

Nanomaterials Based Sensors for Corrosion Detection and Monitoring for Proactive Aircraft Management

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ABSTRACT

Corrosion of metallic aircraft structures is a major concern due to its impact on aircraft availability and maintenance costs. The transition of aerospace industry to the usage of chromate-free paint systems, new materials and structures, such as metal/composite hybrid structures, and more severe in-service conditions due to climate changes, makes the conventional “find and fix” inadequate to offer reliability and agility required for fleet management. There has been a shift within aircraft maintenance community towards condition- or usage- based aircraft management. The challenges with proactive management of corrosion are the complex mechanisms of corrosion, new coating systems, lack of reliability to detect corrosion, and to predict corrosion growth or severity in conjunction with crack growth. This paper discusses sensor technologies for corrosion detection and monitoring, in particular corrosion in locations with limited access. There is a need for early detection and continuous monitoring systems of corrosion, in particular in the locations that are difficult for visual inspection. To that end, sensors of high reliability, such as integrated environmental monitoring and corrosion by-product sensing, could offer agility and efficiency required for proactive corrosion management. In this paper, integrated physical, chemical and environmental sensing for high fidelity corrosion detection and monitoring on aircraft structures are discussed. Nanomaterial-based sensors has been found to be a viable technology to detect corrosion environments and by-products, including exceptional sensitivity and selectivity for multi-point real-time detection and monitoring of corrosive environments and chemical by-product. The class of sensors also offer great benefits of low power to operate, compact size, and ease of manufacturing. This paper highlights some of the notable corrosion sensors which implement nanomaterials to detect sensor responses to specific environmental factors or corrosion by-products. As demonstrated in this paper, graphene-derived reduced graphene oxide and hydrogenated graphene nanomaterial coatings are implemented within a C60-based n-type organic thin film transistor architecture, capable of detecting chloride ions at controlled temperature and surface wetness, with an extraordinary sensitivity of 4 ppm. The ongoing work of using boron nitride nanotubes (BNNTs) modified dielectric shows that BNNTs is a suitable material for the next-generation OTFT corrosion sensors due to their superior dielectric permittivity and environmental durability. The approach to producing a monolithic sensor node design is the key to a robust and reliable corrosion detection and monitoring system, where in addition to temperature and humidity, chemicals associated with corrosion, such as chloride, pH, metal ions, hydrogen ions and others, are added through modification of nanomaterials to those specific analytes.

Keywords: Proactive aircraft corrosion management, Corrosion sensors, Nanomaterials

1.0 INTRODUCTION

Corrosion is the electrochemical deterioration of a metal because of its chemical reaction with a surrounding environment [1]. Metallic materials in aircraft structures, in particular aluminium alloys and steels, are susceptible to time-dependent effects of corrosion, which is often a slow process of material deterioration. Specific metallic materials are selected to fulfill aircraft design requirements based primarily on the performance attributes they exhibit, such as weight, stiffness, strength, electrical properties etc., rather than their ability to resist the onset of corrosion [2][3]. The impact of corrosion on aircraft structural integrity came to light in the 1988 incidence on a Boeing 737 in service at Aloha Airlines. In that incidence, the separation of a fuselage upper part was attributed to disbonding, corrosion and cracking problem [4]. Prior to the Aloha incidence, corrosion had been accepted as a normal operating condition and no corrective actions had been undertaken. Since then it has been acknowledged that corrosion could affect aircraft structural integrity by reducing material thickness and mechanical strength, or by accelerating fatigue crack development and stress corrosion cracking for the safety-of-flight structural components, ultimately jeopardizing the overall structural integrity.

After the mandate of active corrosion prevention and control programs by the Federal Aviation Administration and military technical orders, catastrophic incidence and excessive downtime for structural repairs directly associated with corrosion have been largely avoided. Nevertheless, corrosion has become one of the most costly maintenance and aircraft availability issues. Currently, corrosion prevention measures such as using corrosion-prohibiting primers and protective coating are routinely implemented. Based on annual reports of Logistics Management Institute regarding estimated impact of corrosion, the corrosion costs for all aviation and missiles of the United States Department of Defense were US \$8.97 billion in the fiscal year of 2017, which increased to US \$10.18 billion in financial year FY2018 [5][6]. For the US Air Force, the cost of corrosion in the FY2018 was \$5.67 billion, accounting for 23.6% of total maintenance costs. Furthermore, corrosion also caused 89,653 Non-available Days (NAD), about 14.1% of total NAD for the Air Force aviation and missiles. Another estimate of the corrosion costs conducted using a metric called the cost per day of availability (C/DA) indicates an estimated 4.8 day loss of availability for every weapon system of the U.S. Department of Defense (DoD), rendering over \$20.6 billion or 20.7% of the total DoD maintenance expenditures for FY2016 [7]. The high corrosion management costs are not an issue for US Air Force only; the operators of all fixed- and rotary-wing aircraft with metallic structures and composite/metal hybrid structures face similar challenges.

In spite of huge costs incurred by the current prevention and control programs, the current corrosion management that is typically based on “find and fix” philosophy does not prevent unexpected incidences of severe corrosion. The schedule-based maintenance strategy lacks the agility to address specific flight envelopes and in-service environments of a tail number. It is also insufficient to address the situations where corrosion mechanisms are either not fully understood, e.g., the usage of a new non-chromate paint system, or difficult to anticipate, such as sealant degradation leading to galvanic corrosion. There has been effort towards Condition Based Maintenance (CBM), or a holistic cradle-to-grave “damage and corrosion tolerance” management approach to reduce costs and ensure aircraft safety [8]. These maintenance methodologies are probabilistic-based prognostics and health management approach that utilize statistical tools such as failure modes and effects criticality analysis for reliability-centred maintenance [9]. The ability to detect and to monitor corrosion will allow for a more efficient and cost-effective corrosion management strategy by means of synchronization of corrosion removal with the maintenance plan to minimize maintenance costs and loss of availability. These “anticipate and manage” approaches require real time data collected by sensors on the ground and on-board to provide information regarding corrosion events, aircraft in-service environment, corrosion state and corrosion rates for corrosion prognostics, in order to mitigate safety risks, improve asset management and reduce costs of aircraft maintenance. Such data could offer the fleet-specific information based on aircraft usage for the development of a new maintenance strategy to ensure aircraft structural integrity and reduce maintenance cost. This paper discusses the current states of corrosion sensing technologies and highlights the potential of emerging

technology of nanomaterials-based sensors for early and continued corrosion monitoring for proactive corrosion management.

2.0 CORROSION MATRIX OF PHYSICAL, CHEMICAL AND ENVIRONMENTAL FACTORS

Corrosion comes in different forms. The forms of corrosion are driven by various mechanisms, alone or synergistically and their effects of the corrosion forms on structural performance vary significantly. The complexity and diversity of corrosion makes it challenging in corrosion monitoring and management. An understanding of different corrosion forms, along with their mechanisms, associated environment and impact on aircraft structures is key to designing and implementing appropriate corrosion detection and measurement technologies.

Table 1 provides a list of the influencing factors for a range of corrosion forms on aircraft. Environmental factors that have been found to influence corrosion include atmospheric pollutants, pH level, salt, temperature, humidity and sand etc [10]. The physical parameters of corrosion pertain to physical damage to protective coatings, paints, and sealants, and mass loss in the corroded metallic structure, and cracks of metallic structures that may be associated with corrosion. While information of corrosion environment is useful to predict corrosion rates, direct detection of corrosion through physical indications when accessible, or chemical by-products including metal ions and hydrogen is critical to increase detection reliability. Figure 2 shows the three categories of corrosion matrix - physical, chemical and environmental factors - for corrosion detection [11].



Figure 1. Corrosion matrix of physical, chemical, and environmental factors [11].

The type of solution formed on the surfaces of metals, along with environmental conditions such as temperature, relative humidity, time of wetness directly influences electrical conductivity and the rate of corrosion [12]. Common electrolytes, especially salt regularly seen in marine and coastal environments, and their concentration are key drivers of corrosion and must be monitored for corrosion management. When salt infiltrates an area of wetness, not only does it promote electrical conductivity, but the chloride ions also prevent the regeneration of protective oxide layers on certain metals, thus inhibiting the metal from using any sort of protection against corrosion. In the instances in which crevices exist and water becomes entrapped in a stagnant state, water evaporation can create a higher concentration of metal ions, creating an electrical potential that can promote the onset of corrosion. High concentration of corrosive

pollutants and metal ions may also exist on the surface of a structure, especially under conditions of alternate wetting and drying. As a result, the time of wetness (TOW), which refers to the period of time during which the atmospheric conditions are favorable for the formation of a surface layer of moisture on a metal or alloy (or the duration of the electrochemical corrosion processes), is a key indicator of the corrosion growth rate [14]. Temperature is also critical to the corrosion process. It does not just promote corrosion itself; it can also change the form of corrosion attack. For example, when temperature exceeds a certain threshold, corrosion can change from uniform to pitting corrosion, a more severe form of corrosion. Detailed and science-based models are required to determine the historical corrosion environment that use meteorological and pollutant deposition data, along with environmental sensor data, and a suite of models that synergistically use this data to determine the historical corrosion environment. The corrosion environment can then be used with surface science and electrochemical models to predict the occurrence and severity of corrosion [15].

Corrosion by-products such as hydrogen and metal ions, produced as a result of chemical reaction of metals and electrolyte is a direct measurement of corrosion, which could enhance early detection of corrosion, as well as detection reliability and robustness. Reliable corrosion monitoring requires comprehensive understanding of corrosion processes and a panoply of sensing mechanisms that offer both direct detection of corrosion events, and continuous measurement of environmental parameters pertaining to corrosion. An integrated sensing system can offer early on, reliable and real-time information about the structural health of aircrafts by providing information about the form, location and rate of corrosion. An integrated sensing system that detects specific corrosion by-products associated with the metals being used on aircrafts, along with corrosion environment, could be the key to enhancing reliability and robustness of aircraft corrosion detection and monitoring, contributing to a robust management strategy to reduce corrosion management costs and increase aircraft availability.

3.0 SUMMARY OF CORROSION SENSORS

Table 2 shows common sensor types/methodologies that have been explored for corrosion detection [17-27]. The commonly used methods for aircraft inspection include visual inspection while other methods such as ultrasound, eddy current are used for local inspection to detect surface indication, mass loss, oxidation, and structural defects such as strain deformation, erosion, cracks and coating degradation. Structural Health Monitoring such as acoustic emission and fibre optics sensors have also been investigated for corrosion detection [13]. Visual inspection often fails to detect early stages of corrosion damage, corrosion in locations with limited access such as crevice corrosion in confined and hidden locations, small cracks and types of corrosions that start within the bulk of the material, such as pitting corrosion. Monitoring of relative humidity, air temperature and other environmental parameters offer valuable information to correlate environmental corrosivity to the location, time and rates of a corrosion event. However, environmental sensors do not detect corrosion directly. Therefore, effective aircraft corrosion management will need a robust and reliable way for early detection and continuous monitoring of both environmental factors specific to corrosion, particularly pollutants such as acidity and salinity, and corrosion by-products, namely, pH and metal ions [16].

Amon those sensors, some are intended for field applications or onboard for corrosion management. The data obtained on the ground may not accurately reflect the real conditions of corrosion events and progression during flights, but they are useful to develop tools for corrosion management with integration of the ground environmental data. On-board sensors installed on aircraft structures often collect data on a continuous basis. There are several aircraft structural health monitoring (SHM) sensors currently in use, such as eddy current sensor Meandering Winding Magnetometer for mass loss and cracks developed by JENTEK [28], and corrosion environmental electrical resistance sensor such as AirCorr O designed by the French Institute of Corrosion [29], and magnetic flux leakage sensor for corrosion induced mass loss measurements developed by Battelle [30]. Current corrosion environmental sensors developed by Luna and Senserion [21-25] monitor for air temperature, relative humidity, and capacitance.

Table 1: Corrosion forms, definition, and influencing factors [10]

Form of Corrosion	Definitions	When to Consider	Influencing Factors
Uniform (general)	A form of corrosion that occurs uniformly over the entire exposed surface of a metal.	Susceptible alloys	pH, temperature, ionic pollutants, aeration, uniformity and adherence of scale surface finish
Pitting	A form of localized corrosion that occurs when a corrosive environment medium attacks a metal at specific points and results in deep cavities in the metal. This is one of the most widespread and detrimental forms of electromechanical deterioration	Aluminum, stainless steel and other alloys in aircraft structures, in particular Al 7000 series	Surface finish including flaws and cracks, stagnant fluids, ionic potential of the electrolytes
Crevice	A form of corrosion that occurs when an electrolyte becomes trapped and stagnant, in particular locations such as joints, corners, and under debris.	Joints, corners, and where debris may accumulate	Stagnation of pollutants due to poor fluid flow results in excessive ions available to precipitate corrosion reactions
Galvanic	A form of corrosion resulting from the formation of a galvanic cell by the galvanic coupling of dissimilar metals (metals having different electrical potentials), which are exposed to an electrolyte.	Two dissimilar metals in direct contact or separated but in electrical contact, such as carbon/Al, or aluminum joints with steel bolts	Dissimilar corrosion potentials between adjacent materials, ratio of exposed anodic and cathodic materials, pH, aeration, and temperature
Erosion Corrosion	The increased rate of deterioration and loss of a material due to the combined effects of corrosion and the repeated motion of the surrounding environment such as repeated impacts from hard particles.	Moving corrosive environment or erosive hard particles or liquid.	Turbulent flow, fluid velocity, density, angle of impact, cavitation
Intergranular	A form of corrosion that attacks grain boundaries in materials. It may occur as a result of a galvanic couple between differing phases within a material. It is also associated with the propagation of pitting and exfoliation corrosion	Susceptible alloy/heat treatments	Alloying content impurities within the metal, heat treatment, welding effects, 2nd phase precipitation products on grain boundaries
Stress corrosion cracking	A cracking process involving the combined factors of corrosive environment and a sustained tensile stress.	Static stresses	Tensile stress (applied mechanical or thermal, residual)
Fretting Corrosion	Caused by repetitive friction between two surfaces in sliding motion with respect to each other while exposed to a corrosive environment.	Typically caused by vibration or repeated thermal expansion and contraction cycles	Vibratory movement between adjacent loaded components
Corrosion fatigue	The failure of a material due to the combined effects of corrosion and fatigue (cyclic stressing).	Cyclic stresses	Cyclic load
Hydrogen damage	Any deterioration of a material due to the presence of hydrogen, whether in the surrounding environment or internal to the material.	Hydrogen generation during processing or in service, in particular, landing gear and engine components	High temperature moist environments, electrolysis, hydrogen evolved from other corrosion mechanisms
Exfoliation	occurs in bands on the interior of the metal that is parallel to the metal's surface. This consequently forms corrosion products that cause separation of the metal into layers. It is often considered a form of intergranular corrosion that attacks metals that have been mechanically treated to form elongated grain structures in one direction.	Rolled and extruded alloys susceptible to intergranular corrosion	Similar to intergranular
Filiform corrosion	A form of corrosion that exists under organic and metallic coatings on metals, blistering the coating, and is characterized by hairline resemblance.	Thin permeable organic coatings on metals	Water permeable organic coatings or edges of alloys coated with metallic coatings exposed to high humidity

Table 2: Corrosion Sensor Types

Corrosion Factors	Sensors	Description	Pros and cons
Physical	Visual/enhanced visual inspection	Inspection of surface, intergranular, exfoliation and pitting corrosion.	<ul style="list-style-type: none"> • Low-cost and broad applications. • Mature technologies. • Online or offline measurements. • May not be able to distinguish corrosion from cracks. • May be applicable to uniform corrosion only. • Difficult to implement in distributed systems. • Difficult to detect corrosion in hard-to-reach locations.
	Eddy current		
	Radiography	Measurements of mass loss, defects, cracks and coating degradation due to corrosion.	
	Thermography		
	Electrical/inductive Resistance based mass loss sensors	Measurement of change in resistance, galvanic current, potential, impedance and acoustic emissions due to the presence of corrosion and the resulting mass loss.	
	Ultrasonic Sensors		
	Corrosion Potential-electric conductivity.		
	Linear Polarization Resistance		
Galvanic corrosion sensor [17]			
Electrochemical Impedance Spectroscopy [18]			
Electrochemical noise [19]			
Acoustic emission			
Environmental	Ion Selective Sensors (ISFET) [20]	<ul style="list-style-type: none"> • Monitoring of environmental factors responsible for corrosion events such as moisture, temperature and pollutants. 	<ul style="list-style-type: none"> • Real time on-board monitoring. • Easy to integrate into moving components and hard-to-access locations. • May be integrated into distributed networks. • Possibility of wireless signal transfer. • Prone to electromagnetic interference. • May be affected by exposure to harsh environments. • Prone to signal noise.
	Electrochemical/biological sensors [20]		
	Capacitive sensors [21][22]	<ul style="list-style-type: none"> • Monitoring of environmental factors through change in electrical, optical and acoustic responses of the sensors. 	
	Fiber Optics		
	Photosensors [23]		
	Surface Acoustic Waves (SAW)		
	Radio Frequency Identification (RFID)		
Hydrogen probes [24]			
Chemical	ISFET [20]	<ul style="list-style-type: none"> • Detection of by-products of corrosion such as, pH variation and metal ions. 	<ul style="list-style-type: none"> • Real-time on-board monitoring. • Easy implementation in distributed systems. • Versatile, compact and sensitive for chemical detection. • High selectivity toward specific chemical species. • Lack of long-term stability and vulnerability to noise. • Selectivity issues in the presence of other chemical species.
	Electrochemical sensors		
	Capacitive sensors [22]	<ul style="list-style-type: none"> • Change in electrical and optical response of sensors is induced by the presence of corrosion by-products. 	
	Fiber Optics [25]		
	Photosensors [23]		
	pH sensors [[26]		
Color-indicating-paints [27]			

4.0 NANOMATERIALS-BASED EMERGING CORROSION SENSORS

One of the key challenges for reliable corrosion sensing is the ability to detect not only the specific in-service environment such as the type of pollutant and its concentration, but also the presence of chemical byproducts such as metal ions. Currently, there are no commercialized chemical sensors for corrosion detection for early detection and monitoring. The discovery of organic semiconductors along with advancements in patterning and functionalization techniques have promoted significant innovation in sensor technology applicable in corrosion monitoring. This gap could be addressed by thin film material-based, surface-mounted sensors for the application of high-fidelity, real-time corrosion detection and measurements. Organic thin film conductors and semiconductors have attracted significant interests in electronic device fabrication due to unique structural, electronic, and chemical features, high sensitivity, solution processability, mechanical flexibility and low-cost fabrication [31]. These properties, combined with high surface area-to-volume ratio, their tuneable selectivity and low power consumption allow for miniaturization of devices. The success of such devices is also due to improvements in fabrication techniques such as deposition conditions and growth rate optimization, in addition to the diversity of organic materials and the versatility of their electronic and optical functionalities. Nanomaterial-based organic thin film-based sensors are excellent candidates to be used in corrosion monitoring which can benefit from their high sensitivity, selectivity, small footprint, compatibility to existing circuits and low power consumption.

4.1 Fiber Optic Based Pointwise and Distributed Sensor Systems

Optical fibers have drawn worldwide attention due to their larger bandwidth, long transmission ranges resulting from low power losses and excellent resistance to high temperature, corrosive environments and electromagnetic interference [32]. Optical fibers can be implemented as point-wise sensors, as well as distributed sensor system that provide multiple point remote measurements using a common source and detection systems coupled with an array of fiber optic sensors. In aircraft structural health monitoring, optical fibers can be used to detect structural parameters such as stress and strain, as well as environmental conditions responsible for corrosion, being moisture, pollutants, pH variations and metal ions. Fibre optics can be integrated into the structure of the aircraft as a surface mounted system, or embedded into composite materials during the manufacturing process. They could provide real-time data about the condition of the structure over its entire lifecycle. In spite of great advantages, the durability and longevity of optical fibres for onboard application, the practicality, and the cost can be barriers as aircraft health monitoring systems of aircraft.

Mendoza et al. developed an optical fiber-based sensor that detects moisture and pH variation in aircraft lap joints [23] as shown in Figure 2(a). Zhang et al. proposed a fiber optic temperature sensor using reduced graphene oxide (rGO) as the sensing material in Figure 2(b), showing high sensitivity, and precision [33]. Cooper et al. developed an optical fiber-based chemical sensor to detect moisture and corrosion by-products such as Cu^{2+} in aircrafts lap joints as shown in Figure 2(c) [34]. When the target species are absorbed by the affinity coating, the refractive index cladding changes, causing a shift in the wavelength of the reflected light. Zhang et al. also developed an ammonia gas sensor using microfiber optics coated with a layer of graphene oxide, demonstrating high sensitivity and good repeatability at relatively small fiber diameters [35].

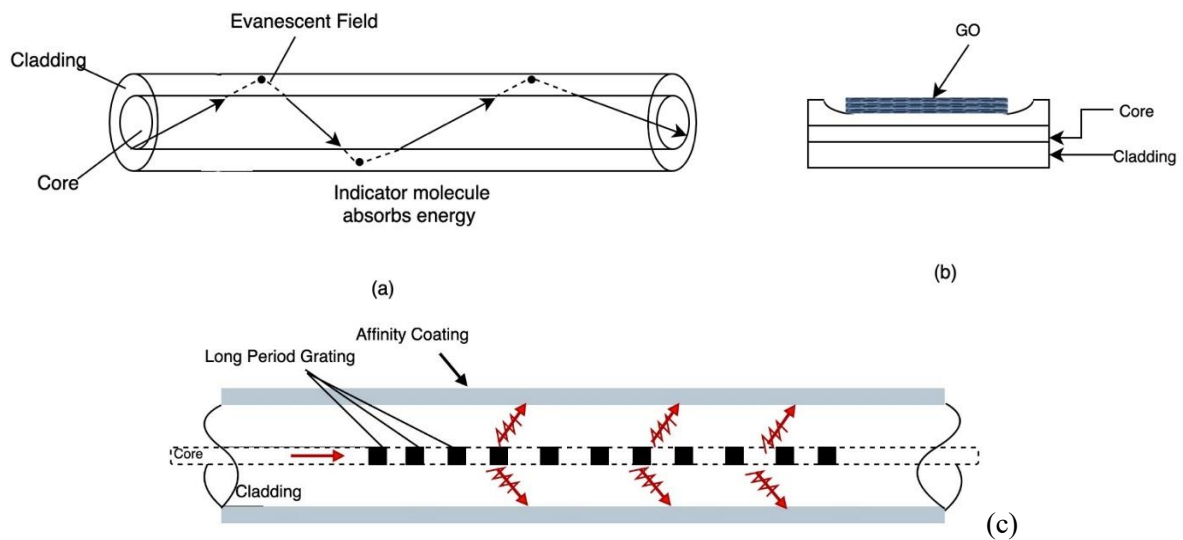


Figure 2: (a) Schematic of intrinsic optical fiber sensing mechanism [23]. (b) Schematic of graphene coated Side Polished Fiber [33]. (c) Schematic of long period grating optical fiber sensing mechanism [34].

Instead of converting the optical signal into electrical signal for processing purposes of fibre optical system, colorimetric sensing is an optical fiber-based scheme in which the sensing is conducted by an array of photodetectors, while the optical fiber operates as the communicating channel between the light source and the photodetectors. Figure 3(a) shows an optical fiber-based calorimetric sensor to measure chloride concentration and pH variations developed by Im et al [36]. The system is essentially a distributed corrosion sensor based on colorimetric multifunctional photo-transmittance, that employs side emitting optical fibers coated with measurand-selective colour variable membranes and a flexible phototransistor array wrapped around the fiber as shown in Figure 3(a) [36]. The color variable membrane is designed to change colour when in contact with specific measurands and acts as a light filter for the light emitted by the fiber. The gradual change in the colour of the membrane is captured by the phototransistor array and converted to electrical signals providing information about the location and the concentration of the measurand. The schematic of an active optoelectronic device consisting of a semiconductor, source, drain and gate electrodes and dielectric material is shown in Figure 3(b).

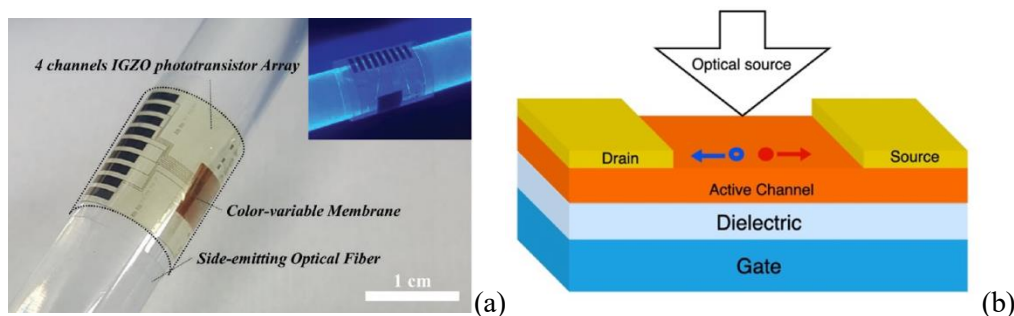


Figure 3: (a) Multifunctional colorimetric light-transmittance-based durability diagnostic system (b) Phototransistor lateral structure [36]

4.2 Organic Thin Film Transistor as Chemical Sensors

Organic thin film transistors (OTFT) in the form of ion selective electrodes, ion selective field effect transistors, electrochemical capacitive sensors and pH sensors offer great promise to meet the requirement for high resolution and low power as real-time corrosion monitoring of atmospheric corrosion parameters and corrosion rate of metallic structures, including the presence of chloride or metal ions.

4.2.1. Capacitive Sensors

Capacitive sensing detects the presence and measures the concentration of chemical species by measuring the changes in the capacitance between two or more conductors in a dielectric environment. Capacitive sensors have been prevalent for a long time, mostly for humidity and pH sensing, and have recently attracted interest in chemical sensing application due to their robustness and high sensitivity. Chemical capacitive sensors have simple structures comprising of two interdigitated electrodes (IDEs) coated with an ion-selective dielectric layer as shown in Figure 4(a) [38]. When the sensitive layer absorbs analyte ions, its physical properties and dielectric constant change, resulting in a change in the generated potential. One example of nanomaterial-based sensor is a flexible capacitive humidity sensor using graphene oxide (GO) sheets as the sensing layer developed by Alrammouz et al.[39]. The sensor was fabricated by depositing a set of interdigitated electrodes on self-assembled GO on a paper as shown in Figure 4(b). The porosity of self-assembled GO allows for the air flow monitoring from both directions which makes it suitable for monitoring of environmental factors. Strain tests showed that the bending angle had little or no effect on the sensitivity of the sensor which was comparable to resistive cellulosic paper-based sensors.

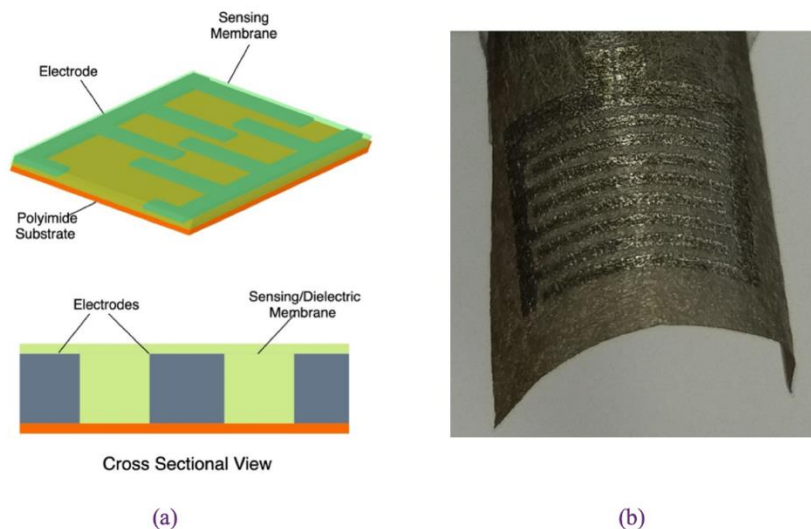


Figure 4: (a) Structure of IDC based chemical sensor. (b) Flexible GO-based capacitive humidity sensor [38]

While ion selective and capacitive sensing approaches have been demonstrated to successfully detect specific ions, there is still room for improvement in terms of selectivity and resolution. The presence of other ions can interfere with the response of the sensors, as they can reversibly bind with the sensing membranes leading to unreliable measurements. Furthermore, maintaining the sensitive membrane at a saturated state for long periods of time can be challenging, especially for polymer membranes, which compromises the long-term stability of the sensors.

4.2.2. Ion Selective Field Effect Transistor and Ion Selective Probes

Ion selective field effect transistors (ISFETs) are a class of potentiometric sensors that have been widely used in a variety of applications such as environmental monitoring, medical diagnostics and industrial process control. They are essentially three-terminal semiconductor devices in which the current is controlled by an electric field. They are typically comprised of a gate channel, source and drain electrodes as well as a gate insulator, as shown in Figure 5(a). Figure 5(b) shows the basic structure of a solid gate ISFET, which is a potentiometric sensor that produces electrical current in response to changes in ionic distribution. The sensing is conducted by coating the gate electrode with an ion-selective material such as ionophore mixtures or metal oxides, to which specific ions reversibly bind to and modulate the FET channel current and surface potential depending on the concentration of the ions. The state-of-the-art ISFET sensors use inorganic semiconductor materials, such as silicon, as the gate channel. However, scaling down such devices may be problematic as the

resolution depends strongly on the channel size, carrier mobility as well as the capacitance between the FET channel and the ion binding sites [38]. Nanomaterial-based ISFETs can potentially overcome these challenges owing to the large surface to volume ratio and high carrier mobility of nanomaterials. Fakhri et al. developed an electrochemical sensor array based on graphene ISFETs that measures the concentration of Cl^- and other ions, using ion selective membranes deposited on a graphene channel. The sensor array provides real-time and simultaneous measurement of multiple ions concentration in multi-ion electrolyte solution and exhibit high sensitivity and high resolution [38].

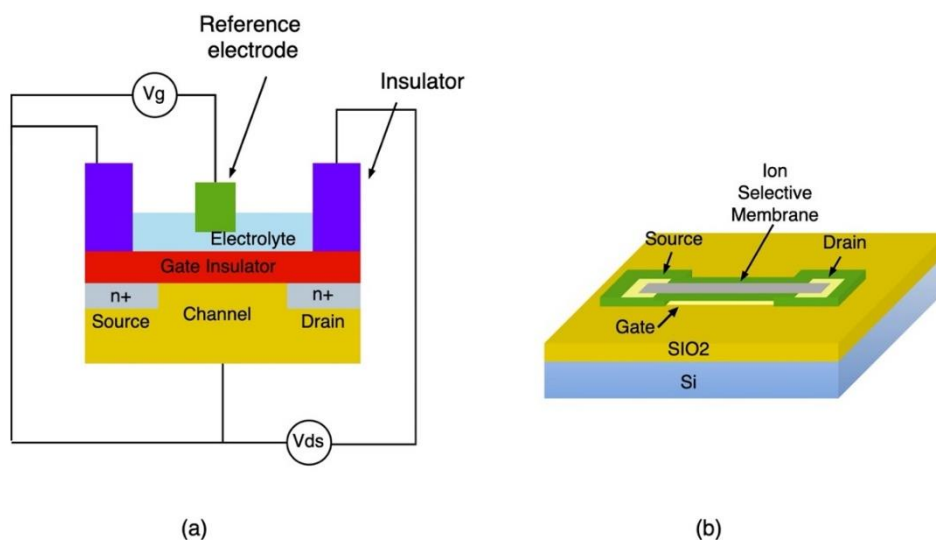


Figure 5: (a) Solution ISFET sensing system (b) Solid gate ISFET sensor structure

Ion selective probes are another class of potentiometric sensors to detect and measure the concentration of specific ions by measuring the voltage variations across two electrodes in the absence of current flow. They have been widely used in clinical and environmental monitoring due to their robustness, selectivity over competing species, fast response and low-cost. Ion selective probes are integrated electrode systems consisting of two or three electrodes depending on their application. One of the electrodes is used as the working electrode (WE) and the others as the reference electrode (RE) and the counter electrode (CE). The electrodes are usually made from the same or different materials, but the working electrode is coated by an ion-selective material or membrane and acts as the transducer element, whereas the RE/CE maintains a constant potential and is insensitive to changes in the analyte composition [40]. The ions concentration measurement is carried out by bringing the electrodes in contact with the analytes. The interaction between the analytes and the ion selective membrane on the working electrode results in variations in the potential, which is correlated with the ion concentration [40]. There have been several attempts in the transition toward the use of nanomaterials in the fabrication of chemical sensors due to their abundance, low-cost and competitive attributes in terms of sensitivity and fast response. For instance, Li et al. developed a graphene-based solid contact ion selective electrode (ISE) to measure the concentration of potassium [41]. The ISE is fabricated by depositing graphene sheets onto polished glassy carbon (GC) electrodes and coating the GC/graphene electrode with an ion-selective membrane as shown in Figure 6. The GC/graphene-based ion selective electrodes performance was found comparable to carbon nanotubes (CNTs)- electrodes with improved long-term stability allowing for miniaturized and mass fabrication of ISE based sensors.

Ion selective field effect transistors (ISFET) have been seen as an emerging pH sensing technology, which are focused on highly sensitive miniaturized robust devices that are able to provide in situ long-term pH monitoring in harsh environment, because they overcome the limitations of conventional methods of paper strips and glass electrodes, including fragility, low sensitivity and unsuitability for miniaturization [26]. Ion selective field effect transistors for pH sensing can be classified in two categories based on type of gate used- Solution-gated ISFETs and Solid gated-ISFET.

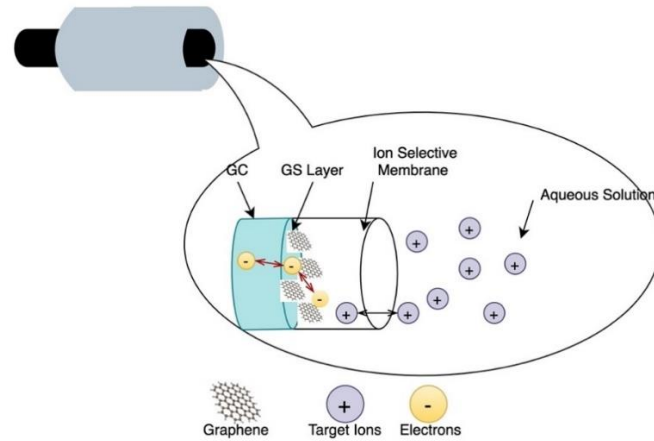


Figure 6. GC/graphene ion selective electrode structure [41]

Solution-gated ISFETs are field effect transistors resembling a three-electrode chemical cell, consisting of a conducting channel, a gate insulator, source and drain electrode and a reference electrode. In this configuration, the electrolyte solution is in direct contact with the conducting channel and the gate voltage is directly applied through the reference electrode as shown in Figure 7(a). Nanomaterials have been used as both the conducting channel and the sensing layer. Zhu et al. proposed a graphene-based solution gated-field effect transistor for pH sensing, showing good sensitivity and resolution [42].

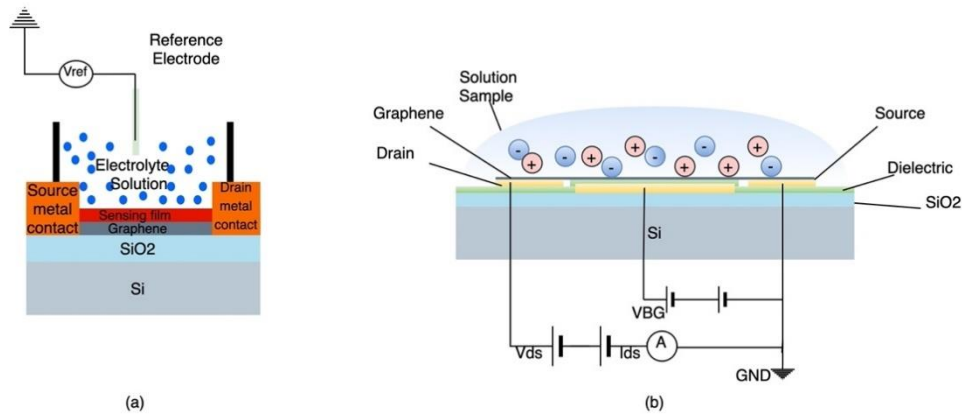


Figure 7: (a) Solution-gated ISFET structure. (b) Solid gated-ISFET based pH sensor [42].

Solid gated-ISFET structure is similar to the solution gated-FET but it is characterized by the absence of the reference electrode, which can be cumbersome in some applications. In this configuration, the dielectric layer is sandwiched between the gate electrode and the conductive layer, and the gate voltage is directly applied to the electrolyte through the gate dielectric as shown in Figure 7(b). Investigations into the influence of the gate dielectric characteristics on the performance of the sensors indicate that a material with a high dielectric constant allows for the dielectric layer thickness to be reduced, thereby lowering the leakage current while maintaining a high sensitivity. For instance, Zhu et al. fabricated a graphene based solid gated-FET using a thin layer of HfO_2 as gate dielectric. Usage of HfO_2 of a high dielectric constant allowed for a significant reduction of the dielectric layer thickness to achieve high capacitance [42]. Since the sensitivity of the sensor is directly proportional to the capacitance of the dielectric, the pH sensor exhibits a large transconductance while operating at low voltages. Another approach to improving the performance of solid gated-ISFETs, is the use of 3D graphene as the conducting channel, which was proposed by Ameri et al [43]. The designed pH sensor makes use of graphene foam as the conducting layer coated with a sensitizing layer made of HfO_2 . The use of suspended 3D graphene configuration, combined with its large surface area, allows for thorough

electrostatic gate control, which enhances the coupling between the electrolyte ions and the graphene layer. The sensitivity of the device is also enhanced by the presence of binding sites for hydrogen ions provided by the HfO_2 coating. The proposed device demonstrated high pH sensitivity in high ionic strength media, offering the potential for real-time pH monitoring.

These experimental results highlight the versatility of graphene-based pH sensors in terms of ease of tunability, processability and compatibility with flexible printable devices. Each configuration offers unique advantages such as the use of reference electrodes in solution gated ISFETs which reduces the effects of solution variability, and its absence in solid gated ISFETs allows for device miniaturization and improves integration and microfabrication.

4.2.3. Sensor Nodes

In the aircraft industry, monitoring corrosion is crucial in ensuring the durability and safety of aircrafts, which requires arrays of sensors that track various chemical species responsible and indicative of corrosion damage. Tracking the various types of chemical species on the same structure can quickly become complex and costly as each sensor needs its own circuitry to relay sensor data. Wireless sensing networks is an attractive concept of distributive real-time monitoring, where a number of sensor nodes are used to monitor different chemical species and transmit their data to a central data acquisition unit [44]. Sensor nodes are simple transducers with sensing, networking and in some cases, processing capabilities, integrated along with other sensors in a sensing interface, all connected to a controller board that manages data acquisitions from the various nodes and performs processing operations. The concept of multi-parameter integrated sensors was investigated by Rinaldi et al., where a distributed corrosion sensing network was proposed to detect hydrogen, relative humidity, and temperature variations [45]. The system includes polyaniline/carbon nanotubes-based sensors for hydrogen gas and humidity detection. Polyaniline (PANI) is an intrinsically conductive polymer characterised by high conductivity, low density, environmental stability and ease of processability. Combining carbon nanotubes (CNTs) with PANI enhances the conductivity of the semiconductor by improving the charge carrier mechanism in PANI. In this configuration, PANI/CNTs composite material was deposited on simple sensing chip made of interdigitated electrodes as shown in Figure 8. The sensor exhibits excellent sensitivity to humidity compared to commercially available sensors. However, its response to variation in hydrogen concentration was limited by hydrogen bonding sites in the PANI/CNTs material or the presence of residual moisture as a result of the fabrication process. Nonetheless, the concept presented is indicative of the feasibility of such networks which features the advantages of sensing nodes in decreasing the complexity of distributed sensing systems combined with the benefits of using organic semiconductors.

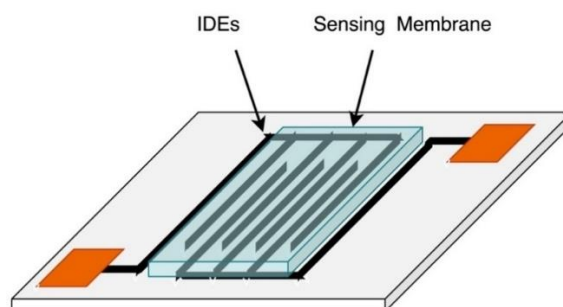


Figure 8. Schematic of an IDEs chip with PANI/CNTs conductive coating [45]

Sensor nodes is a monolithic integration approach that incorporate sensors targeting different analytes in the same node. It is a novel approach that can be useful in aircraft corrosion monitoring, where several sensors targeting the corrosion factors are encapsulated in the same node and placed in critical locations of the aircraft structure. The sensor nodes can have a common data acquisition and processing unit and the connection can be wired or wireless or both using wireless sensors like radio frequency identification. This configuration allows for continuous monitoring of corrosion environment, by-products and progress while decreasing the complexity of the sensing network and the associated installation and maintenance costs.

5.0 ONGOING PROGRESS OF CORROSION SENSORS IN CANADA

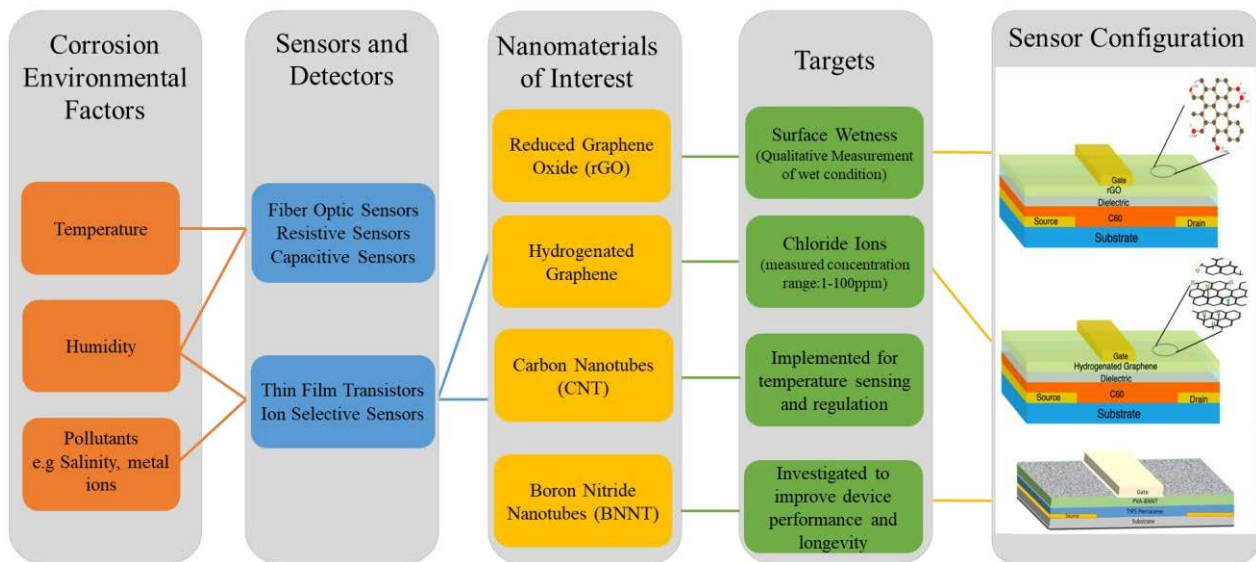


Figure 9: Current approach to creating a nanomaterial-based sensor node for detecting multitude of corrosion environment analytes and corrosion by-products.

Our approach to implementing graphene derived nanomaterials in environmental sensors is inspired by recent work and established evidence of the benefits offered by the nanomaterials for the applications in biosensors, super-capacitors and catalysts [46]. So far, the work in Canada has demonstrated the applications of graphene oxide and graphene modified materials, rGO and hydrogenated graphene as sensing material for Cl^- ion concentration and surface wetness level, respectively [47]. The sensing layers were incorporated in a fullerene C_{60} , n-type organic thin film transistors (OTFT) as illustrated in Figure 9. The idea behind developing the combined sensing node arises from the challenge of detecting Cl^- ions or metal ions in the presence of high humidity. The rGO OTFT wetness sensor was designed for qualitative measurements of surface moisture, while Cl^- ions sensor as used to provide quantitative measurements of Cl^- ions concentrations. In conjunction with the two sensors, resistive temperature detectors were incorporated in the test setup, along with a miniaturized Peltier temperature control module, as shown in Figure 10(a).

The surface wetness sensor response before and after exposure to water under constant bias of 1V is shown in Figure 10(b). The conductivity of rGO is enhanced as a result of chemical bonding between the hydroxyl groups present in rGO and water molecules which results in electronic sensor response to change in surface wetness. The sensor displays a long and sharp linear response after exposure to humidity and saturates at high current, at approximately six-fold larger than the sensor device current under dry conditions. The increase in the device current is due to an increase in charge carrier density in the rGO conductive layer. When rGO is exposed to water, the presence of hydroxyl groups promotes bonding with water molecules which acts as hole donors. Since rGO is a hole carrying material, the increase of hole density brought by water molecules, results in an increase of conductivity apparent in the output characteristic. As moisture is introduced, the sensor switches on as indicated by the increase in output current attributed to the increase in conductivity. After complete removal of humidity using surface heating through the Peltier module, the sensor turns off and the output current returns to the baseline value [47].

The sensing performance of the hydrogenated graphene OTFT chloride ion sensor was then evaluated under controlled wetness condition from the previous step and compared to a control OTFT device with an unmodified graphene nanoparticle conductive layer. Cl^- solutions in water were prepared using various salt concentrations (NaCl and KCl) and were deposited onto the sensor surface. The performance and response of the sensors were tested after complete evaporation of water using test setup shown in Figure 10(c). The chloride ion sensors turn on at $V_{\text{DS}} = 2 \text{ V}$ which is in accordance with turn-on voltage of the OTFT and undergo a decrease in the saturation current that is associated with the decrease in conductivity resulting

from hydrogenated graphene bonding with chloride ions. The effect of Cl^- ions concentrations is also observed in the decrease of the linear response range of the OTFT due to the decrease in charge carrier mobility attributed to the chemical reduction of hydrogenated graphene stemming from the reaction of Cl^- ions and H^+ ions. The observed response of the control device does not display any dependence on Cl^- ions concentration and demonstrates that pristine graphene does not exhibit chemical reactivity to the presence of Cl^- ions.

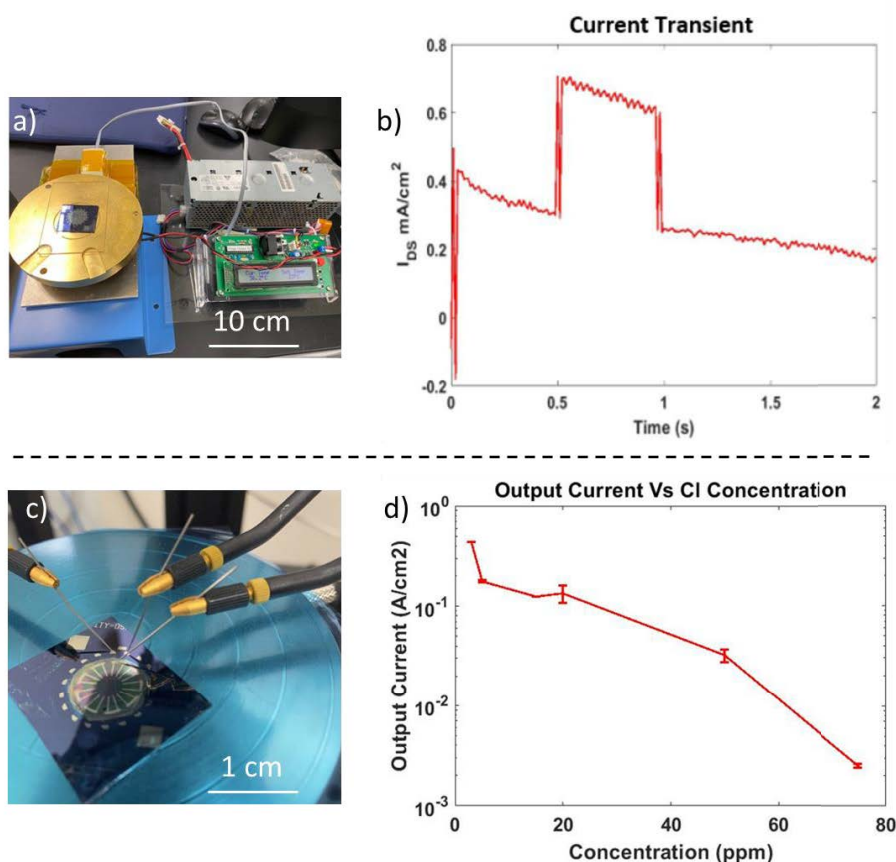


Figure 10: Nanomaterial based OTFT sensors for detecting surface wetness a) at controlled temperature, b) OTFT sensor ON/OFF response to surface wetness, c) Nanomaterial based OTFT sensors for detecting chloride ion concentration, and d) logarithmic sensor response curve.

A total of 80 OTFT chloride ion sensors with channel length ranging between $10\ \mu\text{m}$ and $25\ \mu\text{m}$, and aspect ratio ranging between 10 to 50, were fabricated and tested against the targeted range of Cl^- concentration (3 ppm-75 ppm) with a device yield of 70%. The measurements pertaining to each concentration value were repeated several times to evaluate the spread of measurements and plotted along with sensors response curve in Figure 10(d). The collected data for each concentration does not deviates significantly from the mean, which proves the reliability of the measurements. The sensors were tested repeatedly in a lab environment over a period of four weeks in which they remained operational for an average of two to three weeks. The calculated detection limit of the sensor was found to be 4% ppm.

The reported sensor results in Figure 10 demonstrate the potential of hydrogenated graphene nanomaterials in the detection of Cl^- ions using OTFT sensors. The sensors are capable of detecting minute concentrations of such ions far below reported corrosion initiation thresholds of 100 ppm to 3500 ppm, depending upon the type of metal alloy, ambient temperature, and pH. Our approach to producing a monolithic sensor node design can facilitate adding other peripheral sensing parameters to the existing footprint by suitably modifying nanomaterials to those specific analytes. The logarithmic response of the sensor provides a significant enhancement of sensitivity for low concentration.

In addition to incorporating nanomaterials as chemical sensing materials for corrosion monitoring, unique implementations of nanomaterials as structural and functional materials were also performed to enhance key sensor device characteristics such as inertness to surface moisture, less susceptibility to temperature variation with a predictable response, and device longevity. Susceptibility to moisture and low shelf-life have proven to be a major barrier to entry for academically reported chemical corrosion sensors where conventional low-cost printing materials are unable to meet the desired industry specifications. Currently investigation of an inorganic nanomaterial, boron nitride nanotubes (BNNT), is taken place to create robust composite polymer dielectric and encapsulations which can improve the bespoke OTFT's performance and shelf-life, brining them a step closer to on-site testing and commercial prototyping stage.

BNNTs are structurally similar to CNTs, except carbon atoms are replaced with alternating boron and nitrogen atoms. These polymorphed tubes are in the form of hollow cylinders on the nanometric scale. BNNTs have very dynamic properties such as immunity to electromagnetic interference, resistance to harsh environments and corrosion, thermal stability for up to 800°C [48]. This makes the electrical insulator a very promising candidate for OTFT sensory devices operating in variable or extreme environments [49]. OTFTs are identified as a prime candidate to utilize these favourable properties and a study investigating the dielectric characterization of BNNT modified poly vinyl alcohol (PVA). The expected outcome of using thin films of BNNT-PVA as a dielectric in OTFT is that we can obtain higher and more stable dielectric properties, with high thermal resistance and longer shelf life.

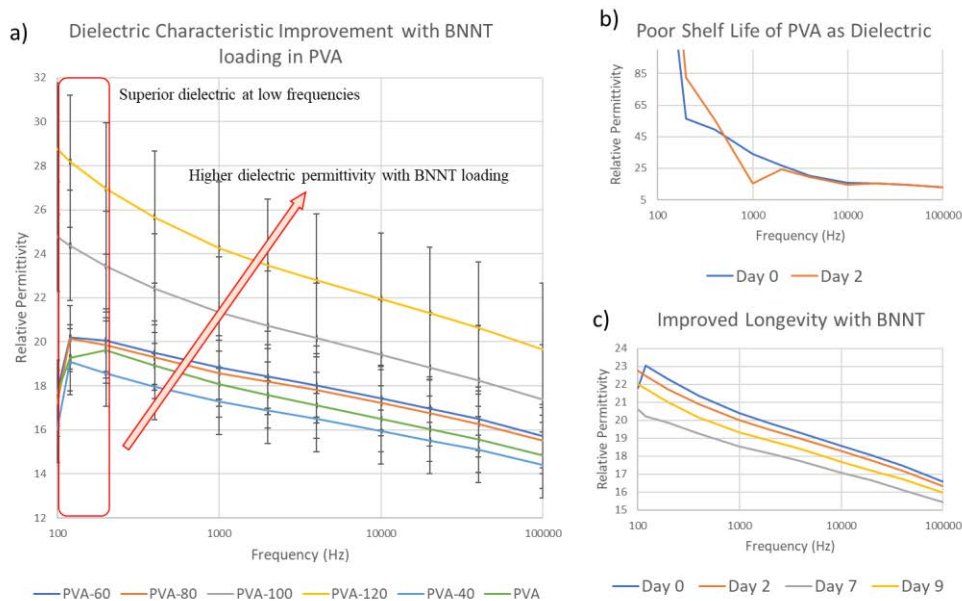


Figure 11: (a) Improved dielectric properties of PVA with BNNT loading, (b) Short shelf life of PVA based devices, (c) Improved shelf life and device performance for BNNT loaded PVA devices.

Figure 11 shows recent experimental findings on incorporating BNNT nanomaterial into the PVA dielectric layer for the next-generation OTFT sensor designs. To investigate dielectric performance and shelf life, over 50 metal-insulator-metal capacitive structure were fabricated and tested daily and intermittently over two weeks period. The BNNT-PVA dielectric were far more stable under low frequency loading and yielded superior dielectric permittivity even at ultra-low BNNT doping levels (< 0.1%) (see Figure 11(a)). Shelf life analysis indicates that the higher BNNT loading films had noticeably less degradation than their lower BNNT loading counterparts and all BNNT loaded films possessed significantly longer device shelf life than the pristine polymer dielectric as shown in Figure 11 (b, c). These preliminary studies have confirmed that BNNT nanomaterial is a suitable dopant for our next-generation OTFT corrosion sensor studies.

Using nanomaterial-derived chemical sensing is still at the experimental stage and requires further improvements toward selectivity, specificity, and long-term sensor stability and durability. The major challenges reside in the complexity of functionalization process which require control of process parameters

at the atomic level to ensure the reliability and repeatability of the sensors as well as their selectivity and specificity. Advancements in chemical functionalization and patterning techniques allow for the transition toward miniaturized and monolithic sensing systems capable of monitoring multiple parameters simultaneously on a continuous basis. Furthermore, implementation of nanomaterial-derived materials in the emerging corrosion sensing techniques offers a wealth of possibilities for the realization of efficient and reliable real-time corrosion sensors for aircraft structural health monitoring while reducing maintenance and repair costs and maintaining the durability, sustainability, and safety of the fleets.

In spite of progress and promised benefits, there remain many challenges. First, corrosion is complex. While there are various mature corrosion sensors for detecting corrosion on the surface of a structure, it is difficult to detect corrosion that takes place underneath the surface, such as pitting and galvanic corrosion. Second, the Technology Readiness Level (TRL) of onboard corrosion sensing systems is not adequate for commercial applications. The onboard systems for corrosion monitoring are based on detection of corrosion environments such as temperature, humidity, time of wetness and conductance of electrolyte. High false-positive call rates are one of the key issues of such environmental corrosion sensors, which raises the question whether corrosion environment data alone is sufficient for reliable detection of corrosion. Qualification for on-board sensor sensitivity and durability is another key area to determine in-service failure rate and the service life of corrosion sensing systems. Third, there remains the challenge to integrate corrosion related information with aircraft conditioned based management systems. This requires not only the development of a corrosion prognostic system to quantify actual corrosion state based on sensor systems, but also structural fatigue life prediction tools to integrate corrosion as part of conditioned-based maintenance.

6.0 CONCLUSIONS AND OUTLOOK

To address the needs for enhanced aircraft availability and reduced maintenance cost, proactive corrosion management is paramount to enable early corrosion detection, and continuous monitoring and integration of corrosion rates into the aircraft structural integrity program. The core to the new management regimes is an integrated management system consisting of reliable and durable corrosion sensors and accurate methods to predict corrosion conditions. This paper provides detailed descriptions of the categories of corrosion factors, namely, physical, chemical and environmental factors for corrosion detection and existing sensing technologies.

The recent work on the sensing performance of a hydrogenated graphene OTFT chloride ion sensor demonstrated its ability to detect Cl^- ions with exceptional sensitivity. The logarithmic response of the sensor provides a significant enhancement of sensitivity for low concentration, with an observed detection limit of 4% ppm. The ongoing work of using boron nitride nanotubes (BNNT) modified poly vinyl alcohol dielectric has been found to offer superior dielectric permittivity, and environmental stability and long shelf life. These preliminary studies have confirmed that the BNNT nanomaterial is a suitable candidate for the next-generation OTFT corrosion sensors. Our approach to producing a monolithic sensor node design can facilitate adding other peripheral sensing parameters to the existing footprint by suitably modifying nanomaterials to those specific analytes.

Overall, the emerging nanomaterial-derived corrosion sensors hold great promise for proactive corrosion management due to the exceptional sensitivity and selectivity for multi-point real-time detection and monitoring of corrosive environments and chemical by-product. The class of sensors also offer great benefit of low power to operate, compact size, ease of manufacturing. This paper highlights some of the notable corrosion sensors which implement various nanomaterials to elicit sensor response to specific environmental factors or corrosion by-products. Notably, CNT, rGO, and hydrogenated graphene have been implemented for monitoring temperature, humidity, surface wetness and chloride ion concentration within a monolithic sensor node design for advanced corrosion control and management. Inorganic nanomaterial BNNT is being tested as a robust insulating material for future OTFT environmental sensors to improve sensor performance and longevity for real-world testing and commercial prototyping applications. The next step of the work is to expand the sensor node to include pH, metal ions and hydrogen ions for next-generation advanced aircraft corrosion monitoring systems. The nanomaterial-based surface mounted sensors have progressed quickly in recent years, but work is needed to increase the technology readiness level for on-aircraft applications.

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