution of 200 μm . The used recipe consists of a speed of 2 mm/s, power of 0.75 W and a z-height of 1 mm. The obtained width of the PDMS micro-channels is 250 μm .

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

Acknowledgements

M.M.H. conceptualized the idea and directed the study. N.E.-A. led the project, fabricated the system, characterized the devices, and evaluated the results. R.A. and A.A. helped in the fabrication and characterization of the ammonia sensors. R.S. and Y.A. assisted in the fabrication and characterization of the salinity sensor. W.B assisted in the integration of the microfluidic channels. S.M.K, S.F.S and N.Q. helped in the characterization of the microcontroller and the Bluetooth Low Energy unit transmitted data. The authors acknowledge support from the King Abdullah University of Science and Technology (KAUST) Office of Sponsored Research (OSR) under Award No. Sensor Innovation Initiative OSR-2015-Sensors-2707 and KAUST-KFUPM Special Initiative OSR-2016-KKI-2880.

Received: ((will be filled in by the editorial staff))

Revised: ((will be filled in by the editorial staff))

Published online: ((will be filled in by the editorial staff))

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Figures with captions

Figure 1. Development of the cubic system. a) Illustration of the development and packaging processes of the cubic MD-IC based on laser patterning and DRIE. b) Cubic MD-ICs with different dimensions showing solar cells, thin-film based antenna and sensors on its outer sides. c) Internal faces of the system integrating a high-performance microcontroller and a solid-state battery. d) An outer side of the MD-IC showing the PDMS encapsulation of the TSVs to avoid the insertion of water into the embedded electronics while the sensor area is exposed to the outer environment (either water or air).

Figure 2. Polymeric encapsulation to realize a floating MD-IC. a) Fully encapsulated MD-IC with PDMS. b) The PDMS encapsulation is designed with a thicker and asymmetric base to enable the floating of the system and the autonomous immersion of a single distinct side with water quality sensors. c) MD-IC floating in water with water sensors immersed in the fluid while other sides are exposed to the air. d) Encapsulated MD-IC with solar cells on the top side, water quality sensors on the bottom side (opposite to the solar cells) and air quality sensors on the rest of the outer sides. A microcontroller and a solid state battery are integrated on the internal faces.

Figure 3. Sensors and solar cells characterization. a) Temperature sensitivity of the sensors when probed from the contacts on top, bottom and adjacent sides in the MD-IC. b) pH sensor performance with water, coffee and vinegar solutions. c) Salinity sensor response to different salinity concentrations at different temperatures. d) Surface profilometer data showing a $\sim 50 \mu\text{m}$ thickness of the 3D graphene foam. Inset shows the RMS surface roughness of the graphene of around $\sim 4 \mu\text{m}$. e) Ammonia sensors response to different aqueous ammonia concentrations, two different sensor designs are studied, square and spiral. Scale bar is 6 mm. f) Response time characteristic of the ammonia sensors when exposed to 7% ammonia concentration at ambient conditions. g) Recovery behavior of the ammonia sensor with spiral design when exposed to 7% NH_3 under ambient conditions. h) Recovery behavior of the ammonia sensor with spiral design when first exposed to 7% NH_3 under ambient conditions then heated at 80 $^{\circ}\text{C}$. i) J-V and P-V characteristics of the monocrystalline silicon solar cells.

Figure 4. Component and system-level reliability testing results. a) Output current from the microcontroller in standby mode under different test conditions. b) Output current from the embedded microcontroller chip which is interconnected with the LED after HTS. c) Thermal image of a Si MD-IC and a PDMS encapsulated MD-IC when heated at 90°C for 2 hours. d) Temperature change of the MD-ICs vs. time when heated at 90°C for 2 hours. e) Current output from the microcontroller during the system reliability test. f) Microfluidic channels (inset) embedded in the MD-IC show a 9°C reduction in the maximum temperature.

In this work, we demonstrate a prototype of an IoT multi-sensory system for water quality monitoring in fish farming ponds based on a multi-dimensional integration of circuit (MD-IC) technology. The MD-IC integrates thin-film components and provides a complete, multi-sensory, and lightweight system. The system can also monitor the air quality surrounding the ponds for undertaking preventive measures.

Keyword: Multi-dimensional integrated circuits, packaging, through-silicon-vias, water and air quality monitoring, food and water security

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Cubic shaped standalone multi-dimensional integrated circuit for water and air quality monitoring application

Supporting Information

Heterogeneous Cubic Multi-Dimensional Integrated Circuit for Water and Food Security in Fish Farming Ponds

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Sensors Design Optimization

Salinity sensor

The salinity sensor operation is based on the measurement of the electrical conductivity of the water between metallic electrodes. The electrical conductivity is defined by the multiplication of the conductance by the cell factor. The cell factor depends on the geometry of the sensor, where smaller values are required to sense and measure smaller concentrations of salinity. For fish farming ponds application where the acceptable range of water salinity is between 20 to 150 $\mu\text{S}/\text{cm}$,^[1] a cell factor of 0.1-0.5 cm^{-1} is required. The fabricated salinity sensor is based on the resistance measurement between platinum interdigitated electrodes with a spacing of 1 mm. The interdigitated electrodes design is chosen as it improves the sensitivity of the device and enabled a lower cell factor for the same spacing when compared with the 2-electrodes design. The obtained cell factor is extracted by normalizing the measured conductance of ultrapure water to the measured conductivity using a commercial salinity sensor. Platinum is used as the sensor material due to its high-resistance to corrosion characteristic.^[2]

Ammonia sensor

3D graphene foam is used as the sensing medium for ammonia. The graphene foam is induced by laser ablation of a 120- μm -thick polyimide sheet using a CO_2 laser with a power of 2.85 W and height of 3 mm. The obtained graphene foam is then transferred onto PDMS by spin coating the PDMS polymer on it and curing it at 60°C for 2 hours. The PDMS is then stripped off of the polyimide sheet

to obtain the ammonia sensors on PDMS substrate. Both square and spiral designs are studied in terms of sensitivity and response time. It is found that the spiral structure with higher surface area-to-volume ratio enables the sensor to be more efficient in absorbing the ammonia, resulting in a faster response and higher sensitivity.

Differences Between SoC, SiP and MD-IC

The presented MD-IC technology does not combine between two existing technologies (SoC and SiP), instead it combines between the benefits of both technologies. Here, we would like to explain the pros and cons of each of these two technologies and show how our proposed technology combines the benefits of both approaches.

As a matter of fact, Moore's law drives the SoC technology. This technology was preferred by multiple IC manufacturers for several years because it is able to achieve systems with high performance and a small form factor, low power consumption in addition to high density integration.^[3-8] The key enablers for this approach mainly include the advances in lithography technique, enhanced materials combinations in addition to the introduction of larger wafers. Even though SoC shows several benefits, however, it is restricted to a single wafer process. As a result, systems with restricted functionalities or a poorer performance of multiple electronic devices are obtained. Additionally, the SoC technology faces several challenges such as 1) extended design times as a result of the complex integration schemes which require multiple lithography masks, 2) high fabrication charges, 3) designs verifications and 4) Intellectual Property (IP) issues.

SiP on the other hand is advantageous over SoC by being capable of integrating heterogeneous chips with multiple functionalities (such as Bluetooth, antennas, imaging and so on) into a single package to achieve a full system. Moreover, SiP which consists of integrating different electronic devices on a PCB, has advantages over SoC with regard to 1) less complex designs and therefore a lower number of masks would be needed, 2) faster implementation, 3) larger memories integration, 4) Intellectual Property reuse, and so on. Nevertheless, SiP is considered as a packaging technology rather than an integration technology, in addition, its performance is generally restricted to the CMOS

characteristics which might not be ideal for specific devices, in addition to being very bulky compared to SoC.

In this work, we applied a novel integration and packaging technique using a multi-dimensional integration of thin-film elements to merge the benefits of SoC and SiP technologies. The MD-IC delivers a comprehensive system with multi-functionalities and a small form factor instead of the bulky printed-circuit-board employed in SiP technology. Although both SoC and SiP systems may include three dimensionally integrated devices, however, MD-IC overcomes 1) the restricted functionality in SoC and 2) the bulky dimensions and inefficient area consumption in SiP.

The key characteristics of the MD-IC that make it a more cost-effective solution are the following:

- It uses both faces of the substrate for the devices integration.
- It uses single-side polished substrates instead of double-side polished substrates. Devices that are based on different active materials than the substrate would be integrated on the unpolished side.
- High-performance electronics can be fabricated on the different sides separately (no need for additional expensive lithography masks to integrate all of the devices on the same substrate as in SoC).
- Every single side can be based on a different substrate material (i.e. Si, Ge, GaAs, etc.) to boost the performance of the overall integrated circuit.
- Every side of the MD-IC system can be tested separately before merging them into a complete system which further increases the yield.
- Finally, previously reported IoT systems for water quality in fisheries are too bulky and require installation by experts which increases the total cost compared to the MD-IC which can be deployed easily by the farmers.

Mechanics of the floating MD-IC

The floating mechanism of the demonstrated MD-IC design under mechanical perturbations can be explained using the governing fluid mechanics. The basic principal to determine whether a body will

float or sink over the fluid depends on the opposite forces acting on it. The MD-IC will be subjected to two forces called the gravitational force (downward force due to its weight) and up-thrust force or buoyancy force exerted by the liquid. If the gravitational force (G) is smaller than the buoyancy force (B), the body would float. In fact, Archimedes' principal claims that any immersed or floating body is lifted up by a force that is equivalent to the weight of the displaced water. The line of action of force of buoyancy is called center of buoyancy. Therefore, the body will keep floating in a stable state if B exceeds or becomes equal to G of the body.

When the body oscillates or experiences the mechanical perturbations, it alters the total volume of the displaced water i.e. changes the original B position to a new position called B_1 , however, G remains at the same position, as shown in Supplementary Figure S5. The intersection of the line passing through the original G and B and the line passing through new B_1 (orthogonally to the water line) is called the metacenter height (M). We may dictate the response of the floating body by looking at the value of M i.e. whether it will return to its original position or not. The larger the M value is (M is positive), the more it will be stable and would readily return to its initial position due to returning moment.

Thus, in the demonstrated design, in order to make the MD-IC stable during the floating (i.e. stabilize its position), the G of the system is shifted from the centroid of body by making the base heavier as compared to the upper body using the thicker PDMS encapsulation. As a result, the G of the MD-IC is shifted downward (i.e. G is below B), thus making the MD-IC always stable. Therefore, when the body is subjected to mechanical perturbations, the heavy base creates a restoring moment that returns the MD-IC to its original position. Additionally, we utilize the off-center location of G by providing the asymmetry to ensure that the system will always assume the preferred position in the water even when the system is rotated by 180° as confirmed in Supplementary Video S4.

Table S1. Summary of the differences between SoC, SiP and MD-IC.

	SoC	SiP	MD-IC
Heterogeneous integration	Low	High	High
Density	High	Low	High
Yield	Low	High	High
Form Factor	High	Low	Medium
Development time	Long	Short	Medium
Multi-functionality	Low	High	High

Table S2. Specifications of the embedded electronic components in the MD-IC.

	Microcontroller	Battery	Solar Cell
Number	MSP430G2252	CBC050	Maxeon C60
Type	Bare die	Bare die	Si solar cell
Size	2.6×2.5×0.28 mm ³	5.7×6.1× 0.2 mm ³	Variable
Specs	Clock frequency up to 16 MHz	Output 4.1 V, 50 μ Ah	Output 3.2 W, efficiency 21%

Figure S1. Schematic comparison between different integration and packaging technologies. 3D-SoC, 3D-SiP and the proposed MD-IC.

Figure S2. 1 Area foot print of MD-IC vs. 3D SiP package integration of the proposed components.

Figure S3. Raman spectrum of the graphene monolayer measured at 532 nm excitation on SiO₂/Si substrate. G band peak at ~ 1587 cm⁻¹, 2D band peak at ~ 2701 cm⁻¹ and $I_{2D}/I_G = 2.42$ which confirms that the graphene is monolayer.

Figure S4. MD-IC optical image showing the different contacts that are used to probe the temperature sensor fabricated on the top silicon face of the MD-IC.

Figure S5. Center of gravity and center of buoyancy of the encapsulated MD-IC explaining the mechanics of the floating system with a preferred direction

Figure S6. Raman spectrum of the 3D graphene foam showing broad peaks which suggest that some layers are defective.

Figure S7. MD-IC with a heater emulating a hot spot and PDMS-based microfluidic channels for heat dissipation.

Video S1. Immersion test on the encapsulated MD-IC. Due to the asymmetric PDMS base on the MD-IC, the cubic system always drops in water such that the side with PDMS base is in always contact with water for water quality monitoring.

Video S2. Mechanical robustness of the MD-IC. Video showing a shock test performed on the MD-IC when dropped from a height of 15 m.

Video S3. Thermal test on the MD-IC using microfluidic cooling technology. The MD-IC side shows an improved heat dissipation when microfluidic channels are integrated in the system. The two samples include a thin-film heater to emulate a hot spot in the system.

Video S4. Data transmission test via Bluetooth Low Energy unit to a mobile phone from the integrated sensors on the MD-IC when the water is heated up and when table salt is introduced.

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